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Tracking consolidant penetration into fossil bone using neutron radiography•

A.S. Schulp[1,2,3,*], R. Schouten[4], L. Metten[5], A. van de Sande[5] & A. Bontenbal[6,]**

1 Natuurhistorisch Museum Maastricht, De Bosquetplein 6-7, 6211 KJ Maastricht, the Netherlands
2 Faculty of Earth and Life Sciences, Vrije Universiteit Amsterdam, De Boelelaan 1085, 1081 HV Amsterdam, the Netherlands
3 Naturalis Biodiversity Center, P.O. Box 9517, 2300 RA Leiden, the Netherlands
4 Earth Sciences, University of Bristol, Wills Memorial Building, Bristol BS8 1RJ, UK
5 Institute for Energy, Joint Research Centre of the European Commission, P.O. Box 2, 1755 ZG Petten, the Netherlands
6 NRG, Petten, P.O. Box 25, 1755 ZG Petten, the Netherlands
* Corresponding author. Email: anne.schulp@maastricht.nl
** Retired

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Abstract

In the conservation of fragile fossil bone material, impregnation by solvent-borne consolidant is often required. Understanding the mode of penetration of consolidants into fossil bone is of crucial importance. It is governed by a variety of factors; neutron imaging is a powerful tool to monitor and visualise this penetration (non-destructively). The consolidation of a vertebrate fossil from the Maastrichtian of the southeast Netherlands was imaged at the High Flux Reactor facility at Petten, the Netherlands. The analysis shows current conservation practice to result in a sufficiently deep and isotropic penetration.

Keywords: Consolidant penetration, neutron imaging, fossil bone, conservation

Introduction

Fossil bones often emerge surprisingly well preserved after having been buried for thousands or even millions of years. By contrast, during or following excavation, it can take only seconds to a few decades at most for the same fossil to deteriorate rather rapidly – at least when no appropriate conservation measures are taken. Application of consolidants will alter the physical properties and interferes with the chemical integrity of the specimen concerned. This would generally render the extraction of biomolecules and sampling for radiocarbon dating or stable isotope analysis useless. Even though these considerations call for restraint in the use of consolidant, the fragile nature of the fossil bone will often require the use of consolidant anyway (Davidson & Alderson, 2009).

Of particular interest is the question under which conditions the consolidant may be carried back to the bone surface by the evaporating solvent. This may result in the unwanted buildup of a concentrated layer of consolidant on the surface of the fossil, while generally a deep and isotropic penetration is desirable instead (e.g., Newey et al., 1992; Słupik, 2000, 2001). Such a layer of concentrated consolidant on the surface of a fossil may show a tendency to flake off, along with the surface layer of the bone. This can be a particularly important issue in the treatment of subfossil bone, as an uneven distribution of consolidant may actually accentuate the differences in mechanical properties between cortical (surface) and trabecular (internal) bone. It is, therefore, important to understand the factors involved in the distribution of consolidant and the development of these unwanted surface buildups, in order to

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evaluate the use of an adhesive/solvent system in various contexts and to understand better and assess quantitatively the various approaches in mitigating this effect.

Various methods have been suggested in order to reduce this consolidant buildup (or its underlying cause, see Newey et al., 1992, fig. 7.3), including wrapping the consolidant-solvent-soaked object in aluminium foil leaving only a small hole for the solvent to evaporate very slowly (e.g., Słupik, 2001; Ohlídalová et al., 2006; Macgregor, 2009). The use of non-solvent based consolidants, such as the reaction-adhesive cyanoacrylate, is a way to circumvent this problem altogether, although cyanoacrylates have other disadvantages, including the fact that their long-term stability has not yet been evaluated satisfactorily (Down & Kaminska, 2006).

Here we use neutron radiography (e.g., Hameed et al., 2009) to assess and visualise the penetration characteristics when using ethylmethacrylate/methylmethacrylate copolymers in the treatment of vertebrate fossils (see e.g., Lopéz-Polín et al., 2008, p. 540 and references therein; Larkin, 2010, p. 236 and references therein for two case studies). Ethylmethacrylate/methylmethacrylate (EMA/MMA) copolymers, marketed under brand names such as 'Paraloid B-72' and 'Osteo-Fix' are adhesives commonly used in palaeontological conservation, both as adhesive ('glue') and – in a (much) more diluted form – as penetrating consolidant (Koob, 1986). Our research focuses specifically on the treatment of a characteristic type of vertebrate fossil, well represented in Dutch museum collections and therefore relevant from our perspective: a mosasaur (extinct marine reptile) vertebra from the Late Cretaceous of Maastricht (age ca 66 Ma).

Physical sectioning, microscopic evaluation and mechanical testing of the sections of a fossil provides a quick, affordable and, by definition, destructive assessment of the penetration characteristics of a solvent-based consolidant system. Other approaches, some equally destructive, in tracing consolidant penetration include Raman spectroscopy (e.g., Ohlídalová et al., 2006), or doping the solvent with a radio-opaque material, followed by X-ray analysis (e.g., Cnudde et al., 2009). In the latter approach the initial properties of the consolidant concerned are compromised and the penetration is measured only indirectly; results are therefore potentially less informative.

In addition to being non-destructive, neutron imaging has the advantage of providing a much more quantitative, spatial impression of the actual distribution of the consolidant (Cnudde et al., 2007; Hameed et al., 2009). Because of its non-destructive nature it could potentially also provide better insight into the effect of multiple applications of consolidant. As neutrons are particularly well attenuated by hydrogen (see e.g., Hameed et al., 2009 for more background information), many consolidants become clearly visible in otherwise neutron-radiolucent materials such as the fossil considered here.

Material and methods

As a proof-of-concept, a mosasaur vertebra was partially consolidated and imaged using the High Flux Reactor facility (HFR) at Petten, the Netherlands.

Consolidant

EMA/MMA copolymers are available in various formulations of various molecular weights; we have used the formulation commercially available as 'OsteoFix', one of the brands widely used in palaeontological conservation and for many years the preferred consolidant at the Natuurhistorisch Museum Maastricht. Osteo-Fix beads (Fig. 1C) were dissolved in industrial-grade acetone, at roughly 10-15% EMA/MMA by volume, and generously applied using a brush on one side of the vertebra. The fossil was left to dry to air for a few weeks at the HFR facility prior to preparation of radiographs.

Imaging

The high attenuation of neutrons by hydrogen makes it imperative to have the fossils as dry as possible prior to imaging. Both fossils were stored in climate-controlled museum collections at ca 50% RH prior to making the radiographs. Air RH at the HFR facility is controlled at 55%. Neutron Radiographs (field of view 230 mm) were made using Sub-Thermal Neutrons, flux $7.88{\cdot}10^9/m^2s$ with a reactor power of 45 MW; exposure time 50 minutes, using a gadolinium backscreen.

Results and discussion

Neutron imaging of an acetone/EMA/MMA system applied on a Late Cretaceous mosasaur vertebra from the Maastricht area has shown the buildup of consolidant due to capillary drag on the outer surface to be rather limited. Penetration of the consolidant, poorly visible and difficult to assess on a physical section, turns out to be effective. A single coating with EMA/MMA adhesive diluted down to a thin, ca 10-15% consolidant, reached down to well over 20 mm into the porous bone, and, more importantly, after the evaporation of the solvent, remained there. Earlier research by Hameed et al. (2009), on the consolidation of building stones using EMA/MMA (Paraloid B-72), had already shown that in dense sandstone, a concentration of 5% was already too thick to achieve a homogeneous distribution; the buildup of a crust is clearly visible in their figure 3b. Application of an even thinner solution (1%) yielded better results.

These results confirm that the ideal concentration for conservation purposes has to be evaluated on a case-by-case basis. A larger sample size and additional experiments could potentially yield a more quantitative assessment. Ideally, such a series of tests should allow a perfect balance between

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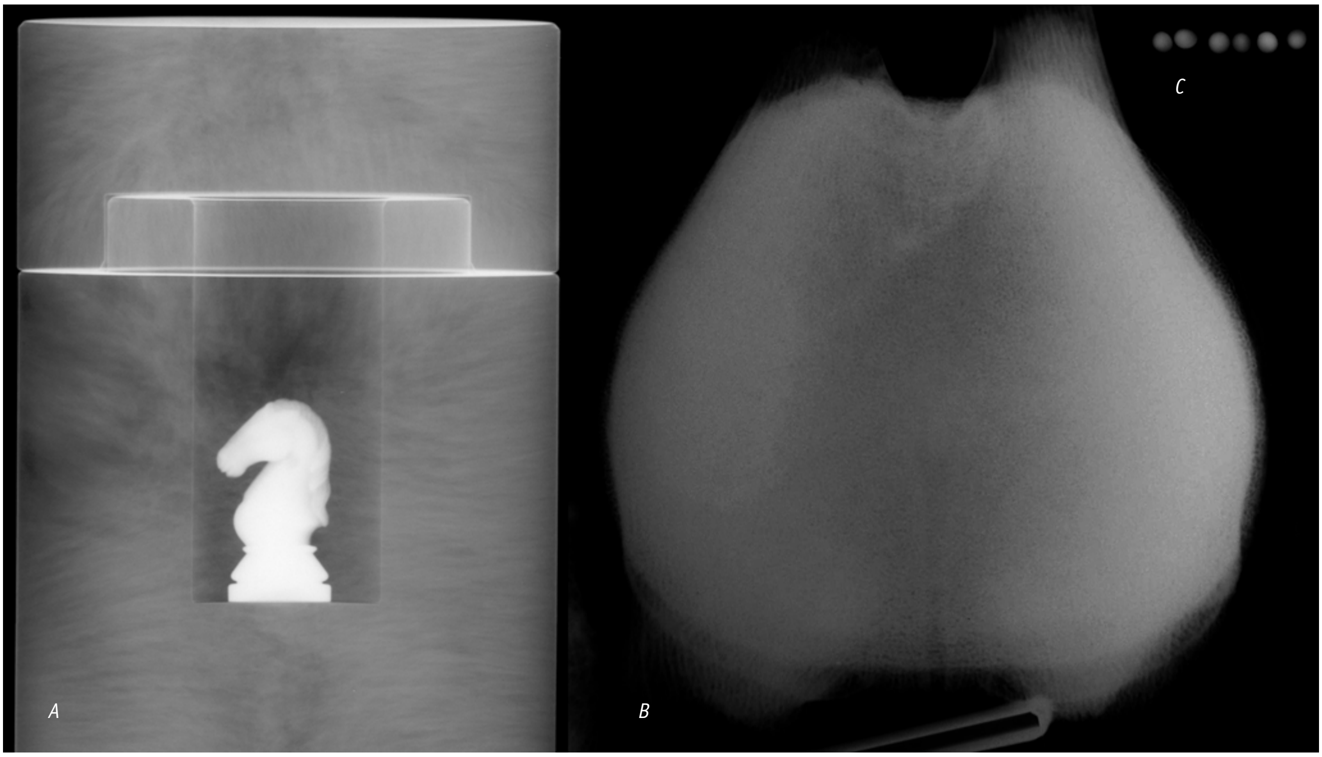

Fig. 1. A. Most metals have little, if any, interaction with neutrons; however, hydrogen bonds attenuate a neutron beam, as illustrated in this neutron radiography: a plastic chess piece in a lead container is clearly visible, with the lead container almost fully transparant and the plastic mostly opaque; B. Neutron radiography of a mosasaur vertebra (in anterodorsal view), impregnated with consolidant on the right-hand side; note the contrast between both sides; C. Single beads of EMA/MMA consolidant, only several mm thick, are clearly visible on the neutron radiograph, indicating the considerable neutron attenuation by EMA/MMA.

penetration effectiveness (i.e., thinner = low viscosity solutions) and amount of consolidant delivered (i.e., thicker, more concentrated solution). As far as Maastricht fossils are concerned, our research shows a concentration of ca 10-15 % to be sufficiently thin as to guarantee a reasonable penetration. We assume the sweet spot to be at different concentrations for each and every single type of fossilisation. Of note in this context is the contribution by Fedak (2006), which provides a simple way of measuring the concentration of consolidant in laboratory settings, relevant to make a quick adjustment when the solution has been in long use and may have thickened due to evaporation of the solvent.

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