THE COWISHT PROJECT: ENHANCING THE IDENTIFICATION OF ARTEFACT RAW MATERIALS

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ABSTRACT

The detection of universal patterning in the way animals were utilised for artefact production is reliant on the correct and confident identification of the raw materials. Associations between classes of artefacts and very specific materials can make a significant contribution to understanding the thinking behind that particular class of object. Unfortunately expectations based on assumptions of a likely suite of materials can be wrong. This paper discusses the outcomes of a three year project; Cultural Objects Worked in Skeletal Hard Tissues (COWISHT), aimed at developing, evaluating and validating identification criteria and techniques for often very highly worked and decayed materials. This project included the identification of bone, antler, ivory, horn, baleen, tortoiseshell and rhino horn. This paper also presents an introduction to the identification web resource, Visualising Animal Hard Tissues, which has been designed to disseminate the results of COWISHT. This resource uses cutting-edge scanning and imaging techniques, combining 2D images and 3D models, to aid the identification of these materials in their raw, worked and decayed states.

KEYWORDS: Raw material selection, identification, osseous, keratinous, cultural significance.

RESUMEN

La detección de patrones universales en la manera de utilizar animales para la producción artefactual está basada en la correcta y confiable identificación de las materias primas. Las asociaciones entre clases de artefactos y materiales muy específicos pueden contribuir significativamente a comprender las ideas detrás de una particular clase de objeto. Lamentablemente las expectativas basadas en la identificación material pueden ser equivocadas. Este trabajo discute los resultados de un proyecto de tres años; Objetos culturales trabajados en tejidos esqueletales duros (COWISHT en inglés), dedicado a desarrollar, evaluar y validar criterios de identificación y técnicas para el estudio de materiales que frecuentemente se encuentran muy deteriorados y trabajados. El proyecto incluye la identificación de hueso, asta, marfil, cuerno, ballena, caparazón de tortuga y cuerno de rinoceronte. Este trabajo presenta también una introducción a la identificación de un recurso web, Visualising Animal Hard Tissues, diseñado para divulgar los resultados de COWISHT. El mismo utiliza tanto escaneo de corte y bordes, como diferentes técnicas de imágenes, combinando imágenes 2D y modelos 3D para facilitar la identificación de materiales en estado natural, trabajado y/o alterado.

PALABRAS CLAVE: Selección de materia prima, identificación, óseo, queratina, significado cultural.

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INTRODUCTION

Knowing the date, provenance and context of an object might be of little use in understanding its cultural significance if there is uncertainty about the material from which it is made. The material may have been used purely because it was readily available and its shape, strength and working properties were adequate for purpose. Alternatively the choice may have been dictated by less tangible reasoning such as tradition and belief systems or to express group identity, power or status. Identification of the animal derived raw materials to skeletal element, species and even sex can be a relatively simple matter where there is extensive retention of natural morphological features on the worked object that allows comparison with reference specimens. The more heavily the surfaces are modified the more difficult it becomes to obtain such precise identifications. This paper presents results from a project that has tested the potential and limitations of imaging and compositional analysis techniques for the material identification of osseous and keratinous hard tissues used to make artefacts.

The underlying factor that leads to many misidentifications of cultural objects in animal hard tissues is our lack of familiarity with these materials when worked. Many of them are no longer used for the commercial production of objects in the developed economies and have been replaced by the use of metals or plastics. The harvesting of others, such as elephant ivory, tortoiseshell and rhino horn have become unacceptable and illegal as conservation efforts struggle to conserve threatened and dwindling populations. The problems of identifying these animal hard tissues are increased by the range of natural variation between individual animals (Fig 1a and b). Population genetics, age at death, sex, disease, trauma and diet can all affect the features of these tissues, for instance, determining shape, limiting size, altering colour, varying the ratio of related features and even influencing the material’s microstructure. Different stages of surface working and prolonged exposure to light may produce further changes in appearance and those materials that are relatively porous will readily absorb grease, particulate soiling and stains through handling and use (Fig 1c and d). Manufacturing techniques that change the natural surface colour, texture or translucency of these materials (e.g. painting, staining, bleaching,

Figure 1. Transverse sections through elephant ivory tusks. (a) Ivory with clear Schreger pattern, (b) ivory with colour banding and indistinct Schreger pattern towards the tusk centre, (c) sawn (below) and fracture surfaces, (d) staining in wet storage. (a,b, and c, University of Bradford, d, kind permission Ken Hawley Collection Trust). Photos S. O’Connor.
dressing with waxes and oils and polishing) or enable it to be deformed into new shapes (e.g. demineralisation, heating and pressing,) confuse the issue further. Sometimes these processes are applied deliberately to create features that imitate those of a more desirable material. Examples recognised by SO’C in historical or archaeological collections have included bone scales from clasp knife handles carved and stained to resemble the natural surface of red deer antler beam; hippopotamus tusks carefully shaped to imitate small elephant tusks; oriental sword hilts in elephant ivory textured with fine dimples to give the whorled appearance of walrus ivory secondary dentine; cattle horn bleached, pressed and dyed to produce combs with the amber and brown dappled appearance of a ‘tortoiseshell’ and cattle horn moulded and mounted on a pink coloured ground to form a dagger hilt of the same shape and characteristic glow of others executed in rhino horn. Whether the original intention was to produce a cheap imitation or to commit fraud by fakery often cannot be determined but the result is that all these objects had been misidentified.

Wet working conditions may promote decay of the organic components, leaching or adding to the inorganic components. However it is the archaeological environment that can be the most transformative, accelerating deterioration that can affect everything from colour and texture to strength, hardness and flexibility. Factors as diverse as the mineralogy, pH, redox potential, level of oxygenation, temperature, moisture content, water permeability and organic content of the deposits and even the presence or absence of other artefactual material, such as different types of corroding metal, will largely determine the state of preservation of the material or whether it survives at all (Fig 2). The osseous tissues are highly mineralised and the rigid and intimate organisation of the organic (collagen) and inorganic components (hydroxyapatite) provide mutual protection; limiting biodeterioration and conferring chemical stability over a wide range of pH conditions (Smith et al. 2007). In contrast keratinous materials, such as horn, have a low mineral content and the protein is far more susceptible to rapid biodeterioration so rarely survives except in frozen or desiccated environments or where micro-organism activity is chemically inhibited.

A wide range of techniques have been used to confirm the identification of these materials. The
COWISHT project was designed to evaluate these techniques and assess their relative merits. Correct visual identification remained the cornerstone of this process and the validation and development of visual identification criteria and the dissemination of this knowledge and skill were a major part of the project. Although specific examples are presented here, this paper does not set out to be a comprehensive and definitive guide to hard tissue identification, and the author’s research in this field is ongoing.

CULTURAL MATERIALS WORKED IN SKELETAL HARD TISSUES

In 2010 the author received Science and Heritage Programme funding from the UK Arts and Humanities Research Council and the Engineering and Physical Sciences Research Council for a three-year post-doctoral project entitled Cultural Materials Worked in Skeletal Hard Tissues (COWISHT). The aim of this project was to advance the confidence with which the raw materials derived from vertebrates could be identified following artefact manufacture, use and deterioration in a range of environments. Objectives included the validation and development of visual criteria and the evaluation of available analytical methods with the emphasis on non-destructive or minimally destructive techniques.

The core materials of the study were to be the vascular osseous materials, mostly mammal bone and antler; osseous non-vascular materials, including ivory derived from elephant, hippopotamus, walrus, narwhal and sperm whale, and other large teeth such as killer whale and the tusks of pig and dugong; and keratinous hard tissues, such as horn, hoof, baleen, tortoiseshell, rhino horn and hornbill. Inevitably in characterising these materials it was also necessary to consider other biomaterials, such as coral and shell, which in a decayed state might be confused with these vertebrate tissues and the mineral, natural organic and synthetic materials that over time have been used as substitutes and imitations, or to produce fakes.

The project started with a survey of the scientific papers related to the development, morphology, structure, chemistry and deterioration of each of the materials and the techniques that have been used in the identification of archaeological, historical and modern specimens. Concurrently, and throughout the whole project, several thousand specimens of animal hard tissues, worked, damaged and decayed objects of known material, were examined and recorded. Of particular interest was how the state of preservation might enhance or obscure specific identification criteria or change the characteristic chemistry of these materials. The knowledge gained was applied to the critical evaluation of published identification criteria and analytical techniques and, where possible, the results of each method or criterion were verified by the application of complementary techniques.

The project was undertaken with the aid of six UK partner institutions. The Hawley Collection, Sheffield; the Horniman Museum, London; Hull Museums and Galleries; Leeds Museums and Galleries; The York Archaeological Trust and the Henry Moseley X-ray Imaging Facility at the University of Manchester. The museums provided access to thousands of worked and unworked examples of these materials, dating from prehistoric times to the present day. The Horniman Museum, Leeds Museums and Hull Museums provided extensive natural history collections (including whole and sectioned specimens) and complementary social history and ethnographic collections. The ethnographic objects were largely handmade but the social history collections, being mainly 19th and early 20th century, were mostly machine worked. Hull Maritime Museum was particularly important in providing a rich marine component to these raw materials and objects. Between them, these museums provided examples of all the target materials and much more.

The Hawley Collection was unique in being entirely devoted to the industrial history of Sheffield, particularly the world-renowned manufacture of cutlery and tools in steel. Handle making was one of the industries and many men were once employed as specialist ivory cutters. Industrial decline of the steel industry in Sheffield in the last century led to the rapid closure of factories and
workshops, many of which had been established for generations. Ken Hawley recognised not only the need to collect examples of the finished articles and the equipment and materials with which they were made but also unfinished objects to document the stages involved in their manufacture. As a result the collection houses hundreds of examples of blanks, preforms, working waste and finished handle components for cutlery, razors, tools, walking sticks and umbrellas. A diverse range of materials were used including cattle and whale bone, European and Asian deer antler, elephant ivory, cattle and buffalo horn and tortoiseshell, as well as mother of pearl, exotic woods and early plastics. In addition, there were examples of raw materials from the workshop stores, some clearly discarded because of blemishes (healed traumas, pathologies and post mortem damage). Others perhaps had been trialled but proved difficult to work or were not available in consistent quantity or quality for mass production as they did not feature amongst the finished pieces. Some may have been kept purely as curiosities. These materials included giraffe bone, mammoth tusk, hippopotamus, walrus and narwhal ivory and sperm whale teeth. The York Archaeological Trust, Leeds Museums and the Hull and East Riding Museum also provided access to extensive archaeological collections, mostly from the UK, with the majority of sites being in Yorkshire. Despite the narrow geographical distribution of these sites, the range of materials and their states of preservation were very diverse. The best preserved osseous and keratinous objects came from the deep waterlogged urban sites, such as York. These stagnant, anoxic deposits inhibited biodeterioration and favoured the survival of both the protein and mineral components of these tissues. On the better-drained sites in the ice-contact sediments typical of the Vale of York, the osseous materials were still relatively well preserved but, due to biodeterioration, the evidence for keratinous materials was limited to traces preserved in metal corrosion products (MPO remains = mineral preserved organic remains) Even when horn does survive it is frequently misidentified as wood (Fig 3). Elsewhere extremes of sediment pH on the fast
draining Chalk of the Yorkshire Wolds and the sandstones and grit of West Yorkshire produced very poorly preserved osseous finds due to alkali degradation of the protein and acid degradation of the mineral component respectively.

The sixth project partner, the Henry Moseley X-ray Imaging Facility, provided the expertise and equipment for micro and nano computed tomography (CT) of the raw materials. This strand of the project would evaluate the potential of micro CT techniques for the non-destructive identification of the material of objects through the high resolution 3D imaging of their internal structure. The differentiation of bone and different species of ivory involved imaging structures in the range of 5 to 100 μm. This was feasible for samples of a few millimetres in diameter but was not achievable for larger objects as the image resolution was progressively degraded as the thickness of the material increased, through which the X-ray beam passed.

As the project developed, many more museums, archaeologists, researchers, private collectors and heritage scientists provided objects for examination, commissioned material identifications or became collaborators on particular research aspects. Visits to 23 museums and private collections across the UK were undertaken to examine rare survivals, such as archaeological baleen, or to broaden understanding of particular materials, such as rhino horn, hornbill and non-elephant ivories. Materials identifications were undertaken of finds from more than 30 archaeological sites from across the UK, from Shetland to Exeter, and from Denmark, Hungary, Mali and the Philippines. Each object provided another learning opportunity, extending our understanding of the behaviour of materials in different burial conditions and how this affected the visibility of its characteristic features or produced changes to its chemistry.

**VISUAL IDENTIFICATION**

The limitations of identification guides

Most identification guides are concerned with elephant ivory and its substitutes, such as bone and large teeth from other species (Espinoza and Mann 1992; Penniman 1952; Thornton 1981), and make little mention of the keratinous hard tissues. These guides deal principally with natural surface morphology and or the cut and polished sections of natural history specimens. This limits their use for the identification of cultural material that cannot be destructively sampled or where these details are obscured or lost through working and decay. Krzyszowska (1990), O’Connor (1987) and Rijkeltijkhuizen (2008), deal specifically with the identification of worked archaeological objects. Each is limited to the description of materials from quite particular burial environments: O’Connor and Rijkeltijkhuizen include keratinous tissues. The identification guides written for antiques and jewellery collectors also concentrate on the features visible on the surfaces of worked objects but the majority of these guides are too simplistic and also contain a lot of misinformation that has become established ‘truth’ through repetition from one unreferenced source to another. Exceptions to this are Campbell Pederson (2004) and Locke (2013). Campbell Pederson’s work is well observed, mostly accurate and beautifully illustrated but the text lacks references to the scientific literature. Locke’s book, like his earlier scientific paper on the structure of ivories (Locke 2008), concentrates to a great degree on these materials from the viewpoint of a biologist and material scientist, whilst often lacking a clear account of the detail observable by eye, or under low magnification. Each of these books is of limited use for less than perfectly preserved archaeological material.

A general limitation of all these guides is that they can only show ‘typical’ examples of the materials they discuss. The illustrations and descriptions in Penniman’s classic identification guide (Penniman 1952), for instance, are based on very few specimens of each material and there is really no discussion of the natural range of variability of these features.

Refining visual identification

Visual identification is an evidence-based, polytypic process. Individual criteria may very strongly suggest a particular material but confirmation comes from the observation of a
combination of features that together are unique to a material. To improve and develop visual identification first an in-depth understanding of the formation, chemistry and structure was gained of each material. Combined with the examination of many natural history specimens and sections and, sometimes, hundreds of worked and decayed objects, this provided a comprehensive picture of their characteristic features. An archive of over 23,000 images of these objects has been produced, that captures the range of variation in structure and form that might be expected, common pathologies and other anomalies, and the effects of different working techniques or deterioration on the appearance of these features. This has greatly overcome the restrictions of working with the limited resources of an identification guide or sections of prepared specimens and has been invaluable both in confirming the identification of problematic material and in disseminating the knowledge acquired through publications and workshops.

The first stage of the identification process is to look for a ‘grain’ in the material, that is, to search for any alignment of natural features that might indicate the basic structure of the material (e.g. vascular, lamellar, cone-within-cone,) and its orientation to the skeletal element from which it is cut (e.g. the long axis of a long-bone, tusk or horn). Once this is done microscopy can confirm the finer details of these features and show whether changes in their appearance on surfaces at different orientations fit the model predicted by one material or another. For the keratinous materials the next stage is to observe the presence, form and distribution of natural pigmentation which can be very specific to different materials. Lastly the characteristics of damaged and decayed surfaces can provide corroborative or even decisive information confirming identification. For ivories, the most telling features are often the patterns of deterioration. Cracking, staining and the texture of fracture surfaces, on the macro, meso and micro scale, can be very characteristic of a particular ivory as they reflect the underlying differences in histology and patterns of mineralisation of the dentine of different species (Figs. 1 and 2).

Equipment for visual identification
To the eye, many of these materials can look remarkably similar but even with a little magnification (20x to 40x), in good lighting conditions, the details revealed can lead to identification. The equipment used in this project was versatile, portable, of moderate cost and consisted of a Wild Heerbrugg M8
stereomicroscope with a Meiji Techno FT190/240 fibre optic, halogen light source with dual gooseneck light guides; a Panasonic Lumix DMC TZ6 16 megapixel compact camera and a small folding photographic booth with two table-top LED lights; and a Dino-Lite AM-7013MZT, 5 megapixel digital microscope with polarizer (Fig 4). The stereomicroscope had a long depth of field and working distance and was mounted on a long armed stand that made it possible to examine objects of all sizes and shapes. The camera was selected for its excellent macro facility and its good low light sensitivity which meant that it could be used to record whole objects and detailed evidence of a material’s structure, even when working in the sub-optimal conditions of a museum store. By simply holding the camera to an eyepiece of the stereomicroscope, it could also be used to record photomicrographs. This ad-hoc arrangement did not produce photomicrographs of particularly good quality but they were useful to add to the image archive that was produced for each of these materials. Photomicrographs of publication quality were captured using the small digital microscope and viewed in real-time on the project laptop. The limitations of this digital microscope, such as the limited magnification options and narrow depth of field, meant that it could not always be used when the surface of the object was not reasonably flat. One particular advantage of this microscope, however, was the integrated polariser that could be used to remove surface sheens that otherwise obscured the underlying structure of the material. Adjusting the polarizer helped to distinguish structural details from surface working marks (Fig 5).

The most crucial element of this equipment was the lighting. The ubiquitous ceiling mounted, fluorescent strip lighting in museum stores is often only just sufficient to illuminate a working environment but inadequate for close work. This also produces a very diffuse light that forms little in the way of shadow and tends to hide textures, fine cracking and subtle variations in surface reflectance. The two LED lamps produced a pool of bright light in which the direction and depth of shadow could be controlled by changing the positions of the lamps relative to each other and the object. These lamps were useful both for photography and for close observation of the objects, particularly when initially exploring and mapping the grain or macro features of the material prior to microscopy. The most important and versatile light, however, was the dual guide optic fibre lamp. Although this variable intensity light is made specifically for microscopy, it is also very useful for macro photography and for illuminating the interior spaces of objects. Each guide has a focusing lens that can be adjusted to form a sharply defined, narrow column of light or removed to provide a wide diffuse field. The goosenecks allow the lights to be oriented in any direction so that the material can be illuminated with incident or transmitted light. Many of these materials are translucent to some extent and changes in their structure can affect the direction of transmission of the light in ways that can be very informative, as illustrated by the walrus ivory in figure 6. The light guides also allow the angle of the incident beam to be tightly controlled. Tilting an object in this beam can remove or enhance the visibility of internal or surface reflections. These

Figure 5. Photomicrographs of a carved and polished cetacean bone object (private collection) taken with the Dino-Lite AM-7013MZT digital microscope. (a) unpolarized light, (b) polarised light. Photos S. O’Connor.
reflections can both hide or enhance the visibility of structural features. Other optical techniques evaluated included scanning electron microscopy (SEM) and confocal laser scanning microscopy (CLSM). Without destructive sampling both SEM and CLSM can only be used on small objects or fragments. SEM is widely used in materials identification and produces high resolution images of topographic features. CLSM, although lower resolution, reproduces both the colour and the topography of a surface, allowing 3D reconstructions of complex features, although reflectance, fluorescence and variations in the translucency of the material can produce imperfections. This project showed, however, that the ability to resolve minute detail is not always particularly useful in resolving the identification of a material. Both bone and antler are vascular tissues and these blood vessels can easily be seen under low magnification. The most distinctive differences in the organisation of these tissues are at the macro, not the micro, level (Ashby 2013) so observing the detail of the canaliculi in the cortical tissue at very high resolution will not help to distinguish them. The distribution, path and cross-section of the dentinal tubules in different species of ivory are distinctive (Locke 2008; Thornton 1981). At 5 to 10 μm in diameter SEM can be used to image these features but identification by comparison with the published data, gained from thin or polished sections, can only be attempted with any confidence if the exact orientation of the observed surface can be established. Conventional radiography, computed tomography (CT) and micro computed tomography (μCT) were also evaluated. Radiography provided some information about internal voids and constructional information but these features were captured more clearly in the sections and 3D visualisations reconstructed from the CT data. Exploring the finer structure of the material required μCT but the resolution of detail was restricted to a great extent by the size of the object; the image resolution degrading as the size of the object increased. Published studies (Enzmann et al. 2008; Reiche et al. 2011) show something of the potential of these techniques for osseous materials but reveal little that might not be observed through surface microscopy of the worked objects and specimens. Where an objects exterior is decayed or obscured μCT may be useful in separating bone/antler from ivory or horn from tortoiseshell but more work needs to be done with greater numbers of specimens of known material (species, skeletal element, state of development) before the identification of ivory species or the separation of terrestrial mammal bone from cetacean bone or antler, for instance, can be achieved on the basis of μCT evidence.

Figure 6. Handle YAT2006.5201.8524 in walrus ivory. (a) incident light, (b) transmitted light revealing the structure of the whorled secondary dentine. Photos S. O’Connor (kind permission York Archaeological Trust for Excavation and Research).
alone.

SEM and CLSM, radiography and CT can all provide additional information that may resolve questions raised by visual examination but there is no virtue in bypassing this first stage and going straight to a ‘high-tech’ solution if the material identification can be achieved by ‘low-tech’, low risk, low cost optical microscopy. At worst, observation of greatly magnified or internal details without an understanding of the wider context can lead to misidentification.

Limitations of visual identification

The refinement of the visual criteria enabled the identification of some thousands of worked objects of unknown material in the collections of the project partners and collaborators to material type and with enhanced separation of skeletal elements and taxa but rarely to species. For instance, distinguishing antler from bone indicates some species of deer and even when the surface morphology is lost, observation of the features and relationship of the cortex and medulla may allow the distinction of species (Ashby 2013). Similarly, the Schreger pattern is a distinguishing characteristic of elephant ivory (Epinosza and Mann 1992:10) but the COWISHT project has shown that it is often possible to identify ivory as elephant from a combination of lesser features when this pattern cannot be directly observed. On the basis of the outer Schreger angles it is possible to separate elephant ivory from mammoth ivory (Espinoza and Mann 1992: 11-13) but only when the edge of the tusk can be confirmed by the presence of the cementum covering (Fig 7). It is not possible, however, to identify the species of elephant from Schreger angle measurements as the ranges in Asian elephant (Elephas maximus) ivory and the two African elephant species (Loxodonta cyclotis and Loxodonta africana) overlap.

Unless deterioration is so extreme that the microstructure of the material is lost altogether, damage and decay often acted to enhance the visibility of the characteristic features of the material. The major limiting factor to identification is size as the smaller an object, the less evidence it can encapsulate and less can be inferred from it.

CHEMICAL APPROACHES TO IDENTIFICATION

The chemical analytical approaches applied to cultural materials identification have always been restricted to those deemed as ‘non-destructive’. 

Figure 7. Transverse sections through the tusks of (a) mammoth ivory and (b) elephant ivory (University of Bradford). Towards the centre of the elephant tusk the angles produced by the crossing lines of the Schreger pattern are similar in size with the outer Schreger angles of the mammoth ivory. Photos S. O’Connor.
If ‘destruction’ includes the removal of even microscopic amounts of material or the physical and chemical changes caused by bombarding an object with beams of high energy particles or electromagnetic radiation, then no chemical analysis can be truly non-destructive. In practise the techniques that are generally seen as acceptable in the heritage sector are those that can be applied without the need for surface preparation or the removal of visually obtrusive samples. These techniques are often termed ‘non-invasive’ but a more honest description would be ‘minimally destructive’. This effectively limits these techniques to ones that detect only surface or near surface composition where contamination and deterioration can be at its most significant.

Evaluation of these techniques was achieved through a comprehensive literature survey and collaboration with appropriate research groups and targeted experimental work. Only the most notable techniques are discussed here.

Micro-Proton Induced X-ray and Gamma-ray Emission (micro-PIXE/PIGE) has been used to determine the chemical composition of the mineral component of osseous tissues enabling the distinction of terrestrial from marine mammal material and bone from ivory (Müller and Reiche 2011; Vercoutère et al. 2011). The separation of modern reference specimens was achieved on the relative abundance of minor and trace elements and the method proved robust for historical material. However, the diagenetic changes (mineral loss and addition) produced by long-term burial prevents the separation of bone from ivory. Although the marine/terrestrial distinction appears more robust, it is doubtful that it would be valid across material from different archaeological environments.

Vibrational spectroscopic analytical techniques, such as FT Raman spectroscopy and various forms of FTIR, have been far more widely applied to the identification of these materials. (e.g. Brody et al. 2001; Edwards and Farwell 1995; Edwards and O’Connor 2012; Edwards et al. 1998; Espinoza et al. 2007; Turner-Walker and Xu 2014; Welsh et al. 2012). In producing spectra characteristic of both the micro structure and molecular chemistry of the organic and inorganic components of a material vibrational spectroscopy can, for instance, reliably separate osseous from keratinous tissues and their synthetic imitations, and osseous materials from other natural materials such as shell. It has also been used to separate bone from ivory and mammalian keratin from keratin formed in birds and other reptiles.

The use of vibrational spectroscopy for species identification has proven much less reliable, despite the application of chemometrics and statistical analysis. The results show some success in acquiring characteristic spectra of modern raw materials, such as different species of ivory, and using these to confirm the identification of historical objects. Ivories of different species produce spectra that are essentially very similar and it is mostly quite subtle detail that allows them to be identified to species. Some of these differences, however, may be significantly affected by diet, the age and health of the individual or the time that has lapsed since death. Aging over time and the chemical and structural changes produced by working (e.g. thermal degradation, chemical denaturing, dressing with organic oils, fats or waxes and soiling during use) will further compromise the detectability of these differences. Certainly more work with greater numbers of samples of securely identified material is needed to determine that the results are separating species and not just individual specimens. For archaeological material, the result of deterioration in different archaeological environments (e.g. biodeterioration, hydrolysis, leaching, demineralisation, contamination from mineral species carried in the groundwater) can make the chances of getting a meaningful identification almost negligible (Edwards and O’Connor 2012; Edwards et al. 2006). The results of work during the COWISHT project showed not only significant intra-site variability between fragments of mammoth ivory but huge variations between the spectra gained from opposing surfaces of the same fragment (O’Connor et al. 2011).

On their own these chemical approaches to identification are less useful and reliable than visual identification techniques and should not be used as a front-line approach. Where they can be useful is when the material’s microstructure is
obscured or insufficient to confirm, for instance, if a material is ivory or shell, tortoiseshell or plastic. If the microstructure is lost through decay, however, the same processes of deterioration may also make the chemical identification less specific and less reliable. Used together, the visual and chemical evidence can be more powerful in distinguishing problematic materials. For instance the open structure of cetacean bone can look very similar to antler but these can be separated on their F and Sr contents, which are significantly higher in marine material (Müller and Reiche 2011).

BIOMOLECULAR DETERMINATION OF TAXA

aDNA analysis can, of course, provide species identification for all these osseous and keratinous materials. However, despite continued developments reducing both the cost and sample size needed, this is still a relatively destructive technique. Contamination of DNA from external sources is a serious issue and the physical condition of the material is not always a good indicator of the state of preservation of its DNA so there is a risk that apparently well preserved material will not yield a meaningful result.

Fortunately ZooMS (Zooarchaeology by Mass Spectrometry), which was developed as a cheap, fast technique for the identification of bone fragments from archaeological sites (Buckley et al. 2009) is proving to be a useful alternative to aDNA analysis. This technique exploits the differences in the amino acid sequences that form the peptides of bone collagen in different taxa and these differences should be greater the more the species have diverged in evolutionary terms. Extraction, digestion and analysis of the collagen provide a peptide fingerprint that is then compared with a rapidly growing library of spectra from known species. The identifications obtained from ZooMS are less specific than DNA and are usually to genus rather than species level. It is possible to identify pig, goat, sheep or elephant, but not the species of elephant. It is possible to differentiate red deer from reindeer but not red deer from elk (Hounslow et al. 2013).

Three advantages of ZooMS over DNA are that collagen in bone is relatively more stable than DNA, contamination is less of an issue (unless an object has been consolidated with a collagen-based glue) and the sampling is minimally destructive. Samples of approximately 10-30 mg were used in the identification of unworked bone (Welker et al. 2015) and soaking techniques were briefly tested for objects (Hounslow et al. 2013). The latter approach was less invasive than cutting or drilling a sample and the buffered solutions used extracted the collagen but protected the mineral component of the bone. During the COWISHT project SO’C collaborated with Matthew Collins and the University of York BioArch researchers on a number of projects relating to the evaluation and development of these sampling techniques, particularly the risk of dimensional changes and cracking when ivory is wetted. Today, dry, minimally destructive techniques, originally developed by Sarah Fiddyment for sampling parchment documents, are being used on bone and ivory objects to great effect. The technique involves lightly rubbing a small area of the surface with an eraser and analysing the particles picked up by the eraser (Fig 8). The diffuse nature of the sampling means that comparison of before and after photographs rarely record any changes to the area sampled. This collaboration also led to the exploration of peptide fingerprinting for the identification of archaeologically preserved keratinous materials and proved very effective even when the structural preservation of the material was very poor (O’Connor et al. 2014). Often visual identification is sufficient to identify material to the level required but it can also raise new questions that can only be answered by the appropriate chemical or biomolecular analytical techniques. The ability of DNA analysis and ZooMS to identify taxa is beyond anything that can be achieved through visual identification techniques but that is also their limitation. They can separate sperm whale from reindeer from cattle but cannot distinguish sperm whale bone from sperm whale ivory, reindeer antler from reindeer bone or cow hoof from cow horn. The only way to separate these material types is by
understanding their characteristic structures. Biomolecular analysis without the guidance and validation of visual identification can lead to the wrong conclusions but together they form a most powerful combination.

OUTCOMES

Keratinous tissues are less durable than osseous materials and rarely survive in archaeological contexts. There is less literature relating to their structure and identification and fewer sectioned specimens of known species than of osseous materials available in museum collections for examination. COWISHT was particularly successful in addressing this area and O’Connor et al. (2014) present recent developments in both visual and biomolecular identification of historical and archaeological material.

Another focus was on the identification of non-elephant ivories. All the teeth of the hippopotamus’ anterior dentition were studied in detail and sometimes smaller objects were found to be made from the ‘peg’ teeth (lower canines and lower lateral incisors) rather than the larger tusks. Sperm whale teeth proved to have a far greater variation in shape and size, both between and within individual sperm whales, than identification guides such as Penniman (1952) or Espinoza and Mann (1992) indicate. These variations include the colour of the dentine, the extent of colour banding and thickness of the cementum covering, the absence/presence and distribution of denticles (Penniman’s ‘nodules of secondary dentine’ (1952: 27)) and the shape and extent of the pulp cavity.

The study of cetacean bone and its differentiation from both antler and bone of large terrestrial mammals was another productive area of study that, combined with these new insights into sperm whale ivory and its deterioration in very different burial environments, revealed a significant pattern of material selection in the UK (Fig 9). SO’C’s examination of finds from High Pasture Cave, Gurness and Mine Howe for Fraser Hunter at the National Museum of Scotland confirmed that the majority of the dagger pommels and guards from these Iron Age sites were cetacean bone and ivory. It is not surprising that coastal communities of Atlantic Scotland would exploit marine mammal resources but as the project progressed a different picture began to emerge. The evidence suggested that cetacean material was being preferentially selected for the manufacture of dagger and sword handle components even by communities that had no close connection to the coast. A study of 15 Early Bronze Age pommels for the Leverhulme-funded project ‘Examination of ritual & dress equipment from British Early Bronze Age graves’ showed that over 30 % (6) were definitely cetacean bone (O’Connor 2015). This was particularly remarkable as this material otherwise rarely featured in the associated finds assemblages (Woodward and Hunter 2015). If ZooMS could be applied to all these pommels the percentage of cetacean materials could potentially rise considerably as a further two were probably
bone from small cetacean species and three, which could only be examined from photographs, were possibly cetacean bone. The author also confirmed the identification of the Bronze Age pommel from Grishtorpe, Yorkshire, as cetacean bone (Sheridan et al. 2013) and that from Ashgrove, Scotland (Henshall 1963-1964), as sperm whale ivory. The acid degraded (gelatinised) pommel of the Bronze Age dagger from Forteviot, Scotland, had previously remained unidentified despite SEM and ZooMS studies but the author was able to identify this too as sperm whale ivory from surviving traces of its structure (Cameron et al. 2013). Further evidence of this preference for cetacean material for the handles of cutting-edge weapons was furnished by the use of both sperm whale bone and ivory in three of the five Iron Age swords from South Cave, East Yorkshire, UK (O’Connor 2013a). Taken together with other published examples and the identification of sword handle remains examined at York, this association can be seen to persist, with the exception of Roman military equipment, from the early Bronze age at least into the early Medieval period.

Amongst the assemblages from archaeological sites it was relatively common that highly decorated objects were erroneously assumed to be ivory whilst plain featured ivory objects were labelled as bone. Form and function were also not infallible indicators of material and for every gaming die in elephant ivory there were others in walrus ivory and many more in bone. ‘Bone’ beads from Hungate, York, turned out to be coconut shell and ivory beads from the Sahara Desert site of Tadmekka in Mali, were identified as a mix of shell and coral from their structural features. Similarly, minute beads found in a Chalcolithic burial at Great Comard, Suffolk, UK that were thought to be sperm whale ivory, were identified by the author as shell. This identification was verified by Richard Telford at the University of Bradford Analytical Centre using Raman Spectroscopy that showed the layers of differing structure and opacity were calcite and aragonite. Subsequent to this the beads became the focus of a novel research project with colleagues at the University of York. The aim was to gain a taxonomic identification of the shells used for the beads with a view to exploring their origins. The study combined light microscopy, SEM, stable isotope analysis, amino acid analysis and Raman spectroscopy to great effect (Demarchi et al. 2014).

Identifications of osseous and keratinous tissues that were undertaken of some of the
archaeological material has already been published as contributions to research papers, site monographs or synthetic volumes (e.g. O’Connor 2013b; O’Connor 2014; O’Connor 2015; Sheridan et al. 2013) and others will follow but most of the identifications undertaken are in the grey literature. The unpublished reports of objects in museum collections will enhance the value and accuracy of future exhibitions and catalogues whilst the CITES appraisals will help to ensure that museum loans will safely pass between countries without risk of confiscation or destruction. Reports undertaken for the police have already led to one successful prosecution related to illegal trafficking of these protected materials. In all these publications and reports, the evidence that supports the identification is always presented and, where possible, this is accompanied by photographic verification of the key features. This should allow other specialists working in this field to have confidence in the identification or to be able to reassess the conclusions reached in light of future developments in this field.

Disseminating the results of this work to help improve the quality and accuracy of material identifications, particularly undertaken by heritage professionals, was a major objective of the COWISHT project. To some extent the papers published and in preparation will address this but the funding body would not support the production of a major identification manual as part of the project. They were happy, however, to support a series of two day identification workshops. These workshops combined lectures and practical sessions using SO’C’s photographic archive and extensive teaching collection, supplemented by natural history specimens and worked objects from the collection of the host institution. Each workshop was delivered to up to 30 participants, depending on the number of microscopes available at the venue, and the content was continuously updated as the project progressed. These continuing professional development courses have remained very popular events and the author has continued to deliver them 2 to 3 times a year since the end of the project.

VISUALISING ANIMAL HARD TISSUES

Teaching is a two way experience and the author gained many insights into the way different people learned identification skills. Correcting the catalogue entries for large museum collections also highlighted common mistakes and misconceptions that threw light on the thought processes that had led to these mistakes. It became clear that errors of scale in the size of expected features was a frequent cause of misidentifications and that many people experienced problems when trying to understand these complex three dimensional structures from two dimensional images. Both difficulties can be overcome by developing a reference collection of worked objects and unworked specimens of known materials but this is a solution available to very few. To address this problem it was proposed to create an interactive website resource for the 3D visualisation of the structures of these animal hard tissues. The plan was to combine laser scanning, CT scanning and texture photography of raw and worked materials to produce fully rotatable, photo-realistic models and to link these to explanatory texts, diagrams, photographs and photo-micrographs of the identification criteria from the COWISHT archive.

In 2012 funding for the development stage of this project, Visualising Animal Hard Tissues (VAHT) was gained through the AHRC Science and Heritage Research Development Awards. This was a collaboration between Archaeological Sciences and the Centre for Visual Computing, a team that had developed its expertise in designing 3D web resources with the JISC funded ‘From Cemetery to Clinic’ (http://www.barc.brad.ac.uk/FromCemeterytoClinic/) and ‘Digitised Diseases’ (http://www.digitiseddiseases.org/alpha/) projects. The principle investigator for VAHT was Andrew Wilson and the co-investigators were Robert Janaway, Hassan Ugail and SO’C. The Hull and Leeds Museums and the York Archaeological Trust were the project partners and the objects selected for scanning from their collections were identified from the COWISHT archive to represent the major osseous and keratinous tissues. The protocols and expertise in producing the highly
detailed photo-realistic 3D models from the laser scan data and texture photographs (high resolution photographs taken of all the surfaces from different angles) had been well established by the scanning team in their previous work to record human bone pathologies to a standard that captured clinically diagnostic detail (Fig 10). However the materials and objects selected for VAHT presented some new and unanticipated challenges. Objects with sharp cut edges and surface sheen or materials with variable translucency, could all produce errors in the laser scan data. Sometimes the sheer size of the scan data that was produced by a single object, for instance that generated by a 2.6 m long narwhal tusk, required days of computing time to reconstruct the surfaces. Where these issues persisted alternative techniques were employed to provide the 3D framework, such as CT and structured light scanning, or, when all else failed, an object movie was created. For selected objects the photo-microscopy of surface details taken during the COWISHT project was supplemented by Z-stack photography, which combines multiple images to produce a single image with an enhanced depth of field. Additional micro-CT images of prepared material samples were also commissioned to complement the information presented in the line diagrams of the tissue structures.

Running alongside this was the development of the web resource’s structure and functionality. The aim was to produce an open access virtual learning environment with flexible and varied output that would work on any platform that supported WebGL (a royalty-free web standard application program interface for 3D graphics). As in the Digitised Diseases web resource, the photo realistic models were fully rotatable and could be zoomed-in to explore the surface detail and texture but in addition there were ‘hot spots’ at critical points on the surface of the model that linked to additional resources, such as photo-micrographs of the materials’ structure in different orientations, explanatory diagrams, CT images or object movies. Zoomify (http://www.zoomify.com/) was also used to facilitate interactive viewing of the high resolution 2D images. This meant that even

Figure 10. Acquiring 3D data from an elephant ivory object with a Faro Quantum arm laser scanner. Photo S. O’Connor.
the largest size photo-micrographs took no more
time to load than small, low resolution images but
could be zoomed and panned to explore the
smallest of details. Another aspect of the resource
was to show how the virtual 3D worked objects
related to the raw materials by orienting the image
of the object within that of the raw material.
The development funding enabled the 3D
visualisations and additional 2D imaging of all the
objects loaned by the project partners. From
these resources and images from the COWISHT
archive storyboards were drafted for each of the
materials and the web pages for elephant ivory
and rhino horn were brought to completion. The
rhino horn pages are available as a pilot for the full
web resource at http://www.3dbones.org/visual/
preview/introduction.php. There are bugs to be
dealt with and comments from users are invited. It is hoped that with the support of Bradford
Visualisation (http://bradford-visualisation.com)
these issues will be resolved and the pages for the
remaining materials will gradually be populated
to provide the resource as originally proposed.

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