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Consolidation of Black-dyed Māori Textile Artefacts: Evaluating the Efficacy of Sodium Alginate

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ABSTRACT

Black-dyed artefacts are found in museums worldwide, many produced using an iron-tannate compound. Deterioration of iron-tannate dyed artefacts is an international preservation issue: in New Zealand the deterioration of paru (iron-tannate) dyed Māori textiles is widespread. This article reports experimental work testing the efficacy of sodium alginate, a consolidant developed for deteriorated paru-dyed muka (fibre from harakeke; *Phormium tenax*). The colour stability, strength retention, and acidity of paru-dyed muka consolidated with sodium alginate (0.25, 0.5, and 1% w/v in water) was tested pre- and post-artificial light ageing. This study found that sodium alginate had no negative effect on paru-dyed muka and in some cases provided benefit. Interestingly, the colour of paru-dyed muka is substantially more stable in UV-filtered light than previously recognised. Also microfading results were in agreement with visual assessments of colour change at 1 Mlux hour exposure, providing confidence in this relatively new technique to assess colour change.

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Microfading; consolidants; iron-tannate dye; tensile-testing; New Zealand flax; Phormium tenax; pH; accelerated light ageing

Introduction

Iron-tannate dyes have been used on a variety of cultural materials, and their potentially negative impact on both cellulosic and proteinaceous substrates has long been recognised. Most commonly, such dyes are produced by combining a plant polyphenol (tannate; which can be derived from a range of sources) with iron salts to create a dye complex resulting in the colour black (Hofenk de Graaff, Roelofs, and Bommel 2004). Black-dyed textiles coloured in this manner often show degradation via fibre embrittlement, increased acidity and loss of physical integrity (Daniels 1999a; Te Kanawa et al. 2002).

Black, paru-dyed Māori textiles represent one such group of artefacts for which conservation treatments are urgently needed. Māori textiles are represented in significant numbers in institutional collections worldwide, with conservators and other kaitiaki (guardians) recognising the need for solutions to prevent the further degradation of such important artefacts. Despite best-practice preventive conservation in cultural institutions, Māori textiles commonly show active deterioration of their paru-dyed components, such as weakening of yarn elements in piupiu (skirts), and the detachment of decorative tassels and structural failure of tāniko borders on kākahu (cloaks, Daniels 1996, 1999a; Tamarapa 2011) (Figure 1). In severe cases, paru-dyed Māori textiles become highly fragmented and lose their physical integrity completely.

The production of iron tannate-dyed Māori textiles

Of the three main dye colours used in customary Māori textiles (*mangu* black, *kōwhai* yellow, and *tanekaha* tan/brown), iron-tannate dyes used to produce black coloured fibre were the most common, and are still in widespread use (Te Kanawa 2001). The tannin component of the dye comes from either the bark of hinau (*Elaeocarpus dentatus*) or a combination of manuka (*Leptospermum scoparium*) and kanuka (*Kunzea ericoides*). Hinau bark extract is a hydrolysable polyphenol (gallotannin) that produces a blue-black hue, whereas manuka and kanuka extracts are condensed tannins that produce a green-black hue (Te Kanawa 2001; Te Kanawa et al. 2002). Hydrolysable tannins are glucose esters of phenolic acids (gallic acid, ellagic acid) and when combined with iron ions form blue – black iron (III) tannate dye complexes (Wilson 2012). Condensed tannins (proanthocyanidins) are polymers of flavan-3-ol (catechin) monomers, and when com-

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Figure 1. Kākahu (cloak) showing detail of tāniko border with deteriorating tannate-dyed muka Ethno 53219, Tamaki Paenga Hira Auckland War Memorial Museum, AM53219).

bined with iron ions form green-black coloured dye complexes (Wilson 2012; Wilson, Carr, and Hacke 2012). Daniels (1999a) suggested that condensed tannins (i.e. manuka/kanuka in the case of Māori textiles) were less stable, and therefore identification of the plant polyphenol source in paru-dyed Māori textiles may be important for long-term preservation.

The production of paru-dyed Māori textiles is a two-stage process. The first involves immersion of the prepared fibre in a bath of the polyphenol (either hinau or manuka/kanuka). After soaking, removal of fibre from the bath and drying, the fibre is then placed in a solution of mud, or paru, which is a mordant providing the source of iron ions, resulting in black colouration. Aside from the plant phenol source having an impact on the stability of the resulting black dye (hydrolysable vs. condensed tannins), the specific paru used (and therefore natural variability in iron ion abundance, and consequently unbound ions remaining after the dye complex is formed) may also have an effect on the properties of the resulting dye.

The degradation of paru-dyed Māori textiles

Despite best-practice care in cultural institutions, paru-dyed components of harakeke artefacts are particularly prone to degradation, due to the properties of the fibre, and the tannins and iron ions used to dye them (Daniels 1999a; Smith et al. 2005). While at least 10 plant species endemic to New Zealand have been used by Māori to create textiles (McCallum and Carr 2012), harakeke (*Phormium tenax*) is considered the major textile plant species due to its ubiquity and the usefulness of its fibres (length, strength, colour/whiteness, ease of removal from the leaf). Such desirable properties lead to its use in a range of woven objects including piupiu and kākahu, objects that often include decorative components coloured with iron-tannate dyes (Tamarapa 2011). In comparison to other plant textile fibres, however, muka (fibre from harakeke) has a high hemicellulose content (30.1%, Daniels 1999a). The chemical formation processes of black dye and/or black ink from tannates also provides an insight into possible degradation mechanisms. Both iron gall ink and iron-tannate dyes are produced by combining extracts from plants (tannates) with an iron containing salt, such as iron (II) sulphate (Daniels 1999a; Krekel 1999). Gallic or tannic acid + iron (II) ions (from ferrous sulphate) produce both iron (III) gallo- or tannate complexes and sulphuric acid (H_2SO_4). Excess iron (II) ions react with atmospheric O_2 to produce hydrogen peroxide (H_2O_2) and hydroxyl radicals (OH, Sato and Okubayashi 2010). The high acidity of the resulting ink or dye, as well as metal catalysed deterioration mechanisms accelerate the deterioration of the substrate the dye has been applied to (Kolar et al. 2005). According to Daniels (1999a), the specific case of paru-dyed Māori textiles degradation is a combination of iron-catalysed oxidation and acid-catalysed hydrolysis related to the high hemicellulose content of harakeke. As fibres become acidic, more iron becomes available by splitting the iron-phenol (iron tannate-dye) complex. This released iron promotes further oxidation, and increases acidity, which deteriorates both the fibre and dye itself, causing it to change from black, to a brown hue.

Treatments

Fibre degradation caused by iron-tannate dyes has been widely recognised in the conservation literature. Previous research carried out both nationally and internationally has developed an understanding of the deterioration mechanisms of tannate dyed cultural materials (for example see Daniels 1996, 1988a, 1988b, 1999a, 1999b; Krekel 1999; Bulska and Wagner 2004; Hofenk de Graaff, Roelofs, and Bommel 2004; Wilson 2007, 2012). Considerable effort has also been directed into averting the effects of iron-tannate dyes in artefacts, with many conservation approaches to textiles originating from the extensive research focusing on iron gall ink corrosion in paper conservation (see for example Daniels 1999a; Barker 2002). The underlying premise is that iron-tannate (plant phenol) dyes are sufficiently similar to iron gall ink that it is therefore likely that treatments already developed for iron gall ink will

also be effective for iron-tannate dyes (Daniels 1999a). Other interventive treatments designed for textiles have trialled various consolidants, introduced to restore strength of fibres and physical integrity to artefacts (Daniels 1998b, 1999b; Te Kanawa 2001; More et al. 2003; Te Kanawa 2005; Te Kanawa et al. 2008; Sato and Okubayashi 2010; Sato, Okubayashi, and Sato 2011; Wilson 2012).

The only consolidative conservation treatment developed specifically for use on deteriorated paru-dyed muka is the seaweed-derived polysaccharide, sodium alginate ($NaC_6H_7O_6$; Te Kanawa 2005). Its development was the subject of a Master of Science research project carried out by Rangi Te Kanawa in 2005 (Te Kanawa 2005), details of which are also described in three publications (Te Kanawa 2001; Fenton et al. 2004; Te Kanawa 2005). Sodium alginate has been used in concentrations from 0.25 to 1% w/v in water by New Zealand and Australian conservators for treatment of deteriorating paru-dyed Māori textiles. Previous testing of the effects of sodium alginate on paru-dyed muka focussed on visual properties, and the increased physical performance of consolidated fibres. However the ageing characteristics of muka consolidated with sodium alginate (colour stability, strength retention, and change in pH), remained poorly understood. As the consolidant was already in use, despite no previous testing of its performance over time under display conditions, there was an urgent need to assess its long-term stability.

This study therefore aimed to understand the effects of sodium alginate consolidant on the properties of artificially aged muka dyed with paru. Most conservation treatments for iron-tannate dyed textiles have been tested on single fibre strands or model woven textiles to reduce experimental variables and more easily conform to standard test methods (Daniels, 1998b, 1999b; Wilson 2012; Wilson, Carr, and Hacke 2012). However, woven textiles have less relevance to a New Zealand context as Māori textile artefacts are not manufactured in this way (i.e. yarn and weave structure are not analogous). Therefore testing for this research took place on muka strands prepared using customary methods, to more accurately represent Māori textiles. To account for the variability inherent in plant fibres, fibre from a single cultivar of harakeke was tested. Similarly, several previous studies have attempted to reduce the variability in the dyeing process by creating a model substance (Te Kanawa et al. 2008; Sato and Okubayashi 2010; Wilson, Carr, and Hacke 2012). In the current research project actual paru and plant extracts (tannins and fibre) collected from New Zealand sources were used, first to provide a more accurate representation of Māori textile artefacts, and second to elucidate the potential effects of tannin type (hydrolysable versus condensed tannins) and paru source. Specifically, our study assessed the individual and combined effects of the concentration of sodium alginate consolidant, tannin and paru source, and accelerated light ageing in terms of consolidated muka (i) colour change, (ii) tensile properties, and (iii) acidity as measures of an effective consolidant.

Methods

A factorial design was used for the experiment (Reedy and Reedy 1992), with three factors, namely concentration of consolidant (four levels; 0, 0.25, 0.5, and 1% w/v in water), dye type (five levels; a non-dyed control plus four combinations of tannin and paru source) and accelerated light ageing (two levels; pre- and post-ageing). Harakeke fibre and dye materials were prepared specifically for the experiment. All harvesting of harakeke leaf, fibre extraction, and dyeing were carried out using customary methods (see Te Kanawa 1992 for details).

Sample collection

To account for the natural variability expected in muka harvested from harakeke bushes, the experiment was designed to obtain replicates from 20 separate bushes of a single cultivar of harakeke. In April 2015, a single leaf was harvested from 20 individual harakeke bushes ('Ngutunui' cultivar, Scheele 2005) at Te Kuiti, Waikato, North Island, New Zealand (38.3350 °S, 175.1650 °E). Each leaf represented the fourth youngest leaf of a randomly selected leaf fan. Leaves were transported to the National Museum of New Zealand Te Papa Tongarewa (Te Papa) and stored with cut ends in tap water for 21 days. Hinau, Manuka, and kanuka bark, and paru (iron-rich mud; Paru 1) were obtained from Te Kuiti, to provide tannin and mordant sources, respectively. A second paru source (Paru 2) was collected from Kaikohe, Northland, North Island, New Zealand (35.2427 °S, 173.4759 °E).

Sample processing

Muka (fibre) was extracted from each leaf, and was not washed or beaten prior to being dyed. Muka from each leaf was divided into 20 groups of strands and each group randomly assigned to one of the 20 combinations of dye type (tannin-paru complex: Hinau-Paru1, Hinau-Paru2, Manuka-Paru1, Manuka-Paru2, non-dyed control) and concentration of sodium alginate consolidant (0.25, 0.5, or 1% w/v in water, non-consolidated control) (5 dye types × 4 consolidant concentrations = 20 combinations). 'Manuka' dye types consisted of tannins sourced from both manuka and kanuka bark, whereas 'Hinau' dye were comprised of tannins derived from this plant species only. The

groups of muka were soaked overnight in the tannin solutions, removed to dry, and then rubbed with paru. The muka remained in the paru overnight, then was rinsed with tap water the following morning and dried in natural diffuse light.

Dried sodium alginate was vigorously mixed in distilled water to obtain 0.25, 0.5, and 1% w/v concentrations, which were then applied using a paintbrush directly to muka as consistently as possible and then allowed to dry. Dyed muka was then packaged in acid-free tissue, and transported to the Centre for Materials Science and Technology Research Laboratory climate-controlled facilities (20 ± 2°C, 60 ± 4% RH) for testing.

Colour change

Colour change caused by accelerated light ageing and in relation to the concentration of the consolidant and each dye type were assessed using visual assessment (Blue Wool Standards, British Standards Institution 1993) and microfading techniques (Lowe *et al*., unpublished; Ford and Smith 2010; Ford and Druzik 2013).

Visual assessments – To enable visual assessment of the colour change of muka, fibres were mounted onto specimen cards together with ISO Blue Wool Standards 1–4 (International Organisation for Standardisation 1994) (115 × 50 × 2 mm; acid-free card; Supplementary Figure S1). Two cards were prepared for each dye treatment, with muka consolidated with each concentration mounted in a random order on the first four sample blocks. Each 10 mm wide strip of muka on the specimen cards consisted of 20 strands (one replicate from each harakeke bush) from each dye type. ISO Blue Wool Standards 1–4 were mounted in order in the second four sample blocks on each specimen card. A single or double slot mask was then placed over each card to restrict the region exposed to light when inserted into the Microsol 495 Light Fastness Machine for artificial light ageing. The use of two cards of each dye treatment in combination with the slot masks enabled four levels of exposure to light (0, 0.24, 1, and 10 Mlux) to be assessed simultaneously, without the need for a repeated measures experimental design (Reedy and Reedy 1992).

Specimen cards were artificially light-aged using a modified Microsol 495 Light Fastness Machine (mercury vapour lamp; 400 W, 19500 lumen/100 h) operated in a climate chamber, following standard test methods (British Standards Institution 1990, 2013). Modifications to the Microsol 495 included new purpose built specimen holders and the replacement of the water cooling system with fan-cooling of each specimen holder, which then enabled temperature and relative humidity control to occur via climate chamber settings, rather than be controlled using soluble salt solutions. Specimen holders were fitted with UV-filter glass (97% of 300–380 nm radiation blocked) (Figure 2). Pre-testing of these equipment modifications confirmed temperature and relative humidity could be controlled as desired at the fibre surface during operation of the machine. Illumination levels (mean 46,000 lux), temperature, and relative humidity were monitored during light ageing with an Elsec 764 lux metre mounted in one of the specimen holder positions. Temperature (20 ± 2°C) and RH (52 ± 2%) simulated current Te Papa display conditions (95% of display time; winter–summer 20–22 ± 1°C, 52 ± 7% RH, UV filtered). Specimen cards were exposed to 0.24, 1, or 10 Mlux hours, corresponding to 10, 41.7, or 417 years maximum display time for sensitive category artefacts (Te Papa lighting guidelines; 50 lux for 13.3 hours per day, maximum accumulated exposure 0.24 Mlux hours per 10 years). Colour change was visually assessed relative to the standard ISO Grey Scale (British Standards Institution 1990, 1993; International Organisation for Standardisation 1994) by a panel of seven judges and reported in terms of the Grey Scale number considered equivalent to one just noticeable fade (1 JNF).

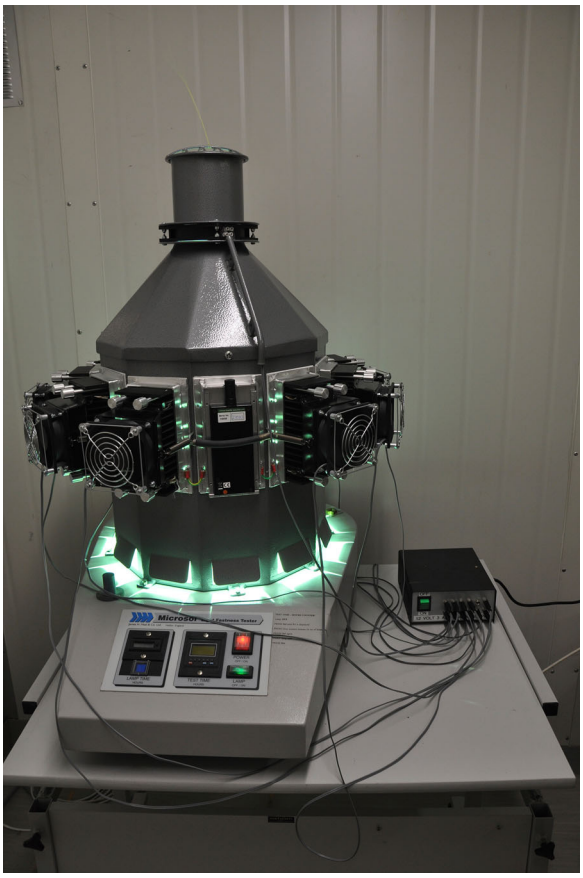

Figure 2. Accelerated light-ageing of muka specimens using a modified Microsol 495 fading machine operating in a climate chamber set to 20°C and 52% relative humidity. Note data logger mounted to provide continuous lux, temperature, and relative humidity readings at specimen holder positions.

Microfading – To quantify the colour change of muka related to dye type and the concentration of consolidation using microfading, 60 muka strands

(3 randomly selected replicates from each of the 20 dye × consolidant combinations) were each folded in half twice to form a single fibre bundle, before being mounted on a black stereo microscope stage plate (100 mm diameter). Blue Wool Standards 1–4 (British Standards Institution 1990) reference samples (25 × 15 mm) were mounted on black matt card (75 × 25 mm), with preliminary analysis indicating that fade resistance was not influenced by the background of the mounting medium.

Microfading was carried out over a period of three days in a draught-free laboratory (ambient environment mean ± sd; 21.7 ± 1.6°C, 827.3 ± 217.6 lux, 13.2 ± 2.6 UV, 41.2 ± 1.3% RH). Each fibre bundle was randomly allocated to a test day, with a maximum of 20 bundles assessed per day, in addition to daily sampling of Blue Wool Standards 1–4 for calibration (International Organisation for Standardisation 2014). All samples were microfaded at 6 Mlux for 10 minutes (approximately 1 Mlux hour exposure) using a Newport Oriel Apex Fibre Illuminator fitted with a UV and IR filtered xenon source (400–700 nm) adjusted to fade a spot approximately 0.3–0.4 mm diameter. Spectral reflectance was measured using a Control Development Inc. spectrometer (298–1107 nm) and associated Spec32 software over a 400–700 nm bandwidth, with spectra saved to file once per minute during the 10 minute fading period. Colour change was calculated from spectra collected at the 0 and 10 minute time intervals using CIE *L*a*b** colour space and the CIELAB (ΔE_{76}) and CIEDE2000 (ΔE_{00}) colour difference formulas (CIE Technical Committee 3-22, 2004). ΔE was calculated using both CIELAB (ΔE_{76}, Equation 1) and CIEDE2000 (ΔE_{00}, Equation 2) formulas to enable comparison with published ΔE_{76} data whilst also reporting results according to the current industry standard for reporting colour change:

$$\Delta E_{76} = \sqrt{(\Delta L*)^2 + (\Delta a*)^2 + (\Delta b*)^2} = \sqrt{(L_2^* - L_1^*)^2 + (a_2^* - a_1^*)^2 + (b_2^* - b_1^*)^2} \quad (1)$$

$$\Delta E_{00} = \sqrt{\left(\frac{\Delta L'}{k_L S_L}\right)^2 + \left(\frac{\Delta C'}{k_C S_C}\right)^2 + \left(\frac{\Delta H'}{k_H S_H}\right)^2 + R_T\left(\frac{\Delta C'}{k_C S_C}\right)\left(\frac{\Delta L'}{k_L S_L}\right)} \quad (2)$$

whereby, ΔL^*, Δa^*, and Δb^* represent the differences in lightness, chromaticity on a red (positive) to green (negative) scale, and chromaticity on a yellow (positive) to blue (negative) scale between two colour samples for ΔE_{76} (Robertson 1977). ΔE_{00} includes the CIELAB metrics for differences in lightness ($\Delta L'$), chroma ($\Delta C'$), and hue ($\Delta H'$) and their respective weighting functions S_L, S_C, and S_H and adjustment factors k_L, k_c, and k_h related to viewing conditions such as texture etc. R_T is a term accounting for interaction between hue and chroma differences (Luo, Cui, and Rigg 2001; Sharma, Wu, and Dalal 2005). Calculations were based on Boronkay (2008) CIE conversions developed from Sharma, Wu, and Dalal 2005, and were performed using Visual Basic routines (© Bruce Ford and Dougald Knuckey, 2014) within Microsoft Excel v14 2010.

Tensile properties

The effects of consolidant concentration and dye type on muka tensile properties were assessed pre- and post-accelerated light ageing using a multiple factor experiment. For each of the 20 harakeke leaves, two single muka strands were selected randomly from each of the 20 groups of previously prepared muka strands (4 concentrations of consolidant × 5 dye types) and assigned to either a pre- or post-ageing treatment. This process gave 20 replicate muka strands per combination of consolidant × dye type × ageing factor giving a total of 800 strands tested, with the 20 replicates representing the inherent variability of muka obtained from different bushes. Each strand was measured for length (mm) and mounted on specimen holders that enabled the centre 100 mm strand portion to be exposed to light-ageing treatments. Strands were light-aged using the Microsol 495 Light Fastness Machine (0, 10 Mlux hours), with climate parameters reflecting Te Papa display conditions (as per above).

After light ageing, muka strands were removed from the specimen holders, conditioned for two weeks (20 ± 2°C, 52 ± 4% RH) before tensile testing the light-aged portion of each muka strand with an Instron 4464 Universal Testing Machine fitted with a 100N load cell (accuracy ± 0.5%). Each muka strand was individually mounted in the Instron clamps with the light-aged portion corresponding to the 100 mm gauge length. A 20 g weight was hung from each strand prior to tightening the lower clamp to ensure a consistent gauge length and pre-load. Strands were extended at a constant rate of 10 mm/min until rupture. Each muka strand was weighed (mg; Mettler Toledo AT400 balance) to calculate the linear density (tex). Six tensile properties were calculated from the force-extension curves recorded for each strand; strength (tenacity N/tex), stiffness (modulus N/tex), extensibility (strain at maximum load %, strain to rupture %), and toughness (specific energy to maximum load mN/tex, specific energy to rupture mN/tex (Yang 2006; Morton and Hearle 2008). Each of these properties provides different information about the behaviour of fibre. Tenacity is a useful measure for understanding how much weight a fibre can hold (for example if a kākahu (cloak) is displayed by mounting on a mannequin, the muka it is made from will be experiencing tensile loads due to the weight of the kākahu). Modulus is a measure of stiffness in tension and

describes the ability of the fibre to resist extension under a tensile load. A stiff fibre (like muka) takes a large amount of tensile force to extend, and will not extend very far before breaking. Modulus is a useful measure for understanding how well a fibre resists permanent stretching (for example if a kākahu is mounted on a mannequin, the muka it is made from may stretch. A decrease in stiffness would result in greater danger of stretching). Extensibility is a measure of how far a fibre can be stretched under tension, and is expressed as strain (the change in fibre length divided by the original length). Two measures of extensibility have been used here, strain to maximum and strain to rupture. Strain to maximum describes how far the fibre can be extended until the moment it begins to break or fray, i.e. permanent damage due to stretching begins to occur. Strain to rupture describes how far the fibre can be extended until it finally breaks. Muka has a relatively low extensibility compared with other fibres. A decrease in extensibility would result in an artefact being unable to accommodate as much stretching before permanent damage or catastrophic failure occurred. Toughness is a measure of brittleness. A tough fibre is able to accommodate large tensile loads and large tensile strains simultaneously. Two measures of toughness have been used here, specific energy at maximum load and at rupture. Specific energy at maximum load describes how tough the fibre is up until the moment it begins to break or fray, i.e. permanent damage begins to occur. Specific energy to rupture describes how tough the fibre is up until it finally breaks. Muka has a relatively low toughness compared with other fibres. A decrease in toughness would result in an artefact being unable to accommodate sudden shocks without permanent or catastrophic damage.

The full range of tensile properties were calculated from the data in order to understand the ways in which dyed muka properties were altered after use of a consolidant and post-ageing. For example an increase in stiffness alone could indicate the fibre is becoming embrittled. However if toughness simultaneously increases the opposite is true and the consolidant is performing well. Calculating extensibility and toughness both at maximum load and rupture also provides a measure of how well a consolidant is performing in relation to fraying of muka under load. If there is not much difference between the values at maximum and rupture this indicates all fibres within the muka strand have become weakened, whilst a large difference would indicate some portions of the muka strand have retained strength and elasticity.

Acidity

The micro-extraction method (Vuori and Tse 2004; Tse 2007) was carried out in a climate-controlled laboratory (20 ± 2°C, 65 ± 4% RH) to test the effects of consolidation and dye type on the acidity of muka, post-ageing. A 50 mm muka sample was obtained from the middle 100 mm region of three tensile tested muka strands from each consolidant/dye/age combination (total $n = 120$). Each sample was weighed (6–40 mg), cut into 2–4 mm lengths and placed into a 2 mL Eppendorf tube of distilled water (50 µL water/1 mg muka). Tubes were briefly agitated to ensure all samples were immersed, with the pH of the extracting solution assessed after 24 hours.

Data analysis

Statistical analyses were computed using R v. 3.1.0 (R Development Core Team 2014), with all data sets conforming to the normality and variance assumptions for parametric testing. Separate general linear models (GLMs) were used to test the individual and combined effects of experimental factors (concentration of consolidant, dye type, accelerated light ageing) on each muka tensile property (strength, stiffness, extensibility, toughness), colour change when microfaded, and acidity. The F statistic was computed for each test, with a probability level of $p = 0.05$ or less used to determine when $F_{a,b}$ indicated significant differences among the measured properties (Reedy, and Reedy 1988). The subscripts of the F statistic describe the degrees of freedom of the mean square ratio used for each F computation (refer to a standard statistical text for further detail) and thereby set the threshold which the value of F must exceed to meet the $p = 0.05$ level of probability. When significant differences were detected for either the main experimental factors or their interactions, Tukey's HSD *post hoc* tests (multcomp:glht, v.1.3-8; Hothorn, Bretz, and Westfall 2008) were used to determine where differences occurred, also using a a probability level of $p = 0.05$ or less. Only two-way interactions were tested (e.g. consolidant concentration × dye type etc.) as three-way interactions (i.e. consolidant concentration × dye type × ageing level) are difficult to interpret (Reedy and Reedy 1992).

Results

Under each sub-heading results are reported for non-dyed and paru-dyed muka (non-consolidated; 0.25%, 0.5%, and 1% w/v consolidated with sodium alginate) comparing pre- with post-light aged to assess the ageing characteristics (colour change, tensile properties, pH) of the consolidant after it was applied to dyed muka.

Colour change

Visual assessment – Consolidated muka was only observed to change colour post-accelerated light

ageing for one dye type, Manuka-Paru1, and only after 10 Mlux hours of exposure. The extent of colour change tended to be less at higher concentrations of consolidant, with muka treated with 0.25 and 0.5%w/v concentration showing colour change equivalent to 1 JNF (Ashley-Smith, Derbyshire, and Pretzel 2002; Grey Scale 4) after ageing, while the colour of muka treated with 1% v/w concentration of consolidant changed just less than 1 JNF (Grey Scale 4.5). In contrast, non-consolidated Manuka-Paru1 showed the greatest colour change (Grey Scale 3.5) (Figure 3). Relative to Blue Wool Standards, non-consolidated Manuka-Paru1 faded at a faster rate (between BW2 and BW3) than consolidated Manuka-Paru1 (between BW3 and BW4). Distinct colour changes were not detected for any other combinations of dye type, concentrations of consolidant, or light exposure.

Microfading-change in colour (measured in ΔE units) of non-dyed and paru-dyed muka was not affected by the application of sodium alginate, at any level of concentration, up to the equivalent of 1Mlux hours of exposure to light (calculated using either ΔE_{76} or ΔE_{00}; $GLM_{\Delta E76\ Consolidant}$, $F_{3,40} = 2.624$, $GLM_{\Delta E00\ Consolidant}$, $F_{3,40} = 2.394$, all $p > 0.05$) (Figure 4). The ΔE colour changes of all non-dyed and paru-dyed muka were less than ΔE changes measured for BW2, and were within the ΔE range for BW3 to BW4. Furthermore, no interactions between concentration of consolidant and dye type were detected ($GLM_{\Delta E76\ Consolidant \times Dye}$, $F_{12,40} = 1.070$, $GLM_{\Delta E00\ Consolidant \times Dye}$, $F_{12,40} = 1.235$, all $p > 0.05$). There appeared to be a difference among the dye types in the amount of colour change after microfading, with the non-dyed muka tending to show the greatest colour change (Figure 4). However this was only borderline significant when calculated using ΔE_{76} colour change ($GLM_{\Delta E76\ Dye}$, $F_{4,40} = 2.637$, $p = 0.0480$) and *post hoc* tests suggest those differences were weak (Tukey *post hoc*, all $p > 0.05$). The ΔE_{76} and ΔE_{00} colour changes of non-dyed and

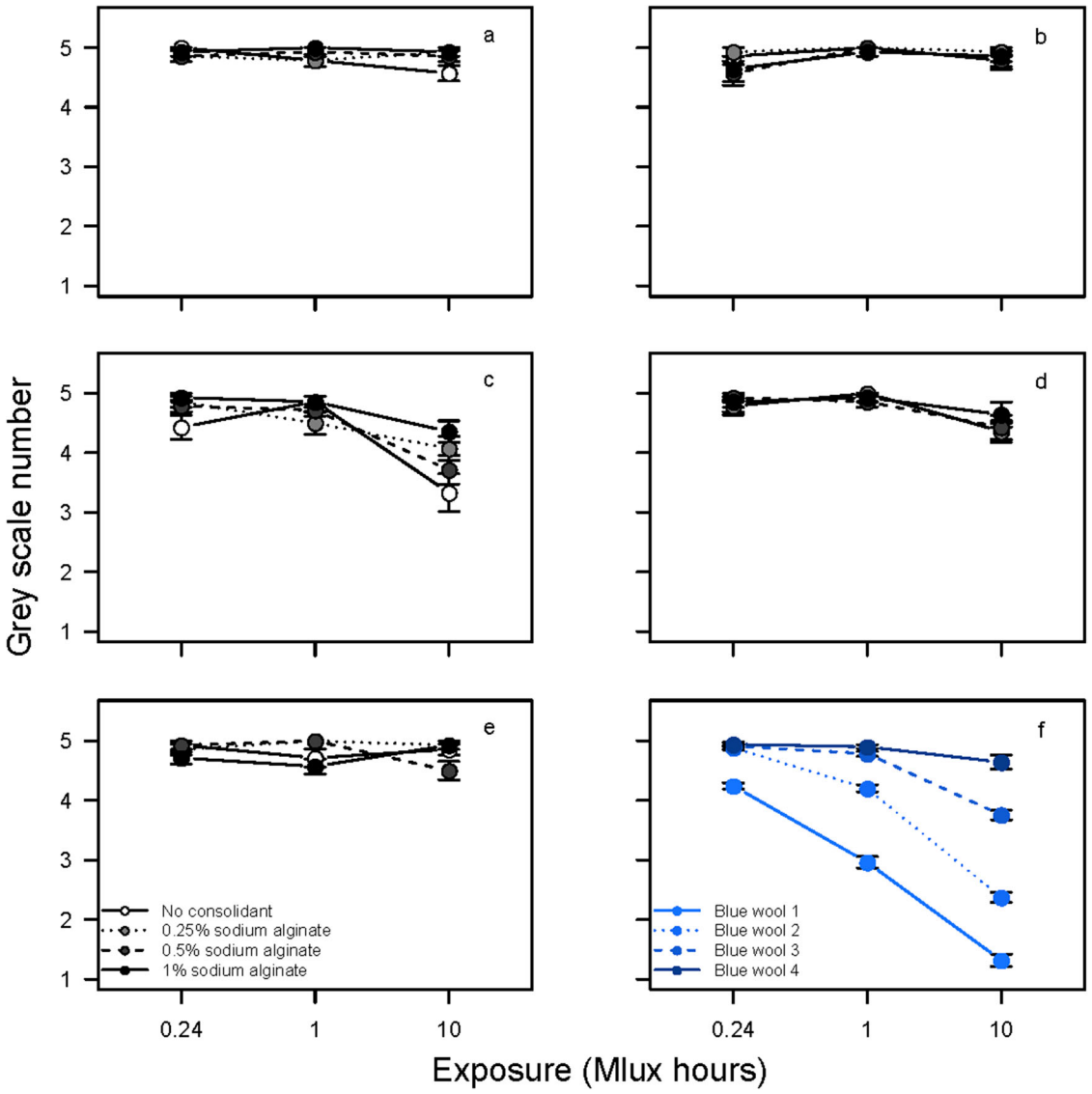

Figure 3. Visual analysis. The mean effects of concentration of sodium alginate consolidant and accelerated light ageing on muka colour change relative to the ISO Grey Scale for (a) Hinau-Paru1, (b) Hinau-Paru2, (c) Manuka-Paru1, (d) Manuka-Paru2, and (e) Non-dyed muka, in comparison to (f) ISO Blue Wool standards 1–4. Error bars indicate standard errors.

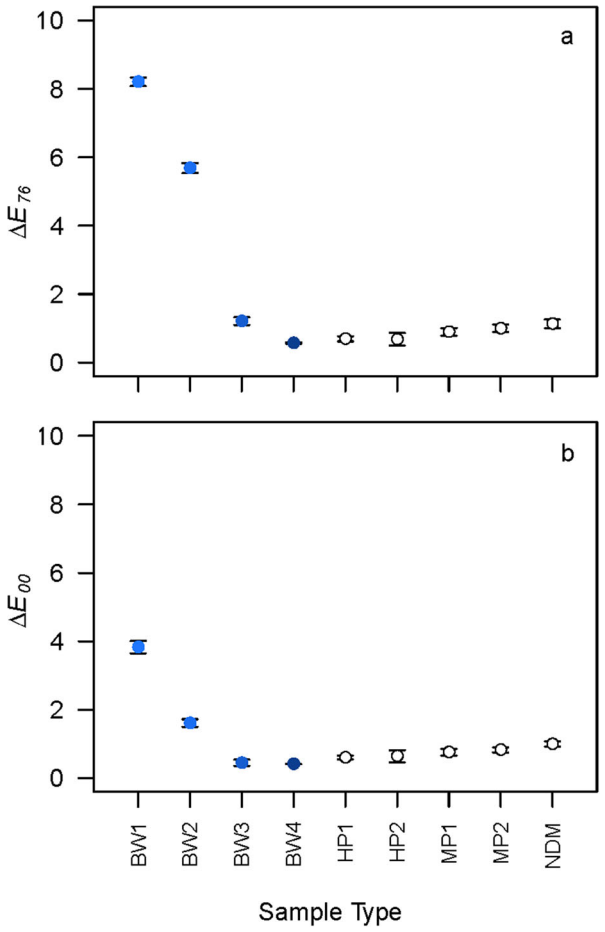

Figure 4. Mean colour change of muka calculated using (a) CIE 1976 (ΔE_{76}) and (b) CIE 2000 (ΔE_{00}) colour difference formulae in relation to ISO Blue Wool standards from microfading assessments. Abbreviations represent BW1–4 – Blue Wool 1–4, HP1 – Hinau-Paru1, HP2 – Hinau-Paru2, MP1 – Manuka-Paru1, MP2 – Manuka-Paru2, NDM – Non-dyed muka. Error bars indicate standard error.

paru-dyed muka were within the Δ*E* ranges for BW3 to BW4 respectively.

Tensile properties

Strength – application of the consolidant did not cause a detectable change in the strength (i.e. tenacity N/tex) of muka compared with untreated muka, for any of the concentrations, nor for any of the dye types, including non-dyed muka. There was also no detectable change in muka strength related to any of the concentration of consolidant post-ageing for 10 Mlux hours. However, light ageing did cause a detectable decrease in muka strength for each of the dye types, except for non-dyed muka for which there was no detectable change. This equates with findings in previous research where dyeing muka with paru, rather than ageing, has the greatest effect in diminishing the strength of fibres. The application of the consolidant does not have a significant effect in mitigating the immediately negative impact of dyeing muka black with paru.

Concentration of consolidant had a weak effect on muka strength (tenacity; GLM $_{Consolidant}$, $F_{3,750} = 3.268$, $p = 0.021$; Tukey *post hoc*, all $p > 0.05$), and did not interact with dye type or light ageing (Figure 5). *Post hoc* contrasts indicated the weak concentration effect was due to differences between 0.25 and 1% w/v concentrations of consolidant (Tukey *post hoc*, $p = 0.07$). Muka strength was influenced by interactions between dye type and ageing (GLM $_{Dye \times Age}$, $F_{4,750} = 3.268$, $p < 0.001$), with paru-dyed muka strength decreasing after ageing (Tukey *post hoc*, all $p < 0.001$), whilst non-dyed muka strength showed no change (Tukey *post hoc*, $p = 0.113$).

Stiffness – The application of consolidant had an influence on stiffness (modulus N/tex) for only some combinations of consolidant concentration and dye type, with a tendency to decrease stiffness of muka dyed with Paru1 (GLM $_{Consolidant \times Dye}$, $F_{12,750} = 2.594$, $p = 0.002$; Supplementary Figure S2). However there was no consistent pattern in relation to concentration of consolidant. For example, the application of 1% w/v consolidant to Manuka-Paru1 dyed muka reduced stiffness in comparison to the 0.25 and 0.5% w/v consolidant only, whereas the application of 0.5% w/v consolidant to Hinau-Paru1 reduced stiffness in relation to the 0.25% w/v and non-consolidated muka. Non-dyed muka showed inconsistent effects of increasing concentration of the consolidant, with the application of 0.25% w/v consolidant increasing stiffness of muka in comparison to 1% w/v and non-consolidated muka. The stiffness of Hinau-Paru2 and Manuka-Paru2 dyed muka were not influenced by the concentration of the consolidant.

Ageing however, showed a more consistent effect in relation to consolidant concentration, with a decrease in stiffness of muka consolidated at the two highest concentrations (0.5 and 1% w/v) after ageing for 10 Mlux hours, compared with non-consolidated fibres. Ageing also had an overall effect on the stiffness of muka that was different amongst the dye types. Significant interactions between concentration of consolidant and light ageing (GLM $_{Consolidant \times Age}$, $F_{3,750} = 4.569$, $p = 0.004$) indicated that artificial light ageing decreased the stiffness of muka treated with 0.5 or 1% w/v concentrations of consolidant, whereas 0.25% w/v consolidant and non-consolidated muka showed no change. Muka stiffness was also influenced by interactions between dye type and light ageing (GLM $_{Dye \times Age}$, $F_{4,750} = 8.221$, $p < 0.001$) whether consolidated or not, with ageing reducing hinau-dyed muka stiffness, increasing non-dyed muka stiffness, and having no effect on manuka-dyed muka. Therefore, muka dyed with hinau based dyes showed a decrease in stiffness, non-dyed muka showed an increase and muka dyed with manuka based dyes showed no detectable difference.

Extensibility – The application of the consolidant did not alter the extensibility (i.e. strain % at maximum load and rupture) of any of the muka, either pre- or

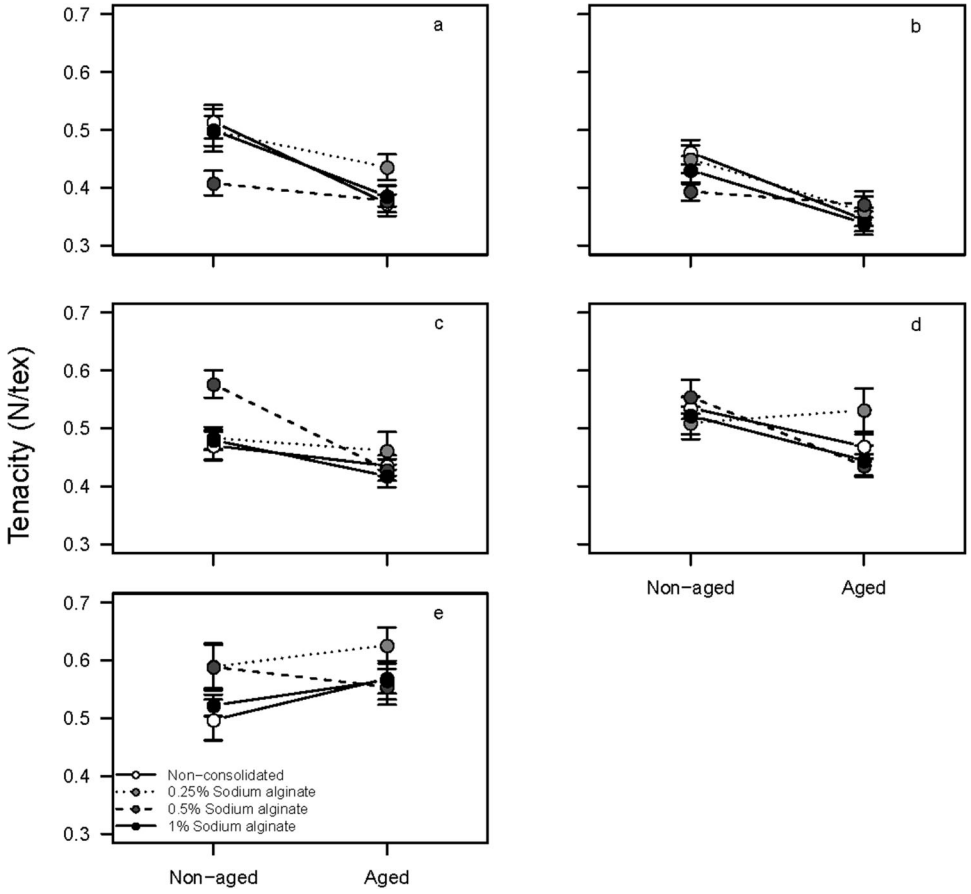

Figure 5. Mean effects of sodium alginate concentration and accelerated light ageing (10 Mlux hours) on the strength (tenacity (N/tex)) of muka dyed with (a) Hinau-Paru1, (b) Hinau-Paru2, (c) Manuka-Paru1, (d) Manuka-Paru2, or (e) Non-dyed. Error bars indicate standard error.

post-ageing. Concentration of consolidant alone, or in combination with dye type or light ageing, had no influence on muka extensibility, in terms of strain at maximum load or strain at rupture (all $p > 0.05$; Supplementary Figures. S3–S4). Muka extensibility was influenced by dye type alone ($GLM_{StrainMax\ Dye}$, $F_{4,750} = 20.232$, $p < 0.0001$), with Hinau-Paru2 having a significantly lower strain at maximum load than other dye types. Light ageing also reduced the strain at maximum load ($GLM_{StrainMax\ Age}$, $F_{1,750} = 55.902$, $p < 0.0001$).

However, ageing reduced the extensibility of muka in terms of strain to rupture in all cases, showing influences of interactions between dye type and light ageing ($GLM_{StrainRupture\ Dye \times Age}$, $F_{4,750} = 2.903$, $p < 0.021$), with light ageing decreasing the strain to rupture for paru-dyed muka (Tukey *post hoc*, all $p < 0.01$), except for non-dyed muka which was unaffected by ageing. No change in strain to rupture with light ageing was noted for non-dyed muka.

There were also differences in extensibility among muka dyed with the different dye types. Hinau-Paru1 dyed muka was the least extensible at maximum load. Again, results accord with previous research findings that application of paru dye to muka has the principal effect on the physical properties of the fibres, rather than age.

Toughness – The application of the consolidant, or in combination with dye type or light ageing, did not alter the toughness (i.e. specific energy N/tex at maximum load and rupture) of any of the muka, either pre- or post-ageing (all $p > 0.05$; Supplementary Figures. S5–S6). However, ageing reduced the toughness of all but the non-dyed muka. Both muka toughness measures were influenced by interactions between dye type and artificial light ageing ($GLM_{Max\ Dye \times Age}$, $F_{4,750} = 4.633$, $p = 0.001$; $GLM_{Rupture\ Dye \times Age}$, $F_{4,750} = 5.274$, $p = 0.0003$), with ageing decreasing toughness of all paru-dyed muka, and having no influence non-dyed muka toughness.

Acidity

In general, the application of the consolidant had no effect on the acidity of non-dyed muka (which were also initially considerably less acidic than the paru-dyed muka, although showing a tendency to become more acidic post-ageing). While the concentration of the consolidant did not influence the acidity of non-dyed muka, the acidity of paru-dyed muka decreased with the application of certain concentrations of the consolidant, as indicated by significant context dependent interactions between concentration of

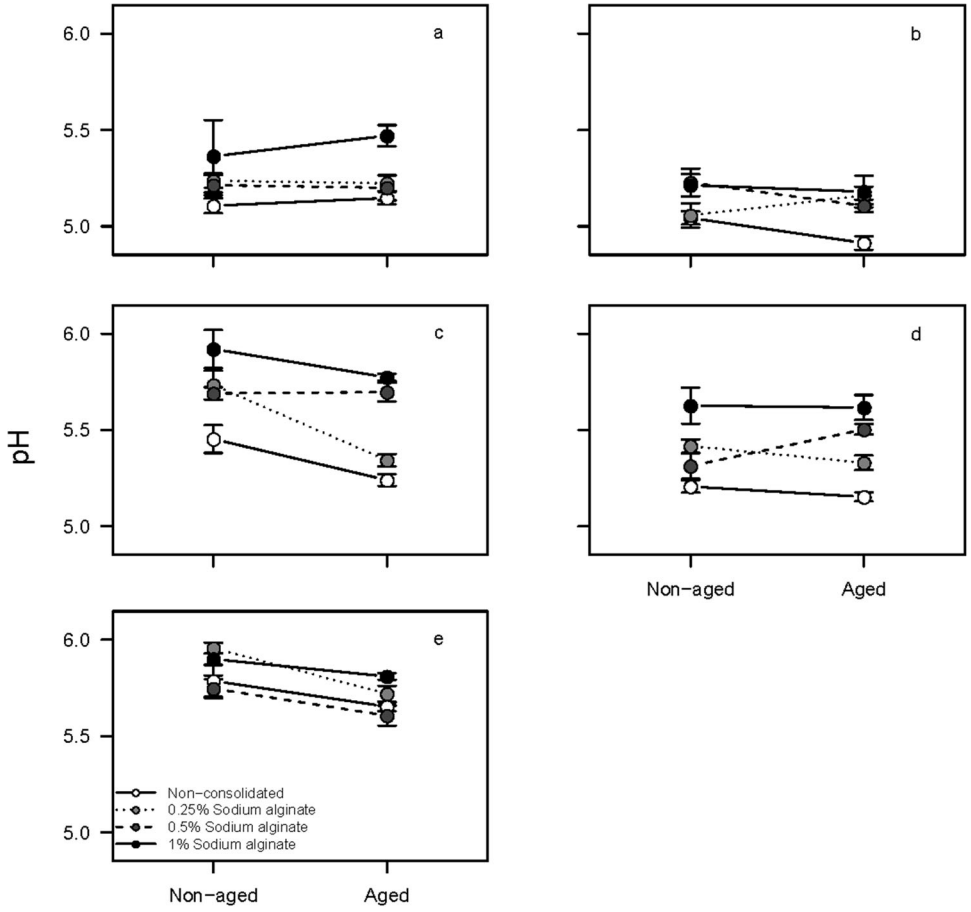

Figure 6. Mean effects of sodium alginate concentration and accelerated light ageing (10 Mlux hours) on the acidity (pH) of muka dyed with (a) Hinau-Paru1, (b) Hinau-Paru2, (c) Manuka-Paru1, (d) Manuka-Paru2, or (e) Non-dyed muka. Error bars indicate standard error.

consolidant and dye type (GLM Consolidant × Dye; $F_{12,80} = 3.641$, $p < 0.001$; Figure 6). For example, Hinau-Paru1 muka treated with 1% w/v consolidant was less acidic than Hinau-Paru1 muka treated with other concentrations (Figure 6(a)). In contrast, Hinau-Paru2 muka treated with either 0.5 or 1% w/v sodium alginate was less acidic than non-consolidated Hinau-Paru2 muka (Figure 6(b)). Manuka-Paru1 and Manuka-Paru2 muka became less acidic with the application of all concentrations of sodium alginate (0 vs. 0.25, 0.5, or 1% w/v), with greatest reduction in acidity with the application of 1% w/v consolidant, and similar changes in acidity with 0.25 and 0.5% w/v concentrations (Figure 6(c)–(d)).

Ageing tended to increase acidity of muka for non-dyed and Manuka-Paru1 dyed muka only, showing interactions between dye type and light ageing (GLM Dye × Age; $F_{12,80} = 5.018$, $p = 0.0012$). Non-dyed and Manuka-Paru1 muka increased in acidity post-ageing (Tukey post hoc, $p = 0.032$, 0.008; Figure 6(c), (e)) and remaining unchanged for Hinau-Paru1 or 2, and Manuka-Paru2 (Figure 6(a)–(b), (d)). No interactions between concentration of consolidant and ageing, or dye type and ageing were noted (both $p > 0.05$).

Discussion

Overall, sodium alginate consolidation had little effect on change in the colour of muka, acidity, and the majority of tensile properties post-ageing, with the exception of specific combinations of the concentration of sodium alginate, dye type, and light ageing. However, differences among dye types were noted, that frequently became intensified post-ageing.

Colour change

Visual assessments demonstrated sodium alginate consolidation was beneficial for reducing the light-induced colour change of Manuka-Paru1 dyed muka, with increasing concentrations of the consolidant sodium alginate associated with decreasing perceptible colour change after 10 Mlux hours exposure to UV-filtered light (e.g. non-consolidated muka: >1 JNF (Grey Scale 3.5); 1%w/v consolidant concentration: < 1 JNF (Grey Scale 4.5)). However, consolidation did not alter the colour stability of non-dyed muka, or of the other dye types assessed. Colour stability of non-consolidated paru-dyed muka in the current study

was consistent with previous findings (Lowe et al. 2014). These results suggest that both paru dye in general, and sodium alginate itself were generally stable to extended periods of light exposure. These results also indicate that both non-dyed and paru-dyed muka colour is substantially more stable in UV-filtered light than previously recognised (Lowe et al. 2016) thus potentially warranting a re-classification of artefacts made from paru-dyed muka to an 'intermediate' category for light sensitivity (e.g. artefacts that perceptibly fade with 0.4–3.6 MLux hours: sensitive, 10–100 Mlux hours: intermediate; Victoria and Albert Museum, London, United Kingdom (Derbyshire and Ashley-Smith 1999; Ashley-Smith, Derbyshire, and Pretzel 2002). Based on Te Papa Tongarewa National Museum of New Zealand's guidelines for display of 'sensitive' items, it would take over 400 years for any noticeable colour change in paru-dyed muka, though perhaps only for specific manuka-paru dye combinations. Nonetheless, our results suggest that the application of sodium alginate may reduce the extent of such colour change over time when on display.

Microfading revealed that colour change of non-dyed and paru-dyed muka, in response to 1 Mlux hour of UV-filtered light, was not influenced by consolidation with sodium alginate. Both non-dyed and paru-dyed muka showed considerable stability at this level of light exposure. Furthermore, all colour change of muka (ΔE) was in the BW3 to BW4 range, with fading at less than BW2 (i.e. $\Delta E_{00} = 1.5–1.6$) considered equivalent to Grey Scale 4 or 1 JNF at this exposure level (Ashley-Smith, Derbyshire, and Pretzel 2002; CIE Technical Committee 3-22 2004; Ford and Druzik 2013). These microfading results are in agreement with visual assessments at 1 Mlux hour exposure, providing confidence in the ability of the microfading technique to assess colour change due to light exposure up to this level. However, comparisons between microfading and accelerated light ageing at greater exposure levels were not possible with the microfader model used, due to the potential for both physical and spectral drift (B. Ford pers. comm. 2013).

However it is important to note that while consolidation of muka with 1%w/v sodium alginate had a limited influence on colour change post-ageing, the application of sodium alginate at this concentration substantially altered the appearance of the fibre, resulting in a shiny and a sometimes flaky surface appearance (see Supplementary Figure S1a). This suggests that regardless of the beneficial properties of the consolidant, the application of 1% w/v sodium alginate may be aesthetically inappropriate. In contrast, sodium alginate was impossible to observe by eye on non-dyed muka, regardless of the concentration, suggesting that it may be more readily absorbed into non-dyed muka.

Tensile properties

Measurement of a range of muka properties in response to ageing was an important aspect of this study, enabling insights into simultaneous ageing processes as well as expanding knowledge of the properties of muka. Generally, strength or breaking load have been the only properties reported in studies investigating the effects of ageing on tensile properties. Daniels (1999b) suggested that tensile tests were limited to measuring loss of strength, and could not give a measure of flexibility, toughness, or handle. However, the current study included a number of parameters in addition to strength, namely extensibility (strain to maximum and complete failure), stiffness (modulus), and toughness (specific energy absorbed to maximum load and complete failure), all common measures in standard textile tensile testing (Morton and Hearle 2008).

With the exception of muka stiffness, consolidation with sodium alginate had very little effect on these muka tensile properties pre- and post-accelerated light ageing. The application of the sodium alginate consolidant at higher concentrations tended to decrease stiffness of muka post-ageing, whereas the concentration of sodium alginate had inconsistent effects on the stiffness of muka pre-ageing. It is important to note that the magnitude of the decrease in stiffness of muka post-ageing was very small (approximately 5% of initial stiffness). Muka remained in relatively robust condition after ageing, compared with paru-dyed muka reported by others and seen by the authors first-hand in some naturally aged artefacts.

Comparisons among non-dyed muka and paru-dyed muka indicate that the initial dye process itself reduces the tensile properties of muka prior to any artificial ageing occurring. This finding is in agreement with previous research (Lowe et al., 2016), and may be relevant to decisions related to the display of artefacts constructed entirely from non-dyed or tanekaha-dyed muka compared with paru-dyed muka (Lowe et al. 2014). Current general display guidelines for sensitive artefacts (i.e. all plant fibres that are naturally dyed), do not incorporate acknowledgment of the differences in stability of different dyes. While unique combinations of paru and tannin sources may drive changes in the direction and magnitude of tensile properties, it appears muka dyed with hinau based dyes may be more susceptible to light ageing than muka dyed with manuka-kanuka based dyes. This is in direct contrast to previous research that considered muka dyed with hydrolysed tannins (i.e. hinau) to be considerably more stable than muka dyed with condensed tannins (i.e. manuka/kanuka, Daniels 1999a). Such differences between dye types may suggest future research to enable the identification of the tannin source in artefacts is therefore important.

Although consolidation had a minimal influence on the tensile properties of muka, the current study revealed changes associated with accelerated light ageing that have potential implications for artefact display. For example, muka artefacts displayed for ~417 years under the conditions stipulated in Te Papa's display guidelines for sensitive artefacts may experience up to 20% loss in tensile strength. At this time, however, it is important to consider what magnitude of tensile property change may be of concern to conservators, since statistical significance does not necessarily equate to noticeable changes in structural integrity. While some loss of tensile properties were found in muka overall post-light ageing (e.g. 10–20% loss of tenacity across all dye types), this was minimal compared with the loss of strength observed in some naturally aged museum artefacts (96–99%, Daniels 1999a). Therefore additional testing may be required to determine how tensile properties of severely degraded muka would respond to the application of sodium alginate as a consolidant.

Current understanding of the response of textile tensile properties to light ageing, and in particular, the effects of UV filtered light, is limited (Morton and Hearle 2008, p. 318). Despite this, the magnitude of deterioration observed in the present study can be evaluated in terms of the inherent range of tensile properties characteristic of muka used in Māori textiles. For example, tensile properties of muka vary considerably among cultivars when prepared with customary techniques and tested under identical test conditions (e.g. tenacity 0.3–0.7 N/tex; Lowe et al. 2010). Non-aged muka from the 'Ngutunui' cultivar was found to have similar strength (N/tex) to that of non-aged Tapamangu and Makaweroa muka (tenacity: 0.50 vs. 0.53 and 0.55 N/tex, this study; (Carr et al. 2005); two harakeke cultivars preferred for muka abstraction (Scheele 1997; Harris and Woodcock-Sharp 2000; Scheele 2005). In contrast, Daniels (1999a) observed non-aged muka (cultivar unknown) and degraded museum artefacts tested under different test conditions to have considerably lower tensile strength (0.24–0.25, 0.002–0.01 N/tex, respectively; Daniels 1999a). These comparisons suggest the decrease in strength observed in the current study falls within the range of strengths expected from non-aged muka from different cultivars, and relative to residual strengths in aged museum artefacts, is unlikely to be a concern in relation to display guidelines for sensitive objects.

In addition to standard tensile testing during experimental work, all muka consolidated with 1% w/v sodium alginate was observed to appear thicker, with markedly higher resistance to bending when handled than non-consolidated muka, with a similar effect noted for muka consolidated at lower concentrations. These observations reflect how the increase in bulk of each muka strand associated with the addition of consolidant has influenced bending properties. All other things being equal, an increase in fibre diameter will always result in a less flexible fibre in bending. In comparison, the decrease in stiffness (tensile modulus) measured for some combinations of concentration of consolidant, dye type, and ageing may be due to microstructural or molecular level effects, such as the penetration of water molecules from the consolidant solution into the cellulose polymer of the muka.

Acidity

In general, application of the consolidant did not influence the acidity of non-dyed muka, which tended to become more acidic post-ageing, though it was considerably less acidic than paru-dyed muka prior to light ageing. In contrast, sodium alginate had a desirable effect on the pH of all paru-dyed muka, with no instances of increased acidity with application of the consolidant. In some instances, consolidant application decreased the acidity of paru-dyed muka, however only for specific dye-consolidant concentration combinations. For example, the 0.25% w/v consolidant influenced the pH of Manuka-Paru1 only, whereas a sodium alginate consolidant concentration of 1% w/v was required to have an effect for other dye types. Therefore, understanding the dye type (tannin-paru combination) may be important for determining the concentration of the consolidant required to halt or minimise the degradative effects of increased acidity on paru-dyed muka. In cases where no knowledge of the dye type is known, then a conservative approach of applying a 1% w/v concentration may be needed in order to have an effect.

Whilst the acidity of all paru-dyed fibres ranged between pH 5–6, muka dyed with hinau was more acidic than muka dyed with manuka/kanuka. Only non-consolidated muka dyed with Hinau-Paru2 reached pH values below five post-ageing, levels that have been reported to promote both acid hydrolysis and iron mediated redox reactions (Daniels 1999a). In comparison, many paru-dyed Māori textile artefacts previously tested by Daniels (1999a) were considerably more acidic (pH 3.5–4.3). This suggests additional artificial ageing may be required on experimental fibres to ensure consolidants are tested on fibres with pH ranges reflecting those of naturally aged artefacts.

Limitations of this study

A possible limitation of the micro-extraction method for assessing the pH of textile artefacts is that measurements represent only a small portion of the entire artefact, and therefore may not provide a true reflection of the overall pH, especially if pH varies across an artefact (Vuori and Tse 2004; Tse 2007). However pH of muka demonstrated considerable consistency within each

consolidant/dye/ageing treatment combination, which not only suggested that treatments applied to the muka were consistent along the entire length of each strand, but also that little variation among muka strands existed. This provided confidence that differences observed among treatment combinations were not a consequence of the pH assessment method.

Another potential limitation of this study is that the accelerated light-ageing regime used was not sufficiently extreme to mimic the state of deterioration of artefacts made from paru-dyed muka in institutional collections. Light intensity was the only ageing mechanism used, while particular care was taken to keep UV exposure, temperature, and relative humidity within the range typical of desirable museum display conditions. The advantage of this is that the relative ranking of the effects after artificial ageing of sodium alginate concentrations and dye types on muka properties are likely to hold true (Feller 1994). However, the experimental programme did not simulate muka that was already in a degraded state prior to the consolidant being applied, or less than desirable ambient conditions. Both of these circumstances need to be explored to develop a more complete understanding of the potential long-term effects of consolidation using sodium alginate. For example, accelerated ageing at elevated temperatures and fluctuating relative humidity, as well as exposure to light, is one approach used for simulating age in less controlled conditions, such as poor storage (e.g. Garside and Wyeth 2005; Sato, Okubayashi, and Sato 2011). Very little quantitative data is available regarding the range of chemical and physical properties of naturally aged muka (see Daniels 1999a, for a summary). The design of an experiment using pre-aged muka would require careful consideration of pre-ageing criteria, in consultation with conservators familiar with the 'typical' properties of aged muka.

Aqueous v/s non-aqueous application of consolidant

It is also important to note that conservation treatments applied to iron-tannate dyed specimens in an aqueous solution may be highly effective due to water causing the fibres to swell and thus increase the penetration of the treatment into the fibres (Wilson 2012). However immersive aqueous treatments seem likely to have undesirable side-effects. For example, Reissland (2000) highlighted that aqueous (immersive) treatment of iron gall ink corrosion could result in the migration of soluble components (bleeding), colour change in the ink, and additional mechanical damage to degraded paper due to differential hydrophobicity. Daniels (1999b) also reported that use of an aqueous consolidant had the effect of causing the fibres in each bundle to draw together when drying, reducing the diameter of the fibre bundle, probably due to the surface tension of the water and the adhesive qualities of the consolidant, and that some conservators were reluctant to use water-based treatments on very degraded fibres. Therefore, conservation science researchers at the British Museum and University of Manchester have most recently focused on the development of non-aqueous treatments for iron-tannate dyed organic materials (Wilson 2012; The British Museum 2015). However, it is important to distinguish between immersive treatments and those that use a consolidant that is soluble in water, applied in an alternative manner. The use of immersive conservation treatment for Māori textiles is uncommon in a New Zealand or Australian context. However the delivery of an aqueous consolidant via vapour or brush can prevent any removal of water soluble components of the artefact or dye bleed, and indeed the drawing together effect of water and consolidant mentioned in Daniels (1999b) may well be considered an advantage for conservators when attempting to repair very deteriorated textile elements/yarns. Because of the reported efficacy of aqueous consolidants, their potential for greater absorption into deteriorated fibres, and their greater safety for practitioners, the authors of this report consider aqueous consolidants suitable for consolidation of paru-dyed muka, or indeed other organic objects dyed with iron-tannate dyes, using non-immersive delivery.

Summary

Overall, this study found that sodium alginate appears to have no negative effect on paru-dyed fibres (Ngutunui cultivar, New Zealand flax, *Phormium tenax*) from two specific plant phenol and paru sources, and in some cases provided some benefit. Our results also indicate the complexity of dye/fibre interactions specific to the efficacy of different concentrations of sodium alginate. Therefore any recommendations made about the most effective concentration of sodium alginate may always be reliant on the ability of a conservator to be able to easily identify the plant phenol source of the dye. There is currently no easy way to identify the plant phenol source in artefacts, an area of future research that should be pursued. In increased concentrations, and for more deteriorated fibres, it is possible that the effects of consolidation with sodium alginate may be greater than measured here, indicating that further testing, on model fibres artificially aged to more accurately represent the high levels of deterioration found in paru-dyed Māori textiles is a research priority. It is important to note that the colour of paru-dyed muka is substantially more stable in UV-filtered light than previously recognised, meaning artefacts made from it could be re-characterised as more stable

to light, and able to withstand longer periods of display. Another finding, that microfading data were in agreement with visual assessments of colour change at 1 Mlux hour exposure, provides confidence in this relatively new technique to assess colour change up to this level of exposure to light.

List of suppliers

Elsec 764 Environmental Monitor, Littlemore Scientific Engineering: Dorset, UK

Instron 4464 Universal Testing Machine, Instron Corporation: Canton, Massachusetts, USA

Microelectrodes Inc. Micro pH Electrode – Model MI-4152 40 Harvey Road Bedford, New Hampshire 03110 USA

Microsol Light Fastness Tester Model 495 fitted with a mercury vapour lamp (400 W, 19500 lumen/100 h) James H. Heal and Co. Ltd.: Halifax, UK, modified for alternative relative humidity and temperature control by Centre for Materials Science and Technology, University of Otago, Dunedin, NZ

Newport Oriel Apex Fibre Illuminator, Oriel Fading Test System (O-MFT), Newport Corporation: Irvine, California, USA

Orion pH/mV/temperature metre – Model 420Aplus, 40 Harvey Road Bedford, New Hampshire 03110 USA.

Sodium alginate, FMC BioPolymer, 1735 Market Street, Philadelphia, PA 19103, USA

Spectrometer (Model: PDA-512-TS-USB2/1.58/100 µm/298-1107 nm) and associated Spec32 software, Control Development Inc., 2633 Foundation Dr., South Bend, In, USA

Thorlabs neutral density filter, Optical density: 4.0 (ND40B). Thorlabs Inc. 56 Sparta Avenue, Newon, NJ, USA

Tru Vue Conservation Reflection Control®, UV Filter Glass, Tru Vue Inc., 9400 W. 55th Street, McCook, Illinois 60525, USA

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Disclosure statement

No potential conflict of interest was reported by the authors.

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References

Ashley-Smith, J., A. Derbyshire, and B. Pretzel. 2002. "The Continuing Development of a Practical Lighting Policy for Works of Art and Paper and Other Object Types at the Victoria and Albert Museum." In *ICOM Committee for Conservation, 13th Triennial Meeting, Preprints*, edited by R. Vontobel, 3–8. London: James & James.

Barker, K. 2002. "Iron Gall and the Textile Conservator." In *Strengthening the Bond: Science & Textiles, North American Textile Conservation Conference, Preprints*, edited by V. J. Whelan, 7–13. Philadelphia: Philadelphia Museum of Art.

Boronkay, G. 2008. "Colour Conversion Centre." http://ccc.orgfree.com/.

The British Museum. "Interventive Conservation of Black-Dyed Organic Materials: The Problem of Metal-Polyphenol Complexes." Accessed 12 August 2015. http://www.britishmuseum.org/research/research_projects/all_current_projects/collaborative_doctoral_awards/black-dyed_organic_materials.aspx.

British Standards Institution. 1990. *BS 1006: 1990. Methods of Test for Colour Fastness of Textiles and Leather. UK-TN. Colour Fastness to Artificial Light: Mercury Vapour Fading Lamp Test (Withdrawn December 2010)*. London: British Standards Institution.

British Standards Institution. 1993. *BS EN 20105-A02 1993 ISO 105-A02: 1987 Textiles – Tests for Colour Fastness. Part A02 Grey Scale for Assessing Change in Colour*. London: British Standards Institution.

British Standards Institution. 2013. *BS EN ISO 105-B02: 2013 Textiles – Tests for Colour Fastness. Part B02 Colour Fastness to Artificial Light: Xenon Arc Fading Lamp Test*. London: British Standards Institution.

Bulska, E., and B. Wagner. 2004. "A Study of Ancient Manuscripts Exposed to Iron-Gall Ink Corrosion." *Comprehensive Analytical Chemistry* 42: 755–788.

Carr, D. J., N. M. Cruthers, R. M. Laing, and B. E. Niven. 2005. "Fibers from Three Cultivars of New Zealand Flax (*Phormium Tenax*)." *Textile Research Journal* 75 (2): 93–98.

CIE Technical Committee 3-22. 2004. "Control of Damage to Museum Objects by Optical Radiation." CIE Technical Report No. 157. Austria: CIE International Commission on Illumination.

Daniels, V. 1996. "Maori Black Mud Dyeing of Phormium Tenax Fibre." *Dyes in History and Archaeology* 14: 9–13.

Daniels, V. 1998a. "Degradation of Artefacts Caused by Iron-Containing Dyes." In *Dyes in History and Archaeology*, edited by J. Kirby, 211–215. Greenwich: Archetype Publications.

Daniels, V. 1998b. "The Conservation and Deterioration of Black Dyed New Zealand Flax, Part 3." Mechanical Properties. British Museum Report No. 23. Unpublished Report.

Daniels, V. 1999a. "Factors Affecting the Deterioration of the Cellulosic Fibres in Black-Dyed New Zealand Flax (*Phormium Tenax*)." *Studies in Conservation* 44 (2): 73–85.

Daniels, V. 1999b. "Stabilisation Treatments for Black-Dyed New Zealand Flax." In *ICOM Committee for Conservation, 12th Triennial Meeting, Preprints*, edited by J. Bridgland, 579–84. London: James & James.

Derbyshire, A., and J. Ashley-Smith. 1999. "A Proposed Practical Lighting Policy for Works of Art on Paper at the

V&A." In *ICOM Committee for Conservation, 12th Triennial Meeting*, edited by D. Grattan, 38–41. London: James & James.

Feller, R. L. 1994. *Accelerated Aging – Photochemical and Thermal Aspects*. Michigan: The Getty Conservation Institute.

Fenton, G. A., I. J. Miller, G. J. Smith, and R. Te Kanawa. 2004. "Colour Stabilizing Effect of Zinc Alginate Consolidant on Black Iron-Tannate Dyed Maori Textiles." In *AICCM 2nd Biennial Textile Symposium, Preprints*, 61–64. Canberra: Australian Institute for the Conservation of Cultural Material.

Ford, B. L., and J. Druzik. 2013. "Microfading: The State of the Art for Natural History Collections." *Collections Forum* 27 (1–2): 54–71.

Ford, B. L., and N. Smith. 2010. "Protecting the Most Important, Most Exhibited and Most Fugitive Museum Objects from Light Fading." In *American Institute for Conservation of Historic and Artistic Works, Postprints*, edited by J. Thompson, A. Holden, G. Petersen, and S. Stevens, vol. 2, 30–42. Milwaukee: Research and Technical Studies Group of the American Institute for Conservation of Historic Works.

Garside, P., and P. Wyeth. 2005. "Assessing the Physical State of the Fore-topsail of HMS Victory." In *Scientific Analysis of Ancient & Historic Textiles. Informing Preservation, Display and Interpretation. AHRB Research Centre for Textile Conservation & Textile Studies, 2nd Annual Conference, Postprints*, edited by R. Janaway, and P. Wyeth, 118–125. London: Archetype Press.

Harris, W., and M. T. U. A. Woodcock-Sharp. 2000. "Extraction, Content, Strength, and Extension of Phormium Variety Fibres Prepared for Traditional Maori Weaving." *New Zealand Journal of Botany* 38 (3): 469–487.

Hofenk de Graaff, J. H., W. Roelofs, and M. R. Bommel. 2004. "Dyeing and Writing; A Comparison of the Use and Degradation of Iron-Gall Complexes on Textiles and Paper." In *The Colourful Past: Origins, Chemistry and Identification of Natural Dyestuffs*, 31–41. London: Archetype.

Hothorn, T., F. Bretz, and P. Westfall. 2008. "Simultaneous Inference in General Parametric Models." *Biometrical Journal* 50 (3): 346–363.

International Organisation for Standardisation. 1994. *ISO 105-A01:1994(E) Textiles – Tests for Colour Fastness. Part A01 General Principles of Testing Edition. 5*. Geneva: International Organisation for Standardisation.

International Organisation for Standardisation. 2014. *ISO 105-B02:2014 Textiles – Tests for Colour Fastness. Part B02 Colour Fastness to Artificial Light: Xenon Arc Fading Lamp Test*. Geneva: International Organisation for Standardisation.

Kolar, J., M. Sala, M. Strlic, and V. S. Selih. 2005. "Stabilisation of Paper Containing Iron-Gall Ink with Current Aqueous Processes." *Restaurator* 26 (3): 181–189.

Krekel, C. 1999. "The Chemistry of Historical Iron Gall Inks: Understanding the Chemistry of Writing Inks Used to Prepare Historical Documents." *International Journal of Forensic Document Examiners* 5: 54–58.

Lowe, B. J., D. J. Carr, R. E. McCallum, T. Myers, R. Ngarimu-Cameron, and B. E. Niven. 2010. "Understanding the Variability of Vegetable Fibres: A Case Study of Harakeke (Phormium tenax)." *Textile Research Journal* 80 (20): 2158–2166.

Lowe, B. J., C. A. Smith, S. J. Fraser, R. A. Paterson, B. Ford, K. C. Gordon, R. Ngarimu-Cameron, and F. Daroux. 2016. "Light-Ageing Characteristics of Māori Textiles: Colour, Strength and Molecular Change." *Journal for Cultural Heritage*.

Lowe, B. J., C. A. Smith, K. C. Gordon, L. R. Hanton, B. L. Ford, A. Korsten, S. J. Fraser, and B. A. Lomax. 2014. "Relating Colour, Chemical and Physical Characteristics of Artificially Light-Aged New Zealand Plant Fibres." In *ICOM Committee for Conservation, 17th Triennial Conference, Preprints*, edited by J. Bridgland, 1606–1613. London: James & James.

Luo, M. R., G. Cui, and B. Rigg. 2001. "The Development of the CIE 2000 Colour-Difference Formula: CIEDE2000." *Color Research & Application* 26 (5): 340–350. doi: 10.1002/col.1049.

McCallum, R. E., and D. J. Carr. 2012. "Identification and Use of Plant Material for the Manufacture of New Zealand Indigenous Woven Objects." *Ethnobotany Research and Applications* 10: 185–198.

More, N., G. Smith, R. Te Kanawa, and I. Miller. 2003. "Iron-Sensitised Degradation of Black-Dyed Maori Textiles." *Dyes in History and Archaeology* 19: 144–148.

Morton, W. E., and J. W. S. Hearle. 2008. "Tensile Properties." In *Physical Properties of Textile Fibres*. 4th ed., 274–321. Manchester: The Textile Institute.

R Core Team. 2014. *R: A Language and Environment for Statistical Computing*. Vienna: R Foundation for Statistical Computing.

Reedy, Terry J., and Chandra L. Reedy. 1988. *Statistical Analysis in Art Conservation Research*. Research in Conservation 1. Marina del Rey, CA: Getty Conservation Institute.

Reedy, Terry J., and Chandra L. Reedy. 1992. *Principles of Experimental Design for Art Conservation Research*. Newark, DE: Getty Conservation Institute.

Reissland, B. 2000. "Ink Corrosion: Side-Effects Caused By Aqueous Treatments for Paper Objects." In *The Iron Gall Ink Meeting, Newcastle Upon Tyne, 4th & 5th September 2000: Postprints*, edited by A. Jean E. Brown, 109–114. Newcastle-Upon-Tyne: University of Northumbria.

Robertson, A. R. 1977. "The CIE 1976 Color-Difference Formulae." *Color Research & Application* 2 (1): 7–11.

Sato, M., and S. Okubayashi. 2010. "Consolidation Treatment of Japanese Ceremonial Doll's Hair at Edo Period with Polyethylene Glycol." *Journal of Textile Engineering* 56 (3): 65–70.

Sato, M., S. Okubayashi, and M. Sato. 2011. "Development of Conservation Procedures for Late Edo Period Japanese Ceremonial Dolls' Hair: Evaluation of Effective Treating Reagents by Using Artificially Degraded Black-Dyed Silk Fibres." *Journal of Cultural Heritage* 12 (2): 157–163.

Scheele, S. 1997. "Dunedin Botanic Gardens Harakeke Collection." *He Kōrero Kōrari* 2: 3.

Scheele, S. 2005. *Harakeke the Rene Orchiston Collection*. 3rd ed. Lincoln: Manaaki Whenua Press.

Sharma, G., W. Wu, and E. N. Dalal. 2005. "The CIEDE2000 Color-Difference Formula: Implementation Notes, Supplementary Test Data and Mathematical Observations." *Color Research and Application* 30 (1): 21–30.

Smith, G. J., R. Te Kanawa, I. J. Miller, and G. A. Fenton. 2005. "Stabilisation of Cellulosic Textiles Decorated with Iron-Containing Dyes." In *Dyes in History and Archaeology*, edited by J. Kirby, A. Quye, C. Cooksey, M. R. V. Bommel, and I. C. Nederland, vol. 20, 89–94. Amsterdam: Instituut Collectie Nederland.

Tamarapa, A. 2011. *Whatu Kakahu: Maori Cloaks*. Wellington: Te Papa Press.

Te Kanawa, D. 1992. *Weaving a Kakahu*. Wellington: Bridget Williams Books in Association with Aotearoa Moananui a Kiwa Weavers.

Te Kanawa, R. 2001. "Conservation of Māori Textiles Decorated with Traditional Dyes." In *American Institute for*

Conservation, 29th Annual Meeting, Postprints, edited by J. L. Merritt, and V. J. Whelan, vol. 11, 1–10. Washington: The Textile Specialty Group Postprints.

Te Kanawa, R. 2005. "Consolidation of Degraded Iron-Tannate Dyed *Phormium tenax* Fibres Using Zinc Alginate." Master's Thesis, Victoria University of Wellington.

Te Kanawa, R., G. J. Smith, G. A. Fenton, I. J. Miller, and C. L. Dunford. 2008. "Evaluation of Consolidants for Black Iron Tannate-dyed Maori textiles." *Dyes in History and Archaeology* 21: 225–230.

Te Kanawa, R., S. Thomsen, G. Smith, I. Miller, C. Anadry, and D. Cardon. 2002. "Traditional Maori Dyes." *Dyes in History and Archaeology* 18: 47–50.

Tse, S. 2007. "Guidelines for pH Measurement in Conservation." In *CCI Technical Bulletin No. 28*. Ottawa: Department of Canadian Heritage.

Vuori, J., and S. A. Tse. 2004. "Preliminary Study of a Micro Extraction Method for Measuring the pH of Textiles." *ICOM Textile Working Group Newsletter* 19: 4–15.

Wilson, H. 2007. "Analysis of the Current Research into the Chemistry of Iron Gall Ink and Its Implications for Paper Conservation." Master's Thesis, University of Oxford.

Wilson, H. 2012. "Investigation into Non-Aqueous Remedial Conservation Treatments for Iron-Tannate Dyed Organic Materials." PhD Thesis, University of Manchester.

Wilson, H., C. Carr, and M. Hacke. 2012. "Production and Validation of Model Iron-Tannate Dyed Textiles for Use as Historic Textile Substitutes in Stabilisation Treatment Studies." *Chemistry Central Journal* 6: 641–656.

Yang, H. H. 2006. "Polyamide Fibers." In *Handbook of Fiber Chemistry*. 3rd ed. edited by M. Lewin, 107–108. Boca Rafton: CRC Press.