Evidence for the use of madder as a pigment in Nubia

Kate Fulcher

Research on the pigments used in ancient Egypt is well established, but much less work has been conducted on Nubian sites. The paint and painting materials excavated at Amara West have formed the basis of a PhD project, and the investigation of the coloured paintings in the royal Napatan tombs at el-Kurru are ongoing (Therkildsen 2015). However, many sites retain colour on walls or on objects that have not yet been researched, for example K XI in the eastern necropolis at Kerma (Bonnet and Valbelle 2000, 65-101), and the temples at Jebel Barkal.

The site of Kawa (ancient Gematon) is located between the Third and Fourth cataracts, near the modern town of Dongola. The site was occupied from the mid 14th century BC into the 4th century AD (Welsby 2000). A mud-brick three room shrine at the south end of the town dating to the reign of the Kushite king Taharqo (25th Dynasty) 690-664 BC, retained Egyptian style polychrome decoration (Welsby 2000; Plate 1).

Plate 1. The painted shrine, Building A1, at Kawa (photo: SARS NDRS Archive).

Fourteen samples of polychrome decoration were taken from fragments of painted wall plaster from the shrine for analysis at the British Museum. One sample of blue pigment was taken from a pot sherd used as a palette, found in Building A2, a domestic building close to the painted shrine and probably of similar date (Welsby 2002, 37-38).

Red and pink pigments in Egypt

Analyses of red Egyptian pigments have in most cases identified red iron oxides (Blom-Böer 1994; Lee and Quirke 2000). A less commonly used red/orange pigment, from the New Kingdom onwards, was realgar, an arsenic sulphide (Blom-Böer 1994; Daniels and Leach 2004; David *et al.* 2001; Pagès-Camagna and Guichard 2010, 28; Saleh *et al.* 1974; Vandenabeele *et al.* 2009). Pinks in all instances prior to the Ptolemaic Period were red iron oxide mixed with a white, usually gypsum or calcite, but also huntite (Blom-Böer 1994; Scott 2016; Uda *et al.* 2000).

Madder

Madder is an organic dyestuff derived from the roots of the Rubiaceae family, notably the Rubia species (Daniels *et al.* 2014; Eastaugh *et al.* 2004a, 250). It can be precipitated or adsorbed onto an inorganic substrate, commonly aluminium hydroxide or calcium sulphate, to form a pigment, known as a lake (Daniels *et al.* 2014).

Madder was used in Egypt from the 18th Dynasty for dying textiles; numerous examples are known from elite and non-elite contexts (Germer 1992; Wouters *et al.* 1990). Madder pigments in Egypt have been identified on Roman mummy portraits (Cartwright and Middleton 2008), Roman mummy cartonnage (Rowe *et al.* 2010; Scott *et al.* 2009), and in a pot from a Roman tomb at Hawara (Russell 1892). A pink pigment consisting of an organic substance on a gypsum ground was found on a Third Intermediate Period papyrus and tentatively identified as madder (Lee and Quirke 2000, 113). Madder was widely used in the ancient world and has been documented in Greek, Roman, Mesopotamian, and Parthian contexts (Daniels *et al.* 2014).

Research on pigments in Nubia is in its infancy and as yet there are limited sites with which a comparison of pigments can be made. No madder has been found at Amara West, where evidence comes from domestic buildings and coffins; all reds from Amara West are red iron oxide (author's own work). Two samples of reddish pigment from Tombos have been examined, one red from an 18th Dynasty coffin (E091.25), and one pink pigment on a Napatan coffin (M443). The red pigment was found to be red iron oxide, and the pink was red iron oxide mixed with gypsum (author's own work). Reds from the tomb paintings at el-Kurru were examined by X-ray fluorescence and found to be high in iron, and, therefore, likely to be red ochres (Therkildsen 2015). Red pigments from wall paintings in the 1st century AD temple of Amun at Dangeil have been identified as red iron oxide (Sweek *et al.* 2014).

Analysis Methods

Samples taken from the painted walls of the shrine at Kawa were examined by polarised light microscopy (PLM) and infrared spectroscopy (Table 1). PLM is a widely used technique for the identification of historical pigments (Clark 2002; Eastaugh *et al.* 2004b; Kakoulli 2002). A small sample of pigment is dispersed on a microscope slide in Meltmount™, refractive index of 1.662, and covered with a cover slip. It is then observed in plane polarised light and cross polarised light. More detail on the method is given by Eastaugh *et al.* (2004a and b). The technique allows the entire mixture of grains in the sample to be observed if a representative sample has been taken, unlike point analysis which will provide information about a single grain.

Infrared spectroscopy (IR) has frequently been used for the analysis of ancient pigments (Aliatis *et al.* 2009; Genestar and Pons 2005; Vahur *et al.* 2016). IR measures the energy absorbed by the sample from a beam of infrared electromagnetic waves. Covalent bonds within atoms absorb the IR energy and vibrate, giving each molecular structure a unique absorption spectrum (Stuart 2004). By comparing the spectra of an unknown substance with a spectra library, inorganic and organic substances can be identified. Samples were analysed using the attenuated total reflection (ATR) accessory (Vahur *et al.* 2016).

Results

Results are shown in Table 1. The plaster ground is gypsum (calcium sulphate). The yellows were all found to be ochres, and the blacks carbon. Frequently calcite (calcium carbonate) was present in the paint, up to 10%, estimated by approximate particle count; calcite can occur naturally in association with gypsum and ochres (Eastaugh *et al.* 2004a). Blue from the shrine wall and from the sherd with pigment from A2 were both identified as Egyptian blue. Of the five areas of red paint sampled, four were red ochre, and the other (PS524) was found to be madder.

The IR spectrum for PS524 aligned well with other madder samples from reference databases (Figure 1), and shows distinctive peaks for aluminium hydroxide, which is the substrate to which the dye is applied in order to create a pigment (Mazzocchin *et al.* 2003, fig. 5). PLM is not conclusive for madder, but PS524 in plane polarised light showed translucent pink crystals as would be expected for a red lake pigment on an inorganic substrate (Plate 2; Eastaugh *et al.* 2004b, 358-9). Madder fluoresces orange-pink under UV light, whereas other red organic dyes do not (Plate 3; Daniels *et al.* 2014; Schweppe and Winter 1997). PS524 was found to fluoresce in this manner (Figure 1), confirming this pigment as madder lake.

Discussion

Madder as a dyestuff is well attested from Egypt from the 18th Dynasty onwards, but evidence of its use as a pigment has been limited to the Roman Period, with perhaps one example on papyrus from the Third Intermediate Period. This paper presents the first identification of madder used as a pigment in a wall painting in Nubia or Egypt, and is a rare example of a madder pigment in this region prior to the Roman Period.

The other pigments identified in the shrine are usual for Egyptian wall paintings (El Goresy 2000; Lee and Quirke 2000). It is interesting that madder is used alongside the much more common red ochre. Samples were taken from very fragmentary pieces of mud plaster, too small to identify the subject matter. It is possible that madder was used only for specific details, and red ochre in other areas. Madder tends to have a more pink appearance than ochre, which may explain the use of both pigments, which also occurs on Roman mummy masks (Rowe *et al.* 2010). Given that madder is only used in small areas, and that sampling is prohibited at Egyptian sites, it is possible that madder has been under-identified in Egypt.

Neither the known pigments from Nubia nor those from contemporary Egyptian wall paintings include madder. Until more research is completed on paintings from Nubian sites the question must remain as to whether the use of madder at Kawa is a Nubian, or more localised, adaptation. Either way, it may provide evidence of the adaptation of an Egyptian

Table 1. Results of analyses of pigments from Kawa.

Paint colour	Pigment identification	Type of object	Analysis performed	Context	Sample number
Black	carbon	painted mud plaster from shrine	PLM	Kawa A1 Room I (AB5)59	PS508
Black	carbon	painted mud plaster from shrine	PLM	Kawa A1 Room I (AB5)59	PS515
Blue	Egyptian blue	painted mud plaster from shrine	PLM	Kawa A1 Room I (AB6)24	PS519
Blue	Egyptian blue	pigment from sherd	PLM	Kawa A2 (AD5)126	PS523
Red	iron oxide	painted mud plaster from shrine	PLM, FTIR	Kawa A1 Room I (AB5)59	PS509
Red	iron oxide	painted mud plaster from shrine	PLM	Kawa A1 Room I (AB5)59	PS510
Red	iron oxide	painted mud plaster from shrine	PLM	Kawa A1 Room I (AB5)59	PS517
Red	iron oxide	painted mud plaster from shrine	PLM	Kawa A1 Room I (AB5)59	PS518
Red	madder	painted mud plaster from shrine	PLM, FTIR, UV	Kawa A1 Room I (AB6)24	PS524
White	gypsum	painted mud plaster from shrine	PLM	Kawa A1 Room I (AB5) 59	PS514
White	gypsum	painted mud plaster from shrine	PLM	Kawa A1 Room I (AB5)59	PS516
White	gypsum, calcite	painted mud plaster from shrine	PLM	Kawa A1 Room I (AB6)24	PS521
Yellow	iron oxide	painted mud plaster from shrine	PLM	Kawa A1 Room I (AB5)59	PS507
Yellow	iron oxide	painted mud plaster from shrine	PLM	Kawa A1 Room I (AB5)59	PS511
Yellow	iron oxide	painted mud plaster from shrine	PLM	Kawa A1 Room I (AB6)24	PS522

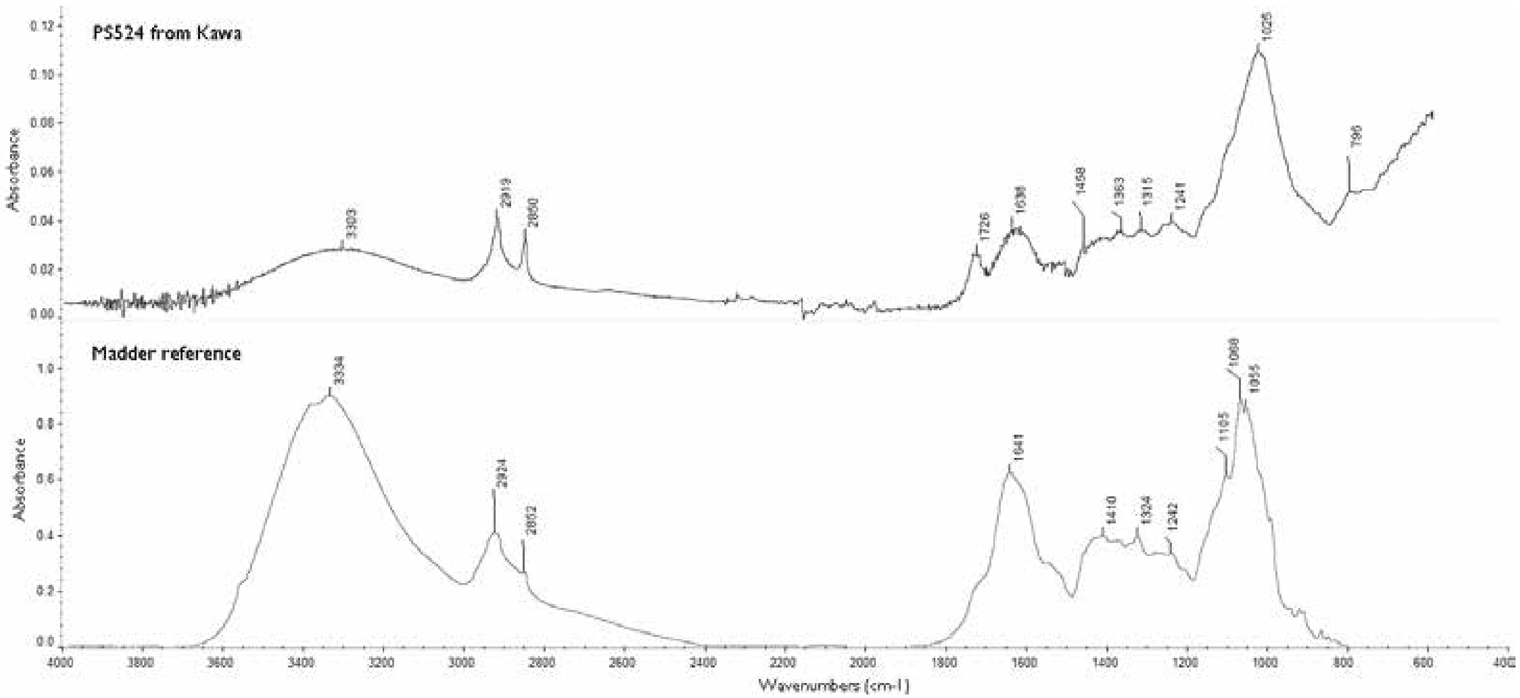

Figure 1. IR spectrum of PS524, peaks correspond well to those from the reference sample IOD00202 Philadelphia Museum of Art (Infrared and Raman Users Group 2017).

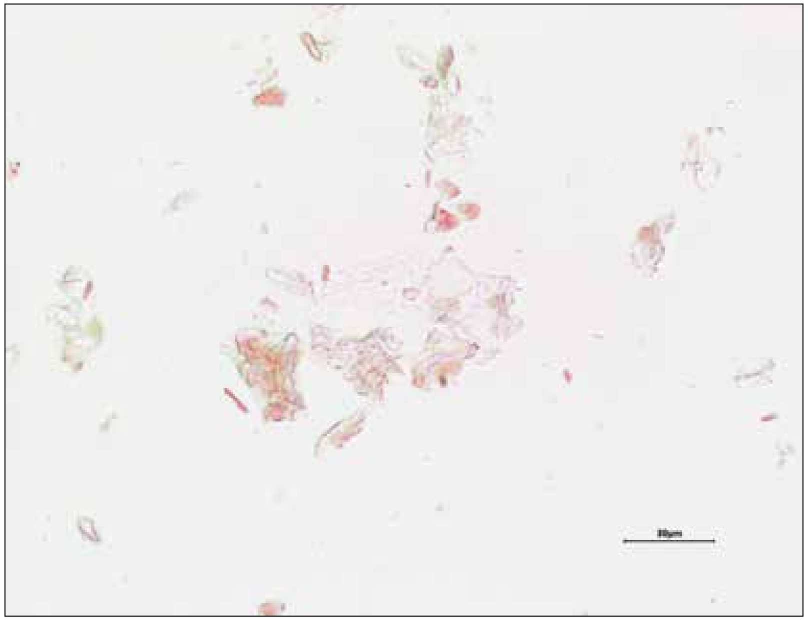

Plate 2. Dispersion of PS524 viewed in plane polarised light x400.

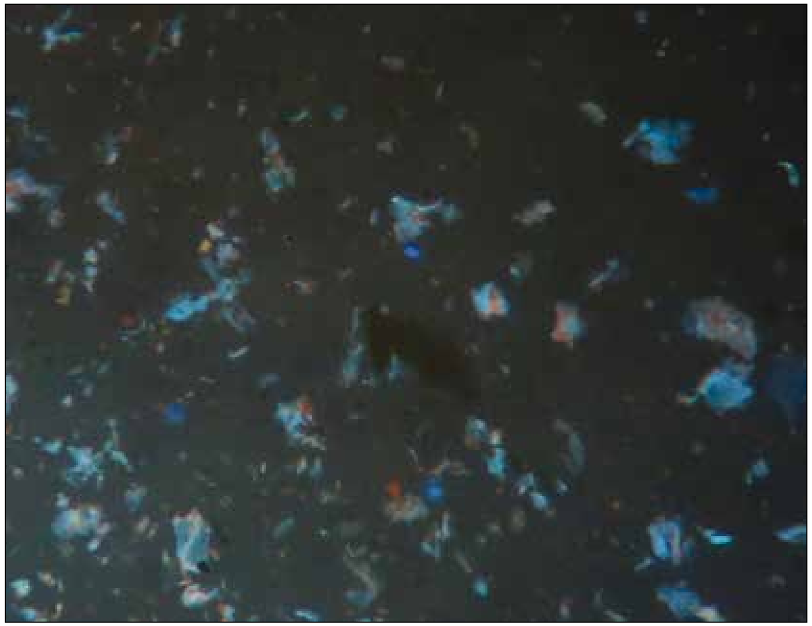

Plate 3. PS524 under UV light at x400. Orange-pink fluorescence identifies the pigment as madder lake on a substrate.

style wall painting by a Kushite king, using non-Egyptian materials.

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