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Bleaching cotton in textile conservation: a closer look using atomic force microscopy

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Abstract

Aqueous bleaching may be used in textile conservation to improve the appearance of historic and culturally significant textiles. It is generally accepted amongst conservators that bleaching imparts damage. The aim of this research is to characterise the condition of cotton fibre's surface pre- and post-bleaching using atomic force microscopy (AFM). Unprocessed cotton calico ('raw' cotton), scoured cotton, and a historic cotton dress shirt (*circa.* 1920) were bleached using three separate methods: $NaBH_4$ for 15 min; $H_2O_2/NaBO_3$ for 1 h; and $H_2O_2/NaBO_3$ buffered to pH 8.4 for 1 h. AFM was used in tapping-mode to obtain height, amplitude, and phase images. AFM imaging was able to distinguish between the cuticle, primary walls, and secondary walls of the cotton fibres. The data shows that bleaching has the effect of softening and removing individual layers of the cotton structure. Unprocessed cotton calico and scoured cotton fared better against the impact of bleaching. This was in stark contrast to the historic shirt where the already damaged surface of cotton fibres underwent further degradation using both oxidative and reductive bleaching. In general, reductive bleaching was more aggressive on the fibre surface compared to oxidative bleaching. The use of AFM provides further evidence of the physical effects of bleaching on historic textiles, and cotton textiles more broadly, and it has the potential to influence the conservator's decision-making.

Keywords: Textiles, Conservation, Cellulose, Atomic force microscopy, Nanoscience, Fibres, Cotton, Bleaching

Introduction

Bleaching in textile conservation is not routinely used although it is a last resort where other methods fail to reduce staining and discolouration to improve the textile's appearance (if this is considered an important aspect of the conservation treatment). In a series of workshops on bleaching, as part of teaching on the MPhil Textile Conservation programme at the University of Glasgow,[1] it has been observed in textiles post-bleaching that, despite using controlled conditions, the fibres can feel drier and stiffer, raising questions about the impact of bleaching at the fibre level. Developing a further understanding of changes taking place at the fibre level is important to enable conservators to evaluate when, and if, it is appropriate to use such methods.

The aim of this research is to provide objective scientific data from high resolution microscopic analysis on the impact of bleaching on the surface of cotton fibres from various samples. To achieve this, oxidative and reductive bleaching methods have been performed separately on unprocessed cotton calico (described as 'raw' cotton for the purposes of this research), scoured cotton, and a historic cotton dress shirt (*circa.* 1920). Analyses have been carried out using atomic force microscopy (AFM) and scanning electron microscopy (SEM). AFM can probe the surface of individual cotton fibres, enabling the surface to be characterised through properties such as elasticity and topography. With this data, it is possible to establish processes such as stripping of the outer layers, general damage, and even a rearrangement of the cellulose polymers. The decision to use AFM, with its high

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[1] On the MPhil Textile Conservation programme bleaching has been routinely taught over several years.

resolving power, was taken as initial studies using optical microscopy of cross-sections of bleached cotton samples over extended periods (ranging from 0 to 24 h) demonstrated damage to the fibre surface and interior [1].

The structure of the cotton fibre has been researched for many decades by various methods including optical microscopy [2, 3], electron microscopy [3], Fourier transform infrared (FT-IR) spectroscopy [4–6], and X-ray diffraction (XRD) [6]. In the most recent textbook on the cotton fibre [7] a contributor states, "Still, at the time of this writing, much remains to be learned about many of the details of the cotton fiber structure. These structural details must become known to understand the relations between the structure and performance properties of the fiber" [8]. In this paper we will give a general accepted description of the cotton fibre.

Cotton, as a vegetable fibre, has a chemical composition of cellulose (82–96%), hemicellulose (2–6.4%), lignin (0–5%), and pectin (< 1–7%) [9]. The cotton fibre has a multi-layered structure, and its components are distributed throughout these layers (Fig. 1). The main layers of the fibre are the cuticle, primary walls, and secondary walls with the lumen at the centre being a hollow channel that runs the full length of the fibre [10]. The primary wall has two components, namely, the primary wall and transition (or winding) layer. Cotton fibres are characterised by their flattened shape, convolutions (ribbon-like twists throughout the fibre), a diameter of 18–25 μm, and a length of 22–50 mm [10].

The cuticle consists primarily of waxes which provide protection. The primary walls contain cellulose fibrils and other components in a semi-crystalline arrangement.

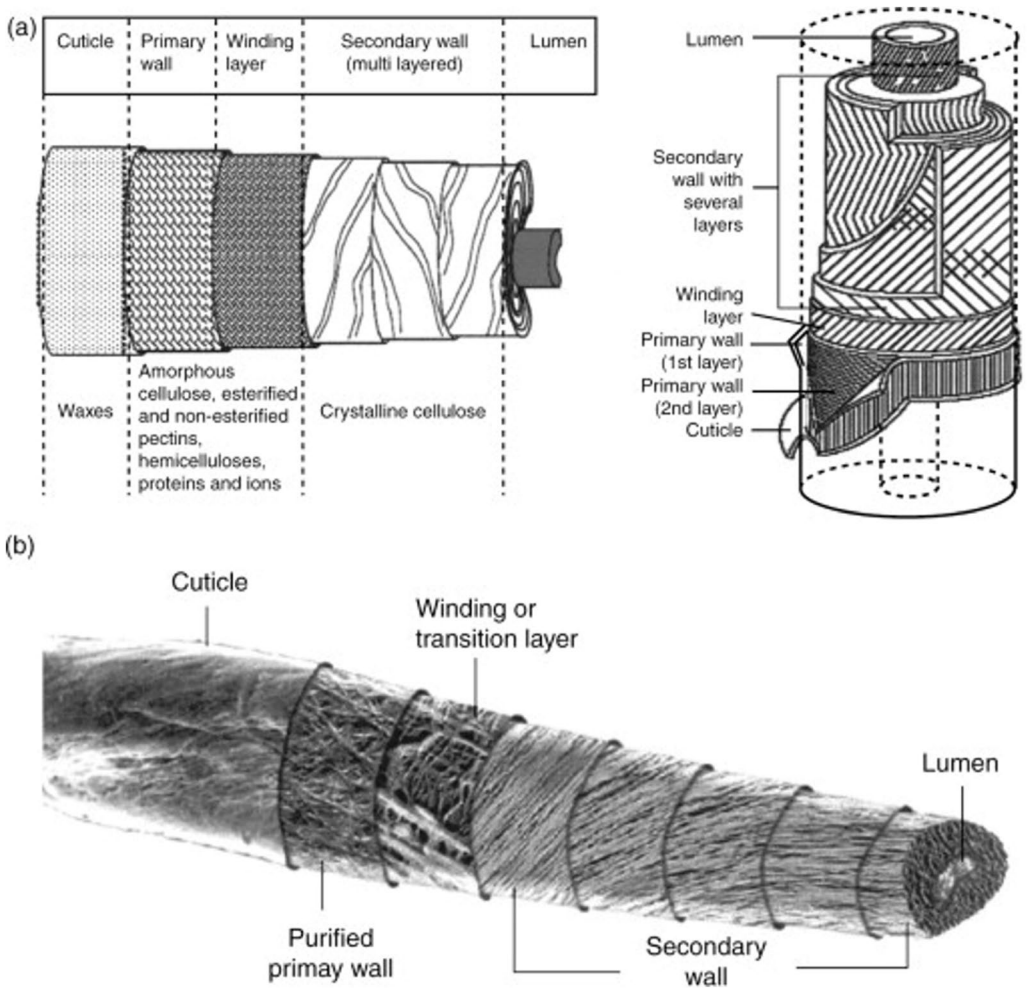

Fig. 1 A schematic representation of mature cotton fibre showing its various layers. (**a**) Cross section of cotton fibre. Typical components in dry, mature cotton fibres and compositions of each layer. (**b**) Morphological model of cotton fibre. Reproduced by permission of Taylor & Francis Group from [10]

Fig. 2 Historic cotton dress shirt (*circa.* 1920, Karen Finch Reference Collection, University of Glasgow)

The winding layer (alternatively called the S1 layer) is also composed of semi-crystalline cellulose and contains fibrils orientated at 40–70° to the fibre axis. This is a unique feature of cotton which varies along the fibres, giving rise to the characteristic convolutions. Cellulose is mostly found in the secondary wall (alternatively called the S2 layer). The secondary wall is organised as layers of nano-fibrils of cellulose. The nano-fibrils themselves, though described as being crystalline are believed to consist of amorphous and crystalline components. The crystallinity of the cellulose is determined by its crystallinity index (CI) [11]. Models have been proposed and research to establish the real structure of the nano-fibrils in the S2 layer is on-going [12]. The layers of the secondary wall are orientated at different angles from each other by a few degrees (Fig. 1). The innermost layer is normal to the fibre axis whilst the outermost layer is approximately 40° to the fibre axis. The layers are held tightly together through hydrogen bonding. Lastly, at the very centre of cotton fibres is the lumen, a hollow channel important for cell growth.

To understand how the different sections of a cotton fibre are affected by bleaching, it is important to investigate several types of cotton textiles. Aged and new cotton samples were tested. The aged sample was from a dress shirt (*circa.* 1920) (Fig. 2). The shirt has likely endured many washings, contact with sweat, abrasion, and natural ageing. All these factors have certainly contributed to damage observed at the fibre level. Two other cotton textiles used for this research are raw (unprocessed cotton calico) and scoured cotton. Calico is a woven cotton textile prepared with unbleached cotton that has undergone minimal processing. The 'unfinished' quality of calico makes it close to a raw cotton sample. In contrast, scoured cotton has undergone wet processing and so the texture and finish of the completed textile will show some distinction when compared to the calico. The traditional method of scouring is alkali scouring and is performed to remove greases, waxes, and other undesirable substances from the textile. The appearance of the textile becomes whiter and scouring makes the textile more absorbent for any further bleaching that is required [13]. Generally, scouring includes soaking the cotton textile in a caustic solution at a high temperature. This causes waxes and pectin to become water-soluble products which are removed at the final stage of the process where the textile is thoroughly rinsed in water [13–15]. The disadvantage of scouring is that it can introduce changes to the crystalline structure, leading to changes in the physical properties of the textile. Damage noted on cotton textiles post-scouring are a decrease in strength and flexibility [15]. The main advantage in favour of scouring is that it yields a high-quality cotton textile suitable for further dyeing and finishing treatments [14].

Historic textiles may have been exposed to different mechanisms of ageing which can be divided into five main categories: physical, mechanical, thermal, chemical, and photochemical [16]. Aged cotton textiles may be discoloured or stained due to age and/or use. Photochemical and chemical attack can break and form new covalent bonds in the fibres leading to changes such as yellowing of cotton items. Such discolouration and staining can be lessened through chemical changes and solubilisation of the yellowing/staining or with a 'whitening' of the object using several methods including oxidative or reductive bleaching [17, 18].

Oxidative bleaching with hydrogen peroxide (H_2O_2) can be successful under acidic or alkaline conditions. With acidic conditions, reducing oxycellulose can form giving rise to aldehydes and ketones which may carry out reduction reactions [19]. Under alkaline conditions, carbonyl groups can oxidise to form carboxyl groups, presenting a change to the fibre structure whereas reducing oxycellulose may fragment the polymer chains in cellulose. The consequence is the fibres have impaired mechanical strength and decreased flexibility [19, 20].

Within textile conservation, oxidative bleaching is believed to be more damaging than reductive bleaching [19, 21, 22]. The behaviour of H_2O_2 is considered more destructive particularly through chemical interactions. Shifts towards gentler processes are employed involving bleaching at room temperature, with low concentrations, stabilised with sodium perborate, and the pH buffered to pH 8.5 to minimise any damaging effects [19, 23].

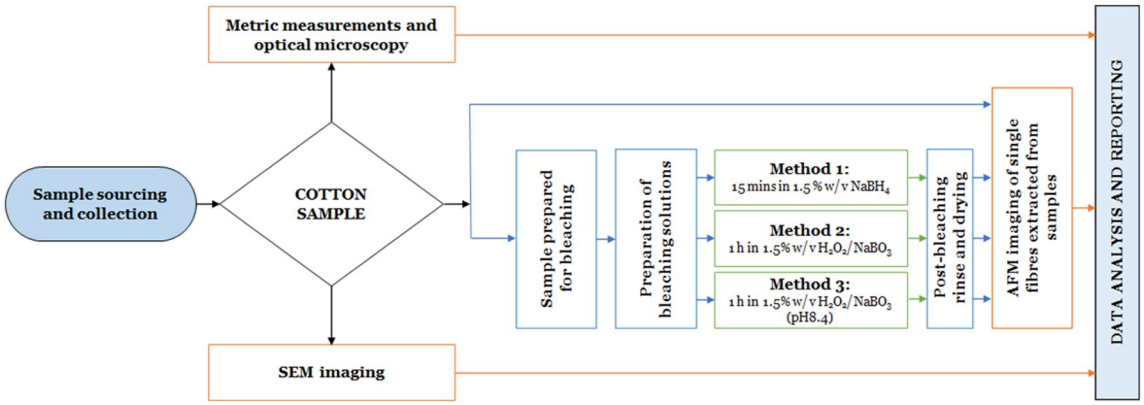

Fig. 3 Flow chart of experimental steps in the bleaching and analysis of each cotton sample

With regards to reductive bleaching with sodium borohydride ($NaBH_4$), there is some research into the effects of reducing agents on historic cotton textiles [24–26]. Like their oxidative counterparts, reducing agents work against stains and reduce discolouration to improve the visual appearance of textiles, converting certain insoluble stains into soluble products. Reducing agents are also considered to have a stabilising effect on oxidised cellulose [26, 27]. The change in visual appearance is achieved through converting carbonyl groups back into their original hydroxyl functionalities which appear colourless [26].

Predominately, conservation research has focused on the chemical changes to the fibres post-bleaching, specifically concerning tensile strength, degree of polymerisation, and changes to the oxidative state e.g., carbonyl group content and molecular weight [24–27]. Based on work carried out by the authors on the study of thin cross-sections using light microscopy as mentioned above, the use of microscopic techniques has the potential to study the morphological properties to provide further insight into the effects of bleaching on historic cotton fibres. One of the more common microscopic techniques used in textile conservation is the SEM [28, 29], with two previous investigations which used historic textiles concluding that SEM was highly informative when determining the extent of degradation [30, 31]. AFM has been used in the study of fibre condition [32] but is not widely used in this context. Regardless, AFM does have promising potential to show changes to the cotton fibre surface post-bleaching which could be advantageous for conservators and their textile artefacts.

Methodology

Samples

Three textile samples were used in this study: 'raw' cotton calico (Whaleys (Bradford) Ltd.), commercial scoured cotton (Mandors Fabric Store, Glasgow), and a historic cotton dress shirt (*circa.* 1920, provided by the University of Glasgow, Fig. 2).

The protocol for the experimentation is outlined in the flowchart shown in Fig. 3. Descriptions for each stage are provided below.

Sample preparation

Prior to bleaching, each of the three textiles were cut into 0.5 × 0.5 cm squares to accommodate the number of experiments carried out simultaneously. All glassware was rinsed using detergent, dilute 0.1 M hydrochloric acid, acetone, and deionised water (in that order).

Oxidative bleaching

Two methods were used for oxidative bleaching according to reported methods [24]: 1.5% w/v $H_2O_2/NaBO_3$ (hydrogen peroxide/sodium borate) and 1.5% w/v $H_2O_2/NaBO_3$ (buffered to pH 8.4).

The $NaBO_3$ solution was prepared by weighing 1.2007 g of $NaBO_3.4H_2O$ (Fisher Scientific) and fully dissolving in 150 ml deionised water. To this, 50 ml H_2O_2 (3% w/v, Fisher Scientific) was added to achieve a working strength of 1.5%. The second buffered solution was prepared by adjusting the pH of the $H_2O_2/NaBO_3$ solution by adding glacial acetic acid to reduce the initial pH of approximately 10 to 8.4.

The two bleaching solutions were then divided into 10 ml aliquots, in which individual 0.5 × 0.5 cm cotton samples from the three textiles were immersed. After initial agitation, the samples were left in individual sealed bottles for the duration of the bleaching (1 h). The samples were then removed and thoroughly rinsed in deionised water before being left to dry on acid-free blotting paper at room temperature.

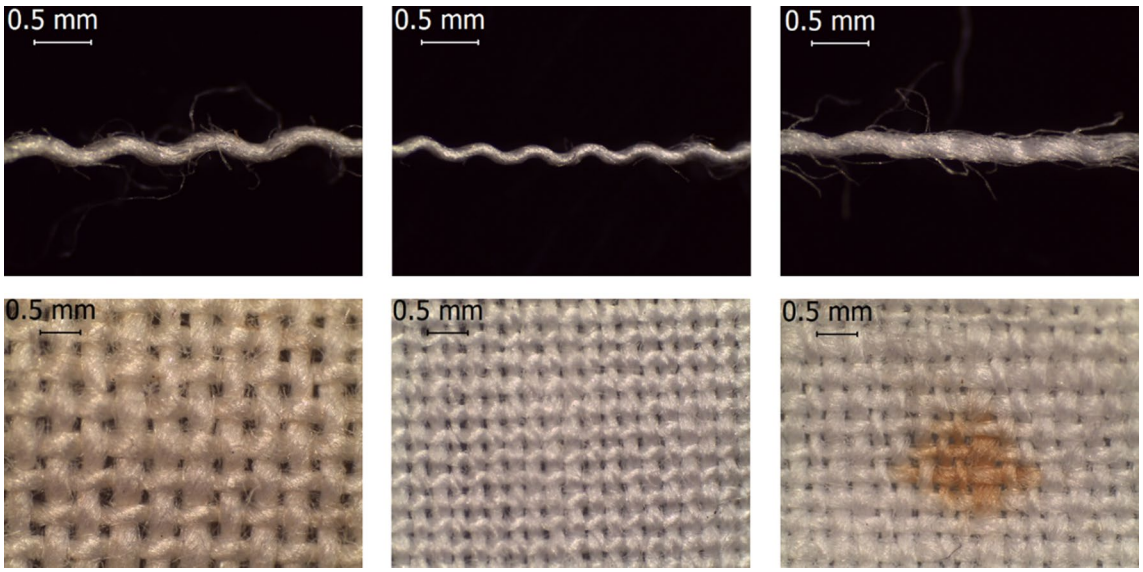

Fig. 4 Images of cotton threads and weave patterns recorded via a stereomicroscope; (*left*) raw cotton, (*middle*) scoured cotton, (*right*) historic cotton

Reductive bleaching

A 1% w/v $NaBH_4$ solution was prepared by adding 2.0072 g of $NaBH_4$ (98% powder, Fisher Scientific) in 200 ml deionised water. The production of H_2 bubbles, which occurred during the dissolution, was allowed to continue in the open beaker for approximately 1 week before use.

The $NaBH_4$ solution was divided into 10 ml aliquots. A 0.5 × 0.5 cm cotton sample was added to each aliquot and bleaching allowed to occur for 15 min before the samples were removed and rinsed in deionised water. The samples were left to dry on acid-free blotting paper at room temperature.

Microscopy

AFM analysis was performed on single cotton fibres mounted onto magnetic specimen discs using double-sided tape. The atomic force microscope used was the Nanoscope IV/Dimension 3100 SPM (Veeco Metrology). Images were captured through Nanoscope IV software (version 6.121) using tapping-mode and a silicone (phosphorous doped) cantilever/tip (Model: RTESP, Bruker) to collect height, amplitude, and phase data. The scan speed was 0.5 Hz. For each sample, a 5 × 5 µm and/or 1 × 1 µm area was captured. Measurements were made at five separate locations that were randomly selected for every sample.

SEM imaging was performed on the EVOXVP SEM (Carl Zeiss SMT). Samples were sputter coated with gold.

Optical microscopy images were obtained using the EZ4 Stereomicroscope (Leica Microsystems). The microscope CCD camera was connected to a PC running the LAS EZ (Leica) software.

Results and discussion

Characterisation of cotton samples pre-bleaching

Optical images taken using the stereomicroscope are shown in Fig. 4. Table 1 provides the physical characteristics of the three cotton textiles under investigation. All the samples were white or off-white in colour and constructed using a plain weave. Typical staining on the historic sample is also shown in Fig. 4.

Table 1 Physical characteristics of the cotton textiles and threads

Fabric	Raw	Scoured	Historic
Colour	Off-white	White	White with staining
Weave	Plain	Plain	Plain
Yarn Twist	Z	Z	Z
Thickness (mm)	0.489	0.322	0.365
Weight (g m^{-2})	853.4	485.4	526.2
Sett (threads cm^{-1}) (weft × warp)[a]	24 × 24	35 × 39	35 × 36
Tex (g km^{-1})[a]	33.16	11.98	12.90
Cover factor (weft/warp)[a]	1.38/1.38	1.21/1.35	1.26/1.29

[a] Only one measurement was made of the small pieces of cloth from each of the textiles under investigation

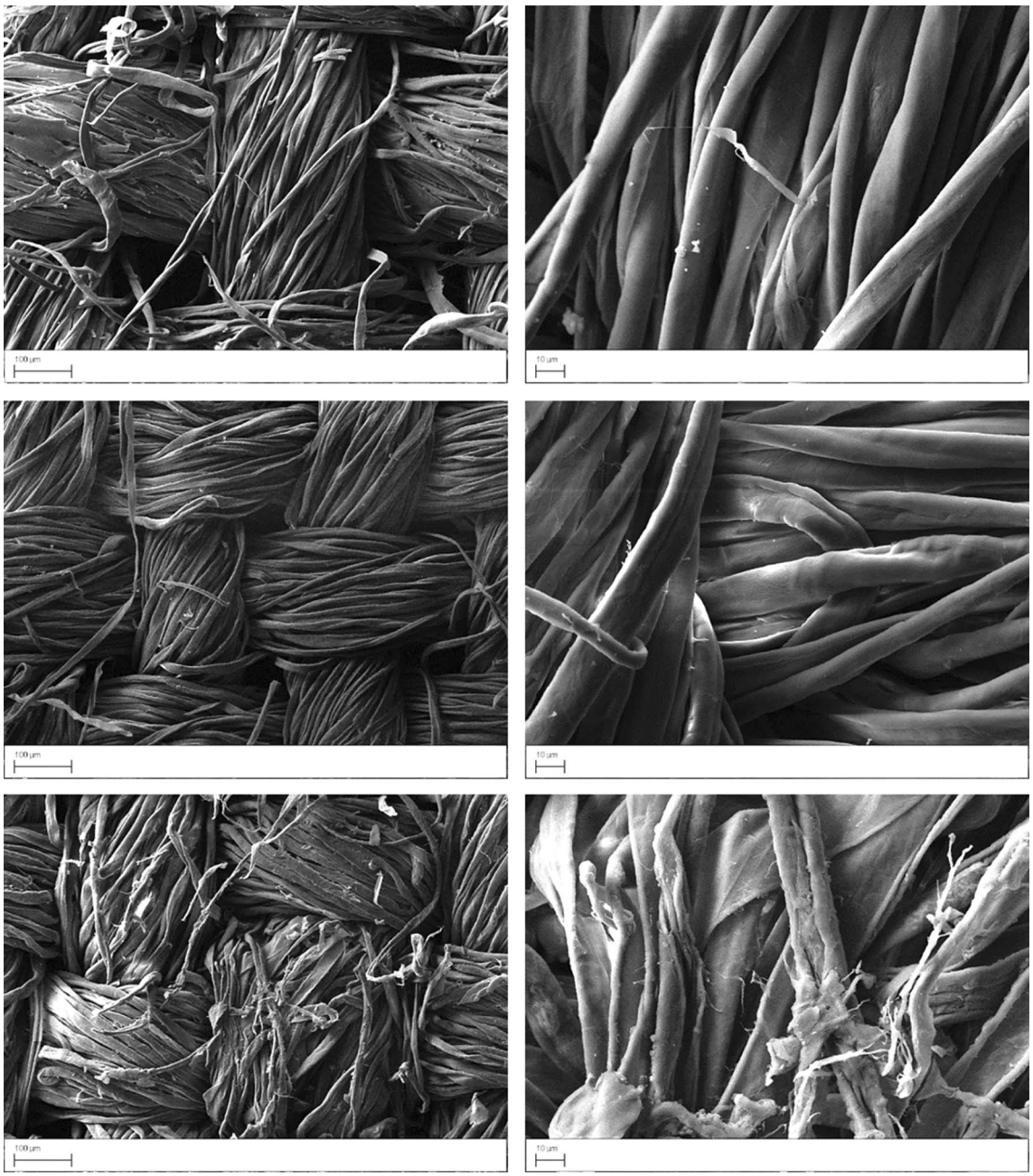

Fig. 5 Scanning electron micrographs using SE1 imaging of cotton samples pre-bleaching; (*top row*) raw cotton, (*middle row*) scoured cotton, (*bottom row*) historic cotton

The samples were imaged using SEM and AFM. The interpretation of images below is based on features described by Fig. 1 and text provided in the introduction to this paper.

Scanning electron micrographs of each sample pre-bleaching using secondary electron (SE1) imaging are shown in Fig. 5. There is a clear difference concerning the surface features between the three samples. The raw cotton shows a range of fibre conditions and the presence of residual materials scattered throughout. Some damaged and fused areas are seen however, it is not clear whether these are actual cotton fibres. The higher magnification image reveals the fibres to be relatively smooth which is characteristic of an intact cuticle layer. The SE1 images of scoured cotton show a much 'cleaner' sample mostly free from any particles. The higher magnification

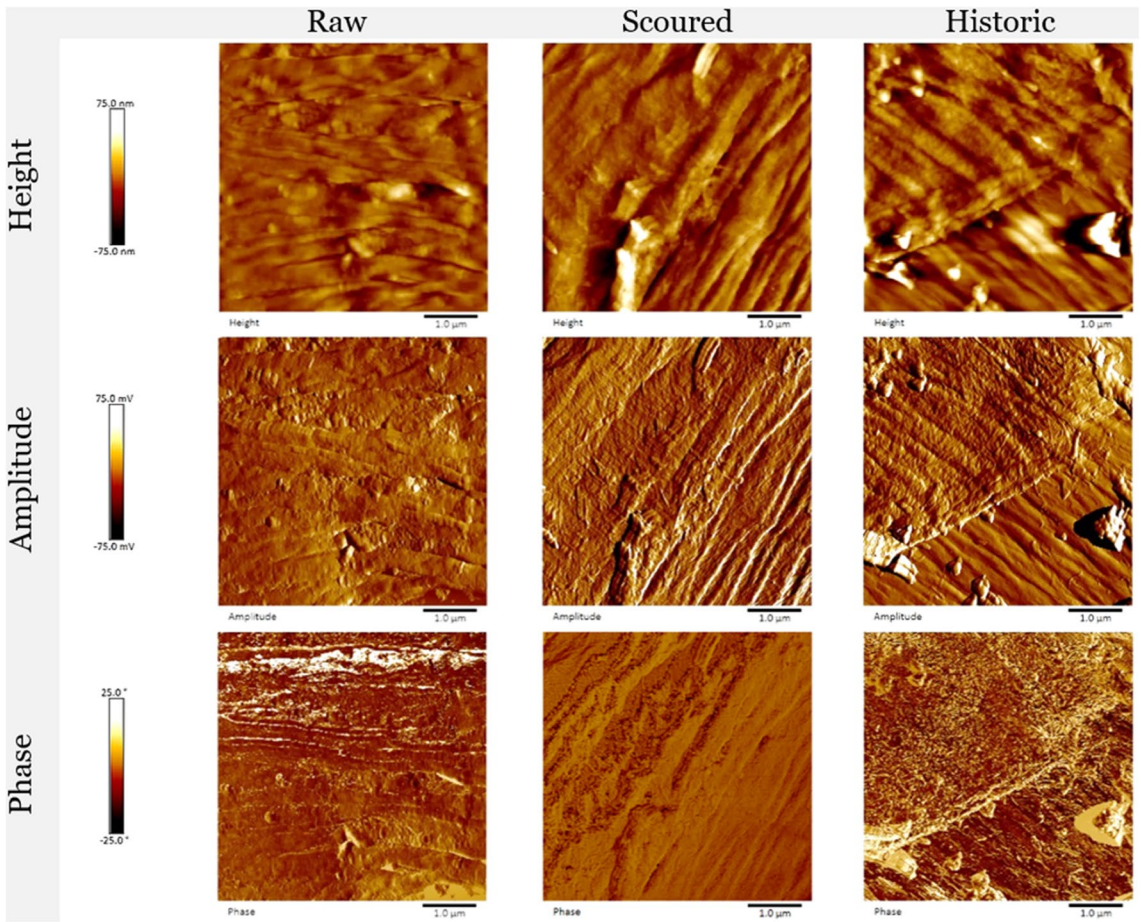

Fig. 6 AFM height, amplitude, and phase images of a 5 × 5 µm area of cotton samples pre-bleaching

image shows the impact of scouring, i.e., the outer cuticle layer has been removed with the primary wall and in some cases the transition layer being exposed. Finally, the historic cotton, through decades of ageing and use shows cotton fibres in various states. Many are torn, deformed, and stripped. The layers beyond the cuticle layer are easily seen (Additional file 1).

AFM images of a 5 × 5 µm area for each sample pre-bleaching are shown in Fig. 6. This is typical data of five randomly selected locations. A full set of images for all other sample locations are provided as supplementary data within this publication (see data in Additional file 1). The amplitude image from AFM shows how the tip is deflected as it encounters sample topography and often displays the shape of the features more clearly than height images. The phase image provides qualitative information of the viscoelastic properties of the sample, i.e., harder regions are identified by darker areas and softer regions by brighter areas of the image.

The height and amplitude image of the raw cotton shows the outer cuticle layer. There are cellulose fibrils up to 1 µm in width. The directions of these fibrils are various and constitutes the amorphous nature of the cuticle surface. The phase image indicates a soft to semi-hard surface.

Scoured cotton shows evidence of the primary wall being exposed. The fibrils are more tightly packed and organised along a directional axis. This is a semi-crystalline layer and hence the phase image shows a semi-hard surface. These observations are consistent with the fact that scouring is known to remove the outer cuticle layer.

AFM data for the historic cotton typically show primary and secondary wall components with particles attached to the surface. The nano-fibrils of the secondary wall are seen particularly well in the bottom right-hand corner of the amplitude image. The molecular arrangement of the wall is visible with nanofibrils typically 50 nm in diameter in compact alignment. The phase image indicates hard secondary walls and a soft winding layer. It is not possible with this data to establish precisely whether the secondary walls are S1 or S2.

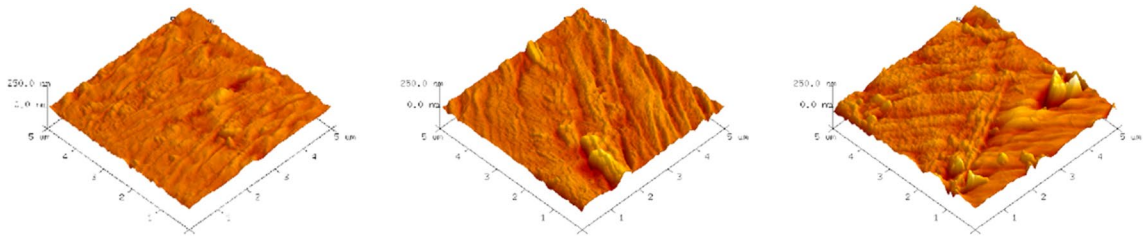

Fig. 7 3D representation of cotton fibre topography pre-bleaching; (*left*) raw cotton, (*middle*) scoured cotton, (*right*) historic cotton

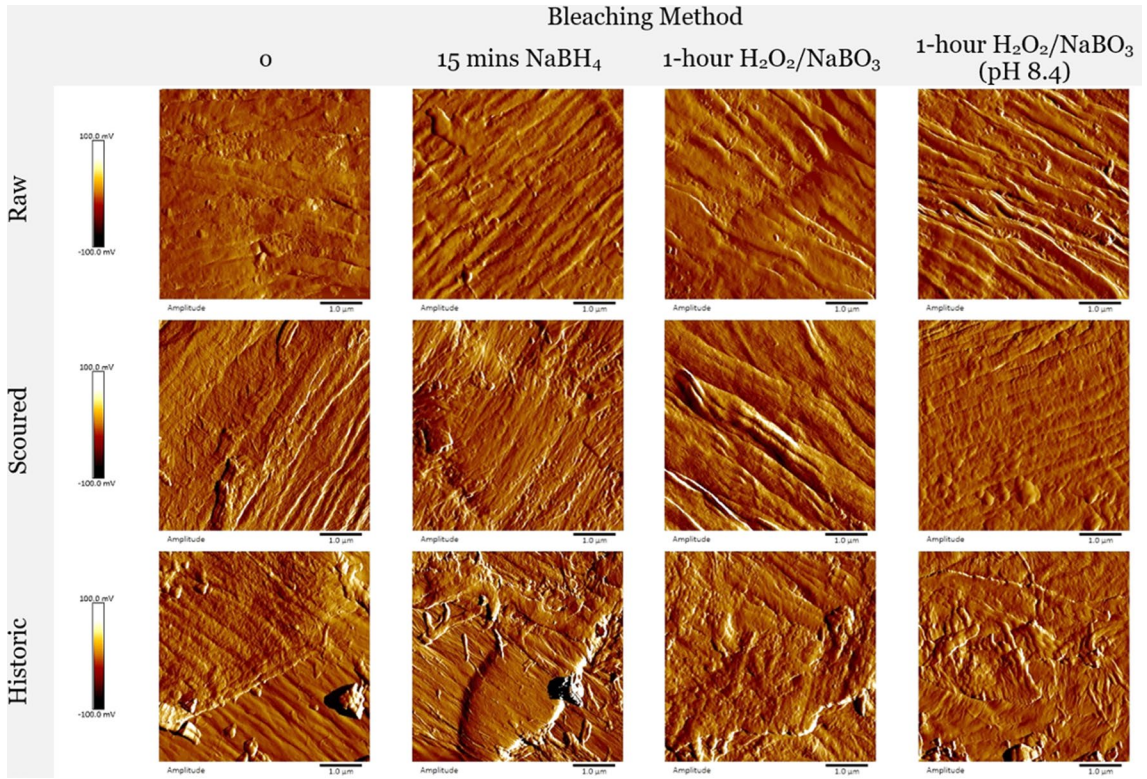

Fig. 8 AFM amplitude images of a 5 × 5 µm area of cotton samples pre- and post-bleaching. The bleaching conditions are typical of normal practice in textile conservation

A 3D representation of the height images of the cotton samples is shown in Fig. 7. The AFM data successfully demonstrates the various conditions of the fibre surface brought about by processes of scouring and ageing hereby justifying its use for the assessment of the bleaching process in conjunction with SEM and potentially other methods.

Characterisation of cotton samples post-bleaching

Representative amplitude images of the cotton samples before and after bleaching are compiled in Fig. 8. The raw cotton possesses an intact cuticle layer pre-bleaching however, it is removed using all the bleaching methods. This is very likely due to the alkalinity of the bleaching agents; typically, pH 10 and 10.5 for the $H_2O_2/NaBO_3$ and $NaBH_4$ solutions respectively. In other words, both bleaching and scouring have a very similar effect on the cuticle layer of the cotton fibre. The directional axis of the cellulose fibres in the primary wall structure can be seen in the bleached samples. The directional axis is a feature of cotton fibres previously observed in one study concerning the effect of mercerisation on directional fibre axis by Rollins et al. [32].

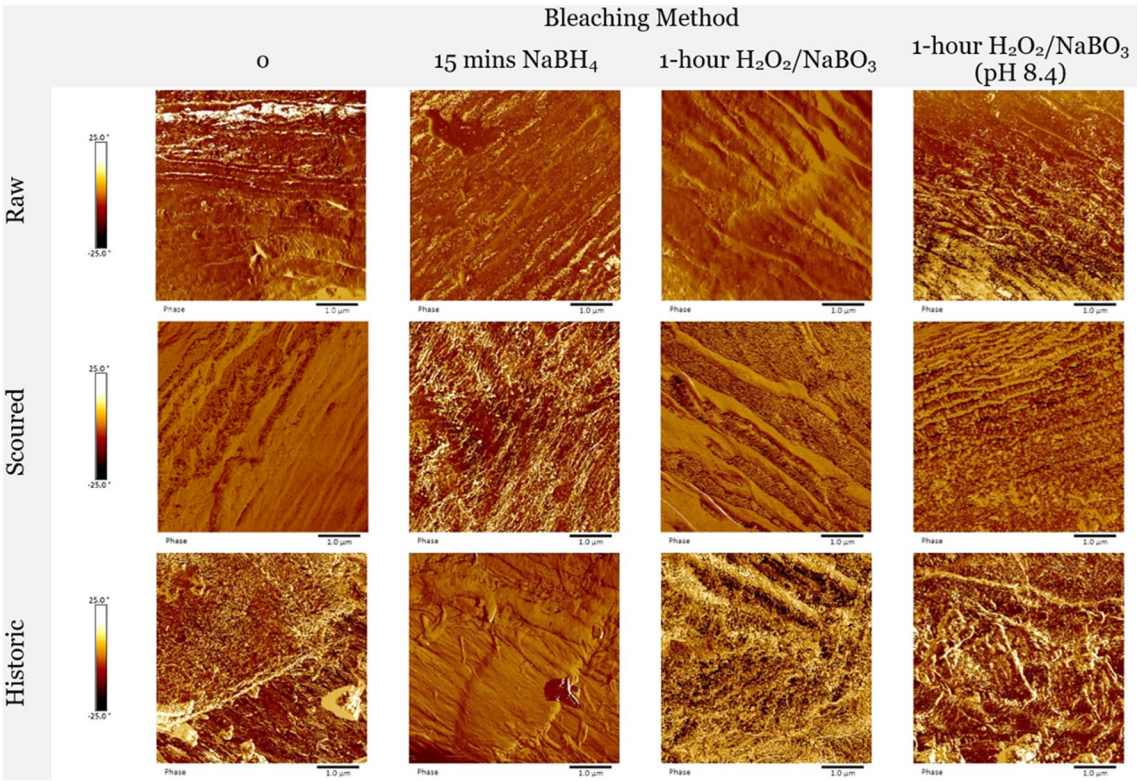

Fig. 9 AFM phase images of a 5 × 5 µm area of cotton samples pre- and post-bleaching. The bleaching conditions are typical of normal practice in textile conservation

The scoured cotton shows no cuticle layer which is removed during the scouring process. Further bleaching shows little to no major disruption of the surface topography. The historic cotton sample with its weak outer layer is further disrupted and removed. Using $NaBH_4$, it was observed that primary walls were removed to reveal the nano-fibrils of the secondary layer. The oxidative methods also have a stripping effect of the outer layers but to a lesser extent when compared to reductive bleaching with $NaBH_4$.

The corresponding phase images pre- and post-bleaching for Fig. 8 are shown in Fig. 9. Some generalised conclusions can be reached on the impact of bleaching on the elastic properties of the cellulose polymer. The images suggest that the bleaching process has several phases including swelling, delayering, softening, and stripping.

For raw cotton, bleaching with $NaBH_4$ has removed the relatively soft cuticle layer to reveal the more semi-crystalline primary wall. Also removed are the waxes and other non-cellulose materials associated with this layer. Oxidative bleaching of raw cotton has removed the cuticle layer but not to the same extent, leaving a surface which is softer as waxes and other materials may still be present. Similarly, the presence of waxes after bleaching using hydrogen peroxide (with a dinuclear manganese catalyst) is noted by Topalovic et al. [20].

The primary wall of the scoured cotton has been weakened by the $NaBH_4$ whereas oxidative bleaching has left the primary wall intact. Regarding historic cotton, this sample has had the primary wall essentially removed by bleaching with $NaBH_4$ to leave an exposed crystalline secondary layer likely to be harder compared to the outer layers. Oxidative bleaching has softened the outer layers of the historic cotton fibres but not removed them.

The raw cotton sample was the only one to show the cuticle layer whilst the historic cotton sample revealed primary and secondary wall components pre- and post-bleaching. Figure 10 collects 1 × 1 µm height, amplitude, and phase AFM data of raw and historic cotton samples showing these various layers.

The cuticle layer of the raw cotton is intact. The fibrils can be seen, and the arrangement of the cellulose is less organised and amorphous in nature. The phase image suggests that this layer is relatively hard. The height and amplitude images of historic cotton also show primary wall components with distinct directionality of the fibrils. The cellulose arrangement is amorphous and much softer compared to the cuticle layer. The secondary wall clearly

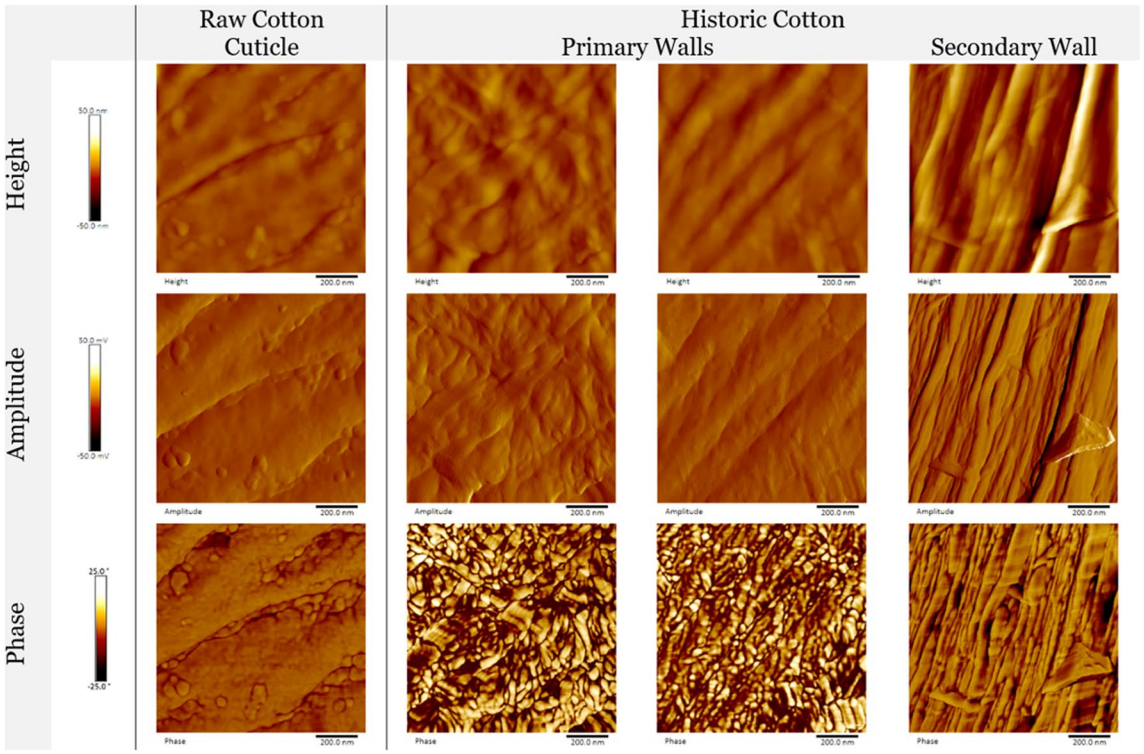

Fig. 10 AFM height, amplitude, and phase images of a 1 × 1 µm area of cotton samples post-bleaching showing cuticle, primary walls, and secondary walls

shows the nano-fibril alignment. The fibrils themselves are crystalline and relatively hard compared to the primary wall. The information from this data is consistent with the schematic diagram of the typical cotton fibre provided in Fig. 1.

Conclusion

The findings of this work using AFM imaging have illustrated that oxidative and reductive bleaching give rise to the stripping and rearrangement of the outermost layers in raw and scoured cotton fibres. Ultimately, the effect on raw and scoured cotton is lesser compared to the observations made when bleaching historic cotton fibres from the 100-year-old dress shirt. This is due to the fewer instances of past treatments, environmental exposure, and general wear that both the raw and scoured cotton have undergone compared to the shirt's lifespan. The outcome is therefore reduced physical and chemical integrity for the shirt. Additionally, the effect of reductive bleaching negatively impacted the historic cotton fibres to a greater degree compared to oxidative bleaching, and this does not fall in line with what has been generally accepted within the textile conservation community.

In this work we have effectively demonstrated the use of AFM to characterise the surface of cotton fibre materials pre- and post-bleaching. In doing so, we are able to add some insight into the impact of bleaching using standard and accepted practices in textile conservation. AFM will also allow the assessment of samples pre-bleaching to assess textile damage and inform a cleaning procedure appropriate to its condition.

The AFM can achieve imaging at higher resolution than SEM and experiments are performed at STP, i.e., the samples are in their natural environment. This technique can, when coupled with other techniques, help solve many unknowns of the cotton fibre structure.

Abbreviations

AFM: Atomic force microscopy; SEM: Scanning electron microscopy; $NaBH_4$: Sodium borohydride; H_2O_2: Hydrogen peroxide; $NaBO_3$: Sodium borate; Cu(II): Copper; H_2: Hydrogen.

Supplementary Information

The online version contains supplementary material available at https://doi.org/10.1186/s40494-022-00830-2.

Additional file 1: Duplication sets of AFM data of all samples pre- and post-bleaching. Table A.1. AFM data for raw cotton - 5 × 5 µm - Pre-bleaching. Table A.2. AFM data for raw cotton - 5 × 5 µm - 15 minutes of reductive bleaching ($NaBH_4$). Table A.3. AFM data for raw cotton - 5 × 5 µm - 1 hour of oxidative bleaching ($H_2O_2/NaBO_3$). Table A.4. AFM data for raw cotton - 5 × 5 µm - 1 hour of oxidative bleaching ($H_2O_2/NaBO_3$ (pH

8.4)). Table B.1. AFM data for scoured cotton - 5 × 5 µm - Pre-bleaching. Table B.2. AFM data for scoured cotton - 5 × 5 µm - 15 minutes of reductive bleaching ($NaBH_4$). Table B.3. AFM data for scoured cotton - 5 × 5 µm - 1 hour of oxidative bleaching ($H_2O_2/NaBO_3$). Table B.4. AFM data for scoured cotton - 5 × 5 µm - 1 hour of oxidative bleaching ($H_2O_2/NaBO_3$ (pH 8.4)). Table C.1. AFM data for historic cotton (circa. 1920) - 5 × 5 µm - Pre-bleaching. Table C.2. AFM data for historic cotton (circa. 1920) - 5 × 5 µm - 15 minutes of reductive bleaching ($NaBH_4$). Table C.3. AFM data for historic cotton (circa. 1920) - 5 × 5 µm - 1 hour of oxidative bleaching ($H_2O_2/NaBO_3$). Table C.4. AFM data for historic cotton (circa. 1920) - 5 × 5 µm - 1 hour of oxidative bleaching ($H_2O_2/NaBO_3$ (pH 8.4)).

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Author contributions
MU and KT designed the project. RTAS carried out all the practical work and wrote the initial draft of this paper. All authors read and approved the final manuscript.

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Availability of data and materials
The datasets used and/or analysed during the current study are available from the corresponding author on reasonable request.

Declarations

Competing interests
The authors declare no competing interests.

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