Colouring of Pacific barkcloths: identification of the brown, red and yellow colourants used in the decoration of historic Pacific barkcloths

T. H. Flowers, M. J. Smith* and J. Brunton

Abstract
Barkcloth textiles made in the Pacific islands and collected by western explorers in the eighteenth and nineteenth centuries form part of many museum collections worldwide. Here high-performance liquid chromatography (HPLC) and X-ray fluorescence (XRF) were used on cloths that were highly coloured or pigmented specifically focussing on identifying the red, yellow and brown colorants. The cloths studied came from collections held at the Hunterian, University of Glasgow, the Economic Botany Collection, Royal Botanic Gardens Kew and the Centre for Textile Conservation and Technical Art History, University of Glasgow. HPLC analysis was carried out following a sequential extraction procedure to minimise changes to the colorants during extraction. A portable XRF was used so no invasive sampling was required. A small number of plant derived colorants were found, *Morinda citrifolia* (noni or morindin or morindone), *Rubia tinctorum* (madder), tree tannins and *Curcuma longa* (turmeric) plus an inorganic colorant, iron oxide. For 40 samples a single colorant was found while in the remaining 12 samples combinations of up to three colorants were found. Madder was found in only 2 samples on the same cloth. The morindone coloured samples were all red whereas morindin samples were both red and yellow. Morindin was used predominantly in combination with other colouring agents. A combination of iron ochre and organic colorant was found in 4 samples. These findings show that despite the numerous potential colorant sources for red, brown and yellow shades listed in the many accounts of historic barkcloth making, only five types of plant colourant and one inorganic pigment were found. There are a number of potential reasons for these findings. Some colours may have faded and so no longer appear coloured. It is also possible that, as some of these cloths were prepared specifically as gifts for visitors or for ceremonial uses, the makers used materials that they knew would retain their integrity over time. Perhaps, like artisans worldwide, experience had taught them that some colorants, although initially bright and vivid, faded over time.

Keywords: Pacific barkcloth, Pigments, Dyes, HPLC, XRF, Colorants, Identification

Introduction
Barkcloth (tapa) is one of the most distinctive products of cultures originating from the Pacific islands. Barkcloth is a non-woven material made from beaten inner bark and often referred to as a bast fibre. Tapa production was central to providing clothing and bedding, as well as for decoration and ceremonial purposes [1]. The name *tapa*, now a universal term for Pacific barkcloth, derives from the Samoan term *tapa* for the uncoloured border of a piece of barkcloth. In Hawaii *kappa* describes a range of different types of barkcloth. Tapa is constructed from different types of plant, most commonly *Broussonetia papyrifera* (paper mulberry), *Artocarpus altilis* (breadfruit), *Ficus prolica* (banyan) or *Pipturus albidus* (mamaki), producing a matted, strong and fibrous structure, beaten to resemble a paper-like material or textile. Varied approaches to making tapa are noted within different
Pacific cultures, but these are variations based on a central methodology [2].

Traditionally, the colourants used to dye plain barkcloth were obtained from plants, trees and earth pigments. When different cultures met through trading, especially in the Tonga–Fiji–Samoa region, different patterns on barkcloth became identifiers of where they originated. The arrival of missionaries and other Westerners during the nineteenth century saw a change in the materials used to produce barkcloth, with imported materials sometimes used.

Whilst the number of different types of plants recorded as being used to provide differing colours is large and varied [3] the literature on the analysis of the various types of dyes found to be present in barkcloths remains narrow [4, 5]. A comprehensive review on the materials used in barkcloth production by Larson [6] listed only a few sources of plant colorants and inorganic pigments as commonly used. There has been very little scientific analysis carried out on the colourants on these cloths excepting work by Bisulca et al. [4] and the published literature on the materials used and their manufacture for the most part comes from historic accounts of early missionaries or explorers. Research carried out to determine the colourants used on Hawaiian barkcloth noted that there are over 10 plant species documented as historic red dyes, however from the barkcloth pieces analysed for red colouring all came from noni (Morinda citrifolia). In cases where the red design was not organic, the red colourant was found to be an iron oxide pigment [4].

The findings presented here focus on the analysis of organic and inorganic colourants used on cloths and detail extraction protocols which minimise damage to colourants under study and also reduce the number of steps used in the process. In this study cloths from the eighteenth and nineteenth centuries from 3 collections are used to determine the variation and/or similarities of these colourants by carrying out analysis using high-performance liquid chromatography (HPLC) and X-ray fluorescence (XRF) on cloths that were highly coloured or pigmented specifically focussing on identifying the red, yellow and brown colourants.

**Materials and methods**

**Barkcloths**

The 54 historic cloths analysed came from three collections, The Hunterian, University of Glasgow, Glasgow (33), the Economic Botany Collection (EBC), Royal Botanic Gardens, Kew, London, (16) and a sample book at the Centre for Textile Conservation and Technical Art History, University of Glasgow (5). These are detailed in Table 1. The Hunterian acquired its collection from several collectors including William Hunter who acquired barkcloths collected during Captain Cook’s voyages during the 1760 s and 1770 s. Alexander Angus donated a collection of Hawaiian and Tahitian cloths in 1810 and George Turner lived in Samoa for many years from 1841 and donated his collection. The EBC barkcloths came from a number of donors mainly in the second half of the nineteenth century. A sample book at the CTCTAH states on the first page that the samples within it were ‘collected during the three voyages of Captain Cook to the Southern Hemisphere’ which took place between 1768 and 1780. It consists of 30 samples of various types of cloths. The origins of the cloths given in Table 1 are based on current curatorial records of each collection.

**Extraction and HPLC analysis**

Samples were taken from the edges of cloths or from damaged areas, from regions of uniform colour. Not all colours could be sampled if they did not appear at edges or the areas were too small or the cloths themselves were small. Depending on the availability of material 1–5 mg samples were used for HPLC analysis. Because the colourants frequently only lightly coat the surface of the barkcloth fibres the weight of cloth sampled is not a good indicator of the weight of dye present.

The extraction method was based on the findings of Wouters et al. [7] who investigated the use of mild extractants for textiles and paints to minimise changes to the dyes during extraction. Acetone was omitted from their mildest extractant (oxalic acid/methanol) to eliminate the need to evaporate the extract to dryness and redissolve to prevent interference in the UV detection by acetone. For more acidic extractions we substituted hydrofluoric acid–methanol with hydrochloric acid–methanol on safety grounds. A sequential extraction procedure was used to enable extractants of increasing acidity to be used with a single barkcloth sample.

The tapa sample (1–5 mg) was placed in a 500 µL polypropylene microcentrifuge tube and 250 µL of the initial extracting solution added. A small hole was made in the tube lid to prevent pressure build up and the tube placed in a water bath as follows. Extraction 1: 60% methanol 0.002 M oxalic acid 80 °C 20 min; Extraction 2: 50% methanol 0.1 M HCl 80 °C 20 min; Extraction 3: 50% methanol 1 M HCl 90 °C 30 min. After each extraction the extract was made back up to the nominal volume with the appropriate extracting solution and filtered through a 4 mm 0.2 µm Teflon syringe filter into a 300 µL fixed insert autosampler vial. A further 50 µL of extracting solution was used to rinse the tube and filter into the autosampler vial. The sample was then extracted with the next extracting solution.

Analysis was carried out using a gradient high pressure chromatography (HPLC) system with a UV
Table 1  Details of the size, descriptions and areas of backcloths sampled

| Accession number | Origin (tentative) | Length (mm) | Width (mm) | Description (mm) | Colours sampled HPLC | Colours sampled XRF |
|------------------|--------------------|-------------|------------|------------------|----------------------|---------------------|
| **CTCTAH sample book** |                    |             |            |                  |                      |                     |
| CTCB2016 No.10   | Tahiti             | 209         | 131        | Plain yellow     | Yellow               |                     |
| CTCB2016 No.18   | Tahiti             | 89          | 52         | Red/yellow/black | Yellow               |                     |
| CTCB2016 No.20   | Hawaii             | 71          | 54         | Plain red        | Red                  |                     |
| CTCB2016 No.23   | Tahiti             | 200         | 128        | Plain red        | Red                  |                     |
| CTCB2016 No.29   | Tonga              | 67          | 49         | Red/yellow/black | Yellow               |                     |

| **Hunterian museum** |                    |             |            |                  |                      |                     |
| E417/2             | Samoa              | 2150        | 1865       | Black/yellow pattern on undyed background | Yellow               |                     |
| E417/5             | Fiji               | 2200        | 590        | Black with red border | Red                  |                     |
| E417/11            | Tonga              | 1570        | 710        | Red zigzags on black background | Red | Red |
| E457/3             | Hawaii             | 1650        | 1350       | Red and yellow stripes on undyed background | Yellow               |                     |
| E458/1             | Samoa or Cook Islands | 1840  | 900       | Yellow/black pattern on undyed background | Yellow               |                     |
| E458/2             | Hawaii             | 950         | 750        | Thin red stripes on yellow background | Yellow               |                     |
| E458/3             | Fiji               | 2120        | 1800       | Brown line pattern on undyed background red/black border | Brown, red | Brown, red |
| E458/4             | Tahiti or Marquesas Islands | 3930  | 1480       | Plain red-brown | Red-brown | Red-brown |
| E458/6             | Tonga              | 3530        | 1580       | Plain brown and brown/yellow pattern | Brown               |                     |
| E591/4             | Polynesia          | 1220        | 950        | Undyed cloth     | Undyed               |                     |
| E594/2             | Tahiti             | 1310        | 430        | Undyed cloth     | Undyed               |                     |
| E594/8             | Tahiti             | 810         | 360        | Undyed cloth     | Undyed               |                     |
| E595              | Tonga              | 2930        | 1590       | Red with undyed spots | Red | Red |
| E595/1             | Tahiti             | 1960        | 1090       | Red pattern on yellow background | Red, yellow | Red, yellow |
| E596/1             | Tahiti or Hawaii   | 440         | 250        | Undyed cloth     | Undyed               |                     |
| E596/5             | Tahiti             | 415         | 290        | Undyed cloth     | Undyed               |                     |
| E596/6             | Tahiti             | 300         | 140        | Undyed cloth     | Undyed               |                     |
| E596/7             | Fiji               | 1550        | 954        | Undyed cloth     | Undyed               |                     |
| E596/8             | Tahiti             | 173         | 153        | Undyed cloth     | Undyed               |                     |
| E598/1             | Hawaii             | 233         | 172        | Red/yellow/black/undyed stripe pattern | Red, yellow | Red, yellow, undyed |
| E598/2             | Hawaii             | 198         | 160        | Red/black pattern | Red | Red |
| E598/3             | Hawaii             | 443         | 113        | Red/yellow/black pattern | Red | Red |
| E598/4             | Hawaii             | 350         | 247        | Red/yellow/black pattern | Red | Red |

| **Hunterian museum** |                    |             |            |                  |                      |                     |
| E599              | Tahiti or Hawaii   | 1300        | 1050       | Dark brown flaking coating | Dark brown  | Dark brown, bare |


Table 1 (continued)

| Accession number | Origin (tentative)     | Length (mm) | Width (mm) | Description (mm)                        | Colours sampled HPLC | Colours sampled XRF |
|------------------|------------------------|-------------|------------|-----------------------------------------|----------------------|---------------------|
| E600  | Tahiti or Hawaii       | 945         | 440        | Red border stripe on undyed cloth       | Red, undyed          | Red, undyed         |
| E601  | Hawaii                 | 910         | 755        | Red, grey and undyed stripes           | Red, undyed          |                     |
| E602  | Hawaii                 | 780         | 500        | Red/yellow/black pattern               | Red, yellow          | Red, yellow         |
| E603  | Tahiti                 | 1100        | 670        | Plain red-brown cloth                   | Red-brown            |                     |
| E606  | Cook Islands           | 522         | 175        | Plain red and undyed blocks             | Red, undyed          | Red, undyed         |
| E608  | Tahiti                 | 530         | 510        | Plain yellow                           | Yellow               |                     |
| E610  | Fiji                   | 1545        | 890        | Undyed cloth                           |                      |                     |
| E611/3 | Hawaii               | 2100        | 1200       | Mottled grey                           | Grey                 |                     |
| E667  | Hawaii                 | 990         | 860        | Red/yellow/black pattern               | Red                  |                     |
| Kew EBC |                      |             |            |                                        |                      |                     |
| 42853A | Hawaii               | 110         | 85         | Plain red cloth                        | Red                  |                      |
| 42861 | Samoa                 | 2340        | 950        | Poncho red/black/undyed pattern         | Red, undyed          |                     |
| 42863 | Samoa                 | 2045        | 1950       | Stripe and leaf pattern on undyed back- | Red-brown, Yellow    |                     |
| 42885H | Hawaii               | 560         | 230        | Plain dark brown cloth                  | Dark brown           |                     |
| 42947A | Hawaii               | 2190        | 955        | Red handprints on yellow background     | Red, yellow          |                     |
| 42947B | Hawaii               | 1550        | 1340       | Red stripes on yellow background        | Red, yellow          | Red, yellow         |
| 42958A | Hawaii               | 1760        | 1713       | Red pattern on yellow background        | Yellow               |                     |
| 42958B | Hawaii               | 1350        | 1180       | Brown and fine red pattern on undyed ba- | Brown, light brown   |                     |
| 42958C | Hawaii               | 1374        | 938        | Red leaf pattern on yellow background   | Red, yellow          |                     |
| 42965 | Hawaii               | 2732        | 2117       | Undyed cloth                           |                      |                     |
| 42966  | Hawaii               | 3455        | 2340       | Mottled red on undyed background        | Red                  |                     |
| 42967 | Hawaii               | 3308        | 2576       | Red/purple mottles in triangular pattern | Red, purple          |                     |
| 42979 | South Sea Islands     | 1983        | 1905       | Plain glossy red coating                | Red                  |                     |
| 67802A | Unknown              | 159         | 133        | Red/yellow stripe pattern               | Red                  |                     |
| 73329 | Tahiti               | 2340        | 1570       | Brown pattern/tassles on undyed backg- | Brown                |                     |
| 98041 | Unknown              | 2915        | 945        | Plain red-brown                        | Red-brown            | Red-brown           |

diode array detector. The Merck-Hitachi HPLC system comprised: L-7200 autosampler, L-7100 gradient pump, Jones Chromatography Genesis C18 4 µm 250 by 4.6 mm column, L-7350 column oven, L-4500 diode array detector and L-7000 HPLC system manager software. The extracts were analysed using acetonitrile:water gradient elution (30% to 98% acetonitrile over 30 min) with 0.1% phosphoric acid (Table 2).
Spectra were collected from 200 to 600 nm and a chromatogram extracted at 425 nm. Where tannins were present a second HPLC gradient was used (10% to 95% acetonitrile over 25 min) with 0.25% phosphoric (Table 2). Spectra were collected from 200 to 600 nm and a chromatogram extracted at 275 nm. 59 samples were tested from 47 cloths (Table 1).

Components were identified primarily on the basis of their UV–visible spectra and retention time. A subset of barkcloth sample extracts were analysed using a Shimadzu LC2010A HT LC system and LCMS2010EV Mass spectrometer HPLC–MS system using the same Genesis column and water:acetonitrile gradient 1 with 0.1% formic acid. UV detection was at 370 nm and MS detection was in negative ion mode to confirm the UV based component identity by the mass of the molecular ion. A soft ionisation was used to preserve the molecular ion.

Because of the scarcity of available standards to generate reference UV–visible spectra and retention times, an in-house spectrum library was generated using methanol extracts of plant materials. Peak identification was based on literature references of the major components present, their relative retention characteristics and their UV–visible data [5]. Confirmation of peak identity was obtained from the mass of the molecular ion using LC–MS.

### XRF analysis

XRF analysis was carried out using a Niton XL3t GOLDD+ handheld XRF in mining mode (Main range 15 s, Low range 15 s, High range 10 s, Light range 20 s). XRF analysis could be carried out anywhere on a cloth but required a uniform area with a minimum diameter of 4 mm. 28 samples (9 undyed controls, 19 dyed samples) were tested from 21 cloths (Table 1).

### Results and discussion

#### HPLC

Seven very light yellow/brown barkcloth samples all yielded chromatograms with no peaks suggesting that this represents the varying colours of the undyed bark-cloth. A further two similarly coloured samples showed the presence of turmeric. The 49 coloured samples where chromatographic peaks were obtained produced organic dyes from 4 plant sources: noni (Morinda citrifolia), turmeric (Curcuma longa), madder (Rubia tinctorum) and non-specific tannins. Identification of the plant sources was based on the presence of their major characteristic peaks. In the case of noni it is generally accepted that the presence of morindin and/or morindone is sufficient [5] even though the mixture of anthraquinones present as minor constituents may be more complex. Noni was found in two forms, dominated by either the glycoside morindin (Fig. 1a shows a typical chromatogram) or the aglycone morindone (Fig. 1b). Many of the anthraquinone plant dyes are present in the plant as glycosides where the coloured anthraquinone molecule is bound to one or more sugar molecules. During dye manufacture the glycosides can be hydrolysed by heat or acid or by the enzymes present in the plant extract. The noni based dyes (morindin and morindone) are naturally yellow but the colour is pH dependent, red above pH 10 [8], and burnt lime can be used to create a red dye [6]. Strongly acid extracting solutions can hydrolyse the glycosides releasing the aglycone anthraquinone and further degrade the molecule thus losing information on the exact nature of the dye molecule and the dye manufacturing process [9]. A review paper by Degano et al. [10] discusses the effects of acidic methanol used for the extraction of dyes in historic paint and textiles samples. Weaker acid extractions using oxalic acid or hydrofluoric acid have been recommended for paint samples to avoid hydrolysis of the dye molecules [7, 11]. The dyes used on barkcloths are frequently present as a surface coating on the barkcloth fibre without use of a mordant and are not strongly bound to the fibres. A weakly acid extracting solution can successfully extract many of the dyes while preserving the glycosides and the information they provide. A sequential extraction procedure with increasing acidity allow extraction of labile weakly bound dyes as well as the more strongly bound dyes.

The chromatogram of turmeric showed 3 characteristic peaks: bisdemethoxycurcumin, demethoxycurcumin and curcumin (Fig. 1c). Some cloths coloured with turmeric which had faded with age often appeared similar in colour to the undyed cloths but these 3 compounds could be detected. The madder chromatogram showed peaks for alizarin and purpurin (Fig. 2a). The tannins produced a broad shoulder on the injection noise at about 4 min.

| Table 2 HPLC gradients |
|------------------------|
| Time (min) | Water (%) | Acetonitrile (%) | 5% H₃PO₄ (%) |
| HPLC gradient 1 |
| 0          | 68        | 30               | 2           |
| 25         | 0         | 98               | 2           |
| 30         | 0         | 98               | 2           |
| 30.1       | 68        | 98               | 2           |
| 35         | 68        | 98               | 2           |
| HPLC gradient 2 |
| 0          | 85        | 10               | 5           |
| 25         | 0         | 95               | 5           |
| 25.1       | 85        | 10               | 5           |
| 30         | 85        | 10               | 5           |
Fig. 1 Typical chromatograms of barkcloth sample extracts. a Untreated noni; b treated noni; c turmeric.
Associated with the high iron content. The 9 undyed con-

ing pigment detected and elevated titanium was

extracts there was some hydrolysis to produce antho-

ate) or inherent in the bark fibres. Burnt coral (calcium

levels < 0.002%. Of the 19 coloured samples tested 13

were negative and 6 positive for iron. Of these 6 samples

4 also had an organic pigment present (2 tannin and 2

traces of turmeric) and only 1 contained iron alone. The

poncho Kew 42861 (Fig. 5c) could not be sampled for

HPLC extraction. Calcium was present in both coloured

and uncoloured cloths up to 1.7%. These levels could be

due to the manufacturing process where some cloths

would have been polished using shells (calcium carbon-

ate) or inherent in the bark fibres. Burnt coral (calcium

hydroxide) can be used to produce the red dyes from

noni but XRF would not distinguish the form of calcium.

The degree of weathering of the ochres indicated by the

ratios of immobile elements (Al and Ti) to more mobile

elements (Si and Fe) might be useful in identifying the

sources of the iron ochre [13] but would require a much

larger dataset than here.

Analysis summary

A table of all the cloths and the colourants found on them

is shown in Table 4 and a summary of all the coloured

cloths is shown in Table 5. From all the analysis carried

out on the coloured cloths listed in Table 4 a small group

of 5 plant derived colourants and 1 inorganic pigment

were all that were found. For 40 samples a single colour-

ant was found while in the remaining 12 samples a com-

bination of up to three colourants were detected. Madder

was found in only two samples on the same cloth. The

noni (morindone) samples were all red whereas the noni

(morindon) were both red and yellow. Noni (morindon)

was used predominantly in combination with other col-

ouring agents. A combination of iron ochre and organic
dyes was found in 4 samples. The ease of extraction of

most of the colourants points to the lack of traditional

metallic mordenting. However, historically tree tannins

were used as mordents [14]. Our findings show (Table 5)

that although tannins were found as a single colourant in

11 cloths they were also found in combination with other

colourants in 9 cloths where they may have been acting as

a mordent.

The reasons for the small number of colourants found

could be that some of these cloths were prepared spe-
cifically as gifts for visitors and the colours and materi-

als that the makers knew would retain their integrity

over time were chosen; these colourants were used in

prestigious cloths which were always intended to be gifts

or for ceremonial uses and crucially like artisans world-

wide experience had taught them that some colourants

although initially bright and vivid quickly faded and

so were rarely used after this discovery. The sequential

extraction procedure showed that noni root was used to

produce colourants based on morindin and morindone.

The colour produced from noni is a stable and true red as

opposed to the more brown/red produced from tree bark

tannins and therefore noni has been frequently used to

create a red colour.

Some colours may have faded so the cloths no longer

appeared coloured and perhaps for that reason they were

not sampled. In other cases the dye residue may not have

been detectable. A number of cloths produced no evi-
dence of colorant but some which appeared pale cream/

yellow and seemed uncoloured when analysed showed

the presence of turmeric. This may be because of a low

application level or the instability of turmeric which is

known to fade significantly due to light [15]. Anthocya-
nins, a widely suggested group of colorants, produce a

variety of vivid colours but their colour is also known to

day. Zaffino et al. [16] reported that analysis of antho-
cyanin samples artificially aged and thus faded could still

be detected.
Fig. 2 Typical chromatograms of barkcloth sample extracts. a madder; b tannins using HPLC gradient 1; c tannins using HPLC gradient 2.
Fig. 3  UV-Vis spectra of a major noni components, b turmeric components, c madder components.
Case studies
In this section 6 cloths have been chosen as case studies as they represent the variations in colorants used in the cloths studied and also the way in which a colorant derived from the same source can have its colour changed by the addition of other compounds. Where details of their origins and donation date to the collections are known this is included. Figure 3 shows 6 cloths.

**EBC 42863**
The red-brown lines on this leaf patterned cloth (Fig. 4a, i), attributed to Samoa, were too thin to sample but samples were taken from a filled in red-brown leaf (Fig. 4a, ii) and the broad yellow stripe. Both samples showed the presence of turmeric with the red-brown area also containing tannin.

**Hunterian E595/1**
Figure 4b (i) shows a cloth, attributed to Tahiti where a red pattern has been painted on a yellow background. It has considerable areas of soiling perhaps caused by it being folded and the top surface left uncovered. The yellow background is coloured with noni (morindin) and the red with noni (morindin) plus tannin, detail shown Fig. 4b (ii). This is the only cloth amongst those analysed that used noni to create both the red and yellow colour.

**EBC 42947(a)**
The yellow background of this ‘handprints’ cloth is turmeric (Fig. 4c, i). It is attributed to Hawaii and donated to the EBC in 1874 by HRH the Duke of Edinburgh. Samples obtained from the curved red stripe in the corner found it was painted with noni (morindin). Although no trace of turmeric was detected suggesting these red curves were painted separately and not over the yellow background from the cloth it is difficult to see how this was done (Fig. 4c, ii). It was not possible to sample the red from the hands (Fig. 4c, iii) but detailed study of the area round them suggests that the red was added on top of the yellow background.

**Unicoloured**

| ID No       | Al (%) | Si (%) | P (%) | S (%) | Cl (%) | K (%) | Ca (%) | Ti (%) | Fe (%) |
|-------------|--------|--------|-------|-------|--------|-------|--------|--------|--------|
| Coloured    |        |        |       |       |        |       |        |        |        |
| E417/11 Red | 0.63   | 2.29   | 0.21  | 0.15  | 0.34   | 0.69  | 0.57   | 0.939  | 1.773  |
| E595 Red    | 5.28   | 6.64   | 0.08  | 0.06  | 0.32   | 0.47  | 0.46   | 0.272  | 4.172  |
| E598/1 Red  | 0.19   | 0.76   | 0.27  | 1.30  | 1.13   | 0.41  | 0.97   | 0.017  | 0.053  |
| E598/1 Yellow | 0.21  | 0.70   | 0.18  | 1.22  | 1.02   | 0.47  | 0.94   | 0.018  | 0.049  |
| E598/2 Red  | 0.26   | 1.14   | 0.38  | 1.28  | 0.75   | 0.42  | 0.92   | 0.016  | 0.057  |
| E598/3 Red  | 0.17   | 0.62   | 0.24  | 1.14  | 1.17   | 0.21  | 1.03   | 0.013  | 0.033  |
| E598/4 Red  | 0.19   | 0.77   | 0.31  | 1.07  | 0.84   | 0.34  | 1.23   | 0.010  | 0.017  |
| E599 Dark brown | 0.12 | 0.41   | 0.11  | 0.23  | 0.06   | 0.04  | 1.62   | 0.046  | 0.333  |
| E600 Red    | 0.18   | 0.60   | 0.30  | 0.81  | 0.30   | 0.04  | 1.10   | 0.021  | 0.046  |
| E602 Red    | 1.08   | 3.04   | 0.50  | 0.52  | 0.39   | 0.45  | 0.44   | 0.330  | 1.031  |
| E602 Yellow | 0.57   | 1.71   | 0.38  | 0.49  | 0.69   | 0.38  | 0.46   | 0.193  | 0.602  |
| E606 Red    | < LOD  | 0.29   | 0.08  | 0.50  | 0.06   | 0.11  | 1.15   | < LOD  | < LOD  |
| E611/3 Grey | < LOD  | 0.07   | 0.03  | 0.33  | 0.97   | 0.05  | 0.46   | < LOD  | < LOD  |
| 42853A Red  | 0.11   | 0.22   | 0.17  | 0.95  | 1.37   | 0.21  | 0.69   | < LOD  | < LOD  |
| 42861 Red   | 0.42   | 1.37   | 0.24  | 0.23  | 0.18   | 0.58  | 0.39   | 0.459  | 1.149  |
| 42947B Red  | < LOD  | 0.06   | 0.15  | 0.29  | 0.44   | 1.00  | 1.69   | < LOD  | < LOD  |
| 42947B Yellow | < LOD | < LOD  | 0.16  | 0.16  | 0.37   | 1.08  | 0.99   | 0.005  | < LOD  |
| 67802A Red  | < LOD  | 0.31   | 0.19  | 0.42  | 1.13   | 0.12  | 1.74   | < LOD  | < LOD  |
| 98041 Red-brown | < LOD | 0.07   | 0.07  | 0.24  | 0.03   | 1.01  | 1.32   | < LOD  | < LOD  |
| Uncoloured  |        |        |       |       |        |       |        |        |        |
| Average     |        |        |       |       |        |       |        |        |        |
| (n = 9)     |        |        |       |       |        |       |        |        |        |
| Max         | 0.190  | 0.858  | 0.289 | 1.499 | 1.185  | 0.463 | 1.042  | 0.022  | 0.048  |
| Min         | < LOD  | < LOD  | < LOD | 0.026 | 0.029  | < LOD | 0.102  | < LOD  | < LOD  |
| LOD         | 0.057  | 0.034  | 0.013 | 0.020 | 0.008  | 0.021 | 0.051  | 0.005  | 0.010  |
### Table 4 Colorants found by HPLC and XRF

| Accession number | Description                                      | Colours sampled | Colourant found |
|------------------|--------------------------------------------------|-----------------|-----------------|
| **CTCTAH sample book** |                                                |                 |                 |
| CTCSB2016 No. 10 | Plain yellow                                      | Yellow          | Turmeric        |
| CTCSB2016 No. 18 | Red/yellow/black pattern                         | Yellow          | Turmeric        |
| CTCSB2016 No. 20 | Plain red                                        | Red             | Noni(T)         |
| CTCSB2016 No. 23 | Plain red                                        | Red             | Noni(T), tannin |
| CTCSB2016 No. 29 | Red/yellow/black pattern                         | Yellow          | Turmeric        |
| **Huntarian museum** |                                                |                 |                 |
| E417/2           | Black/yellow pattern on undyed background        | Yellow          | Turmeric        |
| E417/5           | Black with red border                            | Red             | Tannin          |
| E417/11          | Red zigzags on black background                  | Red             | Tannin, iron oxide |
| E457/3           | Red and yellow stripes on undyed background      | Yellow          | Turmeric        |
| E458/1           | Yellow/black pattern on undyed background        | Yellow          | Noni(U)         |
| E458/3           | Red/brown/black pattern                          | Light brown     | Tannin          |
| E458/2           | Thin red stripes on yellow background            | Yellow          | Tannin          |
| E458/4           | Plain red-brown r                                 | Red-brown       | Tannin          |
| E458/6           | Plain brown and brown/yellow pattern             | Brown           | Tannin          |
| E595             | Red with undyed spots                             | Red             | Iron oxide      |
| E595/1           | Red pattern on yellow background                 | Red             | Noni(U), tannin |
| E598/1           | Red/yellow/black/undyed stripe pattern           | Red             | Noni(T)         |
| E598/2           | Red/black pattern                                | Red             | Noni(T)         |
| E598/3           | Red/yellow/black pattern                         | Red             | Noni(T)         |
| E598/4           | Red/yellow/black pattern                         | Red             | Noni(T)         |
| E599             | Dark brown flaking coating                        | Dark brown      | Tannin, iron oxide |
| E600             | Red border stripe on undyed cloth                | Red             | Noni(T)         |
| E601             | Red, grey and undyed stripes                     | Red             | Noni(T)         |
| E602             | Red/yellow/black pattern                         | Red             | Iron oxide, trace turmeric |
| E603             | Plain red-brown cloth                            | Red-brown       | Tannin          |
| E606             | Plain red and undyed blocks                      | Red             | Tannin          |
| E608             | Plain yellow                                     | Yellow          | Turmeric        |
| E667             | Red/yellow/black pattern                         | Red             | Noni(T)         |
| **Kew EBC**      |                                                |                 |                 |
| Kew 42853/2      | Plain red cloth                                  | Red             | Noni(T)         |
| Kew 42861        | Poncho red/black/undyed pattern                  | Red             | Iron oxide      |
| Kew 42863        | Multi-coloured stripe and leaf pattern on undyed background | Red-brown | Turmeric, tannin |
|                  |                                                  | Yellow          | Turmeric        |
| Kew 42885        | Plain dark brown cloth                           | Dark brown      | Tannin          |
| Kew 42947/1      | Red handprints on yellow background              | Red             | Noni(U), tannin |
|                  |                                                  | Yellow          | Turmeric        |
| Kew 42947/2      | Red stripes on yellow background                 | Red             | Noni(U)         |
|                  |                                                  | Yellow          | Turmeric        |
| Kew 42958(1)     | Red pattern on yellow background                 | Yellow          | Turmeric        |
| Kew 42958(2)     | Red/brown pattern on undyed background           | Brown           | Noni(U), tannin |
|                  |                                                  | Light brown     | Noni(U)         |
| Kew 42958(3)     | Red leaf pattern on yellow background            | Red             | Noni(U), tannin, turmeric |
|                  |                                                  | Yellow          | Noni(U), turmeric |
| Kew 42966        | Mottled red on undyed background                 | Red             | Noni(T)         |
and purpurin indicating natural madder, Fig. 5a (ii). Light microscopy of an area (Fig. 5a, iii) shows the presence of coloured fibres not associated with barkcloth fibres. In the nineteenth century the introduction of dyed cloths to the Pacific Islands through trading enabled makers to incorporate these dyed cloths into the beaten barkcloths. “Turkey cloth” and “Turkey red” are cited as a favoured material used in kapamaking [17, 18]. Arthur et al. [17] describes how the Turkey red cotton fabric was shredded and then beaten into bark fibres resulting in a mottled red cloth which was then cut into shapes and beaten into the top layer of the tapa.

Turkey red cloth is a specific type of cotton cloth dyed with madder and mordented with alum and oil; this process was used by European dyers as early as the late eighteenth century [19]. Extraction solution 2 was used for this sample as the dye was likely to contain a mordant and extraction 1 was not sufficiently strong.

**Table 4 (continued)**

| Accession number | Description                                           | Colours sampled | Colourant found |
|------------------|-------------------------------------------------------|-----------------|-----------------|
| Kew 42967        | Red/purple mottles in triangular pattern              | Red             | Madder          |
| Kew 42979        | Plain glossy red coating                              | Purple          | Madder          |
| Kew 67802        | Red/yellow stripe pattern                             | Red             | Tannin          |
| Kew 73329        | Brown pattern on undyed background, brown tassles     | Brown           | Noni(U), tannin |
| Kew 98041        | Plain red-brown                                       | Red-brown       | Tannin          |

**Table 5 Summary of colorants found in all barkcloth samples**

| Colorant                  | Number of barkcloth samples |
|---------------------------|-----------------------------|
| Single                    |                             |
| Madder                    | 2                           |
| Noni(morindin)            | 4                           |
| Noni(morindone)           | 11                          |
| Tannin                    | 11                          |
| Turmeric                  | 10                          |
| Iron oxide                | 2                           |
| Combination               |                             |
| Noni(morindin) tannin     | 4                           |
| Noni(morindone) tannin    | 1                           |
| Noni(morindin) turmeric   | 1                           |
| Noni(morindin) tannin turmeric | 1                     |
| Tannin turmeric           | 1                           |
| Tannin iron oxide         | 2                           |
| Turmeric iron oxide       | 2                           |

**Hunterian E602**

Figure 5b (i) shows this red, yellow and black diamond patterned cloth and Fig. 5b (ii) shows the pattern in more detail. The red and yellow is produced by differing concentrations of iron oxide. There is a trace of turmeric present in both coloured areas. This cloth is attributed to Hawaii whose geology has red earth (ochre). There was no indication that tannins had been used to create mixtures here as HPLC only detected turmeric in very low levels.

**EBC 42861**

Figure 5c shows a poncho/coat type garment ‘worn as a garment by the natives of Samoa’ (http://apps.kew.org/ecbot/specimen/42861), on which the red and black areas contain aluminosilicate, titanium and iron indicating the presence of an iron ochre pigment. The elevated P in the black diamonds and black area may be indicative of soot. Various nuts such as kukui (*Aleurites moluccana*) are burnt and the soot used to create black [6], the soot can be mixed with water, oil or bark tannins. It was not possible to sample this cloth for HPLC analysis so the presence of any additional dye components could not be investigated.

**Conclusions**

The findings show that despite the numerous colourant sources for the red, brown and yellow shades listed in the many accounts of historic barkcloth making (Kooijman) only 5 types of plant colorants and 1 inorganic pigment used singly or in combination were identified. A similar finding was reported by Bisulca et al. who examined 150 cloths from the Bishop Museum (Hawaii). The mild analytical extraction procedure prevented decomposition of glycosides allowing for a better understanding of the how these colourants were prepared for use. The case studies show the variations in colour that were achieved by the use of these few colourants, either singularly or in combination.
Fig. 4 Images of the case studies cloths: EBC 428633 a (i) and (ii). ES95/1 b (i) and (ii). EBC 42947A c (i), (ii) and (iii)
Fig. 5 Images of the case studies cloths: EBC 42967 a (i), (ii) and (iii). E602 b (i) and (ii). EBC 42861 c (i)
Abbreviations
HPLC: high-performance liquid chromatography; XRF: X-ray fluorescence; MS: mass spectrometer; LC–MS: liquid chromatography mass spectrometer; UV–visible: ultra violet–visible; HCl: hydrochloric acid; M: molar.

Authors’ contributions
HF developed the methodology for the sequential extraction for the HPLC analysis of samples and carried out the HPLC analysis. He also carried out XRF analysis. Collaborated in the writing. MS planned the research, sampled the cloths, carried out light microscopy and assisted with the HPLC and XRF analysis. Collaborated in the writing. JF carried out sampling, light microscopy, assisted with the XRF and contributed to the writing. All authors read and approved the final manuscript.

Acknowledgements
Margaret Smith and Jennifer Brunton were funded by AHRC (Grant Number AH/M00886X/1). Situating Pacific Barkcloth in Time and Place) http://www.tapa.ac.uk. The authors wish to thank Misa Tamura, the project conservation researcher, for her help in sampling and contributing her knowledge on the materials of barkcloth. Fiona Inches of the Royal Botanic Gardens Edinburgh for suppling plant materials. We are very grateful to Aisling Macken and Marika Kesler of the CTCTAH for photographing the cloths. We also wish to thank Ken Granger of Niton UK Ltd for training and loan of the XRF.

Competing interests
The authors declare that they have no competing interests.

Availability of data and materials
Not applicable.

Funding
Margaret Smith and Jennifer Brunton was funded by AHRC (Grant Number AH/M00886X/1).

Publisher’s Note
Springer Nature remains neutral with regard to jurisdictional claims in published maps and institutional affiliations.

Received: 1 October 2018 Accepted: 24 December 2018 Published online: 09 January 2019

References
1. Barrow T. Art and life in polynesia. London: Pall Mall Press; 1971.
2. Neich R, Pendergrast M. Traditional tapa: textiles of the Pacific. London: Thames and Hudson; 1997.
3. Kooijman S. Tapa in Polynesia. In: Bernice P , editor. Bishop museum bulletin. Hawaii: Bishop Museum Press; 1972.
4. Bisulca C, Schattenburg-Raymond L, DuPreez K. Hawaiian Barkcloth from the bishop museum collections: a characterization of materials and techniques in collaboration with modern practitioners to effect preservation of a traditional cultural practice. In: Vandiver P, Li CM, Sciau P, editors. Symposium PP: materials issues in art and archaeology. Boston: Materials Research Society; 2014.
5. Tamburini D, Cartwright CR, Melchiorre Di Crescenzo M, Rayner G. Scientific characterisation of the dyes, pigments, fibres and wood used in the production of Barkcloth from Pacific islands. Archaeol Anthropol Sci. 2018. https://doi.org/10.1007/s12520-018-0745-0.
6. Larsen AK. Evolution of Polynesian bark cloth and factors influencing cultural change. J Anthropol. 2011;130(2):116–34.
7. Wouters J, Grzywacz CM, Claro A. A comparative investigation of hydrolysis methods to analyze natural organic dyes by HPLC-PDA nine methods, twelve biological sources, ten dye classes, dyed yarns, pigments and paints. Stud Conserv. 2011;56(3):231–49.
8. Aobchey P, Sryiam S, Phaharnripoorab W, Lhieochaiphant S, Phutrakul S. Production of red pigment from the root of Morinda angustifolia Roxb. var. scabridula Craib. by root cell culture. Chiang Mai Univ J. 2002;1(1):66–78.
9. Derksen GCH, Naayer M, van Beek TA, Capelle A, Haaksman IK, van Doen HA, de Root AE. Chemical and enzymatic hydrolysis of anthraquinone glycosides from madder roots. Phytochem Anal. 2003;14(3):137–44.
10. Degano I, Ribechini E, Modugno F, Colombani MP. Analytical methods for the characterization of organic dyes in artworks and in historical textiles. Appl Spectrosc Rev. 2009;44(5):363–410.
11. Sanyova J. Mild extraction of dyes by hydrofluoric acid in routine analysis of historical paint micro-samples. Microchem Acta. 2008;162(3–4):361–70.
12. Degano I, La Nasa J. Trends in high performance liquid chromatography for cultural heritage. Top Curr Chem. 2016;374(2):20.
13. Faithful J. Curator of mineralogy and petrology, Hunterian Museum. Glasgow: University of Glasgow, 2018 (Personal Communication).
14. Barnett JR, Miller S, Pearce E. Colour and art: a brief history of pigments. Opt Laser Technol. 2006;38:445–53.
15. Yoshizumi K, Crews PC. Characteristics of fading of wool cloth dyed with selected natural dyestuffs on the basis of solar radiant energy. Dyes Pigm. 2003;58(3):197–204.
16. Zaffino C, Bruni S, Russo B, Pilu R, Lago C, Colonna GM. Identification of anthocyanins in plant sources and textiles by surface-enhanced Raman spectroscopy (SERS). J Raman Spectrosc. 2016;47(3):269–76.
17. Arthur L. Cultural authentication of hawaiian quilting in the early 19th century. Cloth Text Res J. 2011;29(2):103–18.
18. Finnhaber N, Erhardt D. Recent advances in the conservation and analysis of artifacts. In: Jubilee Conservation Conference, London 6–10 July 1987. National Museums of Artifacts, 1986). p. 178–85.
19. Peel RA. Turkey red dyeing in scotland its heyday and decline. J Soc Dye Colour. 1952;68:496–505.