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Exploring the potential of the strontium isotope tracing system in Denmark

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Migration and trade are issues important to the understanding of ancient cultures. There are many ways in which these topics can be investigated. This article provides an overview of a method based on an archaeological scientific methodology developed to address human and animal mobility in prehistory, the so-called strontium isotope tracing system. Recently, new research has enabled this methodology to be further developed so as to be able to apply it to archaeological textile remains and thus to address issues of textile trade.

In the following section, a brief introduction to strontium isotopes in archaeology is presented followed by a state-of-the-art summary of the construction of a baseline to characterize Denmark's bioavailable strontium isotope range. The creation of such baselines is a prerequisite to the application of the strontium isotope system for provenance studies, as they define the local range and thus provide the necessary background to potentially identify individuals originating from elsewhere. Moreover, a brief introduction to this novel methodology for ancient textiles will follow along with a few case studies exemplifying how this methodology can provide evidence of trade.

The strontium isotopic system

Strontium is a member of the alkaline earth metal of Group IIA in the periodic table. Strontium is a trace element whose ionic radius is 1.13 Å, very similar to that of calcium 0.99 Å (Faure 1986). Due to their similar ionic radii, strontium can replace calcium in mineral lattices and thereby is incorporated into the skeletal tissues and body through diet. Strontium is concentrated in Calcium-bearing minerals such as hydroxyapatite, the mineral with which tooth enamel is composed. This is one of the characteristics that make strontium useful to archaeologists.

Moreover, strontium has four naturally occurring isotopes ^{88}Sr (82.53%), ^{87}Sr (7.04%), ^{86}Sr (9.87%), and ^{84}Sr (0.56%) (Faure 1986). Three of the four naturally occurring isotopes of strontium are 'stable', whereas ^{87}Sr is radiogenic and is therefore variable, as it is partially formed by the radioactive decay of naturally occurring ^{87}Rb (half-life of 48.8 billion years) (Faure 1986). The strontium isotopic tracer system relies on the use of two of the four 'naturally occurring' isotopes, ^{87}Sr and ^{86}Sr. The ratio of these isotopes, ^{87}Sr/^{86}Sr, is somewhat related to their natural abundance and is often ~0.7 (~7% ^{87}Sr/~10% ^{86}Sr).

In general, what needs to be kept in mind is that age and the type/nature of rocks are parameters which control the strontium isotopic composition of a geological basement and its sedimentary derivates. These strontium isotopic properties are maintained in recent processes (i.e., the strontium isotopic signatures are not changed in – from a geological perspective – very small time spans, due to the very slow decay of 87-Rubidium) and can be followed through processes of weathering, and on into the food chain, through the diet as previously mentioned (Figure 1). Even though differences in the strontium concentrations may occur along such uptake chains (due to different partitioning of strontium into various media such as water, plants, bones, and so on), strontium isotopes are not significantly fractionated, and strontium isotopic compositions are not affected and thus remain 'stable', making these signatures highly useful for tracing. In conclusion, the rate of production of ^{87}Sr is so slow that the ^{87}Sr/^{86}Sr ratio of a substance can be considered invariant over archaeological timescales. Thus, in terms of strontium delivery to a plant, the groundwater inherits ^{87}Sr/^{86}Sr ratios characteristic of the soluble 'biologically available' fractions/components in a soil which, in turn, are transferred isotopically unchanged and unfractionated on to the plant (Benson *et al*. 2006) and throughout the food chain. Therefore, the skeletal tissues of, for example, a sheep, will reflect the bioavailable strontium isotope characteristics of its feeding ground.

The bioavailable strontium

The strontium isotopic ratio which is incorporated in living organisms and thus characteristic of a particular catchment area can be affected by other exogenous factors which can contribute to the strontium budget of a soil

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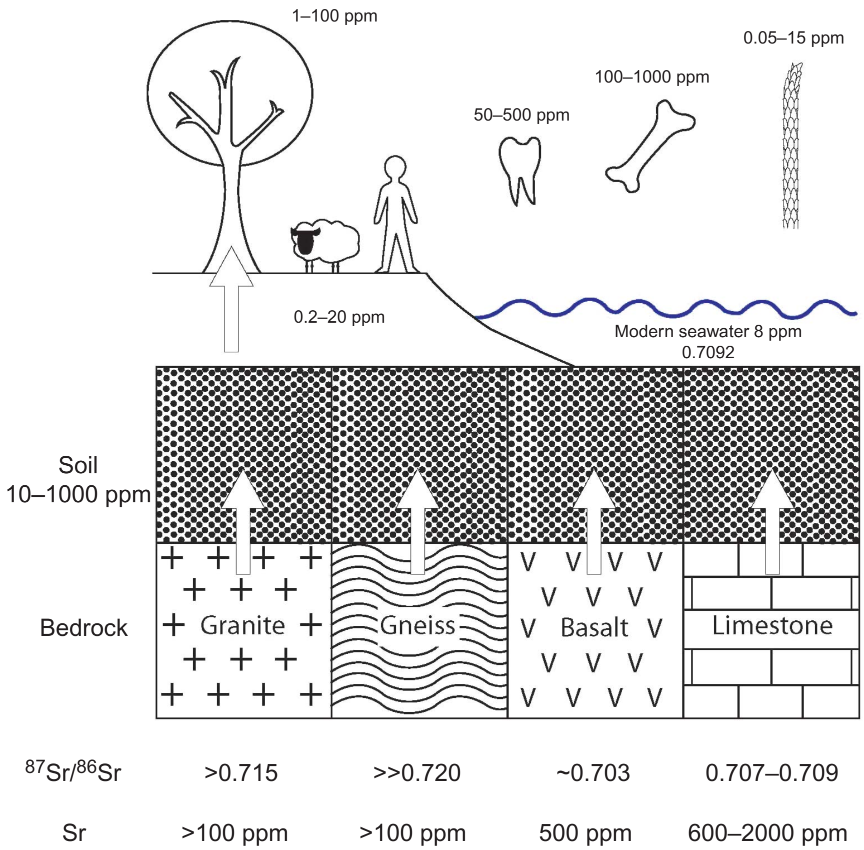

Figure 1. Diagram illustrating the strontium path from the geological strata to the human/animal hair (Frei 2010).

(Price *et al*. 2002). The exogenous factors can be several: some of the most important are seawater spray, atmospheric dust, diet, and modern fertilizer (Frei and Frei 2011).

(1) Seawater contains strontium with a strontium isotopic ratio of $^{87}Sr/^{86}Sr$ ratio of ~ 0.7092 (Figure 1), which has remained unchanged through the archaeological/historical/present timescale. Places like the Faroe Islands or Iceland which are surrounded by seawater have shown to be highly affected by this factor (Price and Gestsdottir 2006, Frei *et al*. 2009a, Frei 2010). This means that the sea-spray contributes to the total strontium budget of a soil when near the coast. Therefore, this is an important issue that needs to be considered in Denmark.
(2) Fertilizers have also often been suspected as being potential contaminants of soils. Recent studies aiming to show the actual effect that modern fertilizers have on soils have demonstrated that concentrations of strontium in fertilizers utilized in Denmark are often so small that their effect is minimal and often not even measurable (Frei and Frei 2011).
(3) Atmospheric particles, for example, ash from volcanic eruptions or sand particles from the Sahara can contribute variably depending on the topography, wind direction, forest canopy, distance to the shore, as well as the nature of the particles. Regrettably, there is little information available on this topic, thus a more detailed evaluation is needed to better estimate their potential effect. However, it is suspected that the influence will be minimal, unless there is a large and continuous deposition of atmospheric particles on the soil.
(4) Diet is also an important factor to consider when interpreting the results of the strontium isotopic analysis, as it can be misleading when high amounts of seafood have been consumed throughout an individual's childhood. Since seawater has a $^{87}Sr/^{86}Sr$ value of ~0.7092, this will affect the strontium isotopic composition of a person living off seafood. Thus, in prehistoric periods when seafood was known to constitute an essential part of the diet, it is highly recommended to perform $\delta^{13}C$ measurements of the same individuals (same tooth if possible) to estimate the percentage of possible strontium deriving from seafood in relation to terrestrial food.

In conclusion, there is no doubt that some of these exogenous factors may contribute in various degrees to the bioavailable strontium isotopic composition of the soil or diet. Therefore, it is important to create bioavailable strontium baselines and not only consider the local basement geology.

Baselines

There are several ways in which baselines of the bioavailable strontium isotopic range of a place can be constructed. For example, Evans *et al*. (2010) constructed a strontium isotope distribution 'map' across Britain based essentially on data from modern plant material. Other studies considered the composition of surface and mineral waters (Montgomery *et al*. 2006, Voerkelius *et al*. 2010, Frei and Frei 2011), others performed combinations of modern and archaeological fauna to achieve the same purpose (Frei and Price 2012). There is as yet no ideal way to perform baselines; therefore, it is plausible that a combination of several methods can provide the most accurate range. To produce such baselines is, however, time-consuming and costly, nevertheless they are crucial for providing reliable information on mobility issues. Once local sites and potential target areas are sufficiently characterized with respect to their bioavailable strontium isotope ranges, we might be able to eventually succeed in linking the composition of an archaeological material to its potential area of origin. However, regardless of the amount of already available data on archaeological finds/materials, it is highly recommended to nevertheless characterize the local bioavailable strontium isotopic range to identify if there is any unexpected local variation. Moreover, it should be noted that there are areas that have similar strontium isotope ranges; thus these areas cannot be differentiated by this tracing methodology, and other isotopes could eventually be applied (for example, Pb, lead).

Danish baseline

Denmark has an excellent potential to conduct tracing studies applying the strontium isotope system due to its relative homogeneous geology (Bornholm excluded), as shown in Figure 2, in comparison to the rest of the Scandinavian countries which present a variety of bedrocks of mostly Precambrian era. However, strontium isotopic studies within archaeology in Scandinavia are at an early stage. A few examples are the Neolithic megalithic tombs from southern Sweden (Sjögren *et al*. 2009), Medieval sites in Norway (Åberg *et al*. 1998) and Iceland (Price and Gestsdottir 2006), the Viking Age fortress sites of Trelleborg in Denmark (Price *et al*. 2011) and Sebbersund: an eleventh- to twelfth-century AD Danish churchyard in northern Jutland (Price *et al*. 2012).

Nevertheless, in Denmark, two recent projects have provided a very rich data set of proxy strontium isotope values and a baseline range for future successful tracing investigations. The first study is based on strontium isotope values of surface waters, including a few samples from Bornholm and northern Germany (Frei and Frei 2011). The second is based on strontium isotope analyses of archaeological and modern fauna from Denmark (Bornholm excluded) (Frei and Price 2012). The results of these two studies are quite similar and can be used together to define the Danish local bioavailable strontium isotope range (Figure 3). The overall bioavailable strontium isotope range in Denmark, Bornholm excluded, is ~0.708–0.711. The lowest values come from the Limfjord area near Mors and Fur, most probably due to the geological outcrops of the Paleogene volcanic ash layers (Frei and Frei 2011). The highest values derive from the island of Zealand, which can be interpreted as a result of glaciogenic silica-rich till topsoils. In general, it seems that the lowest $^{87}Sr/^{86}Sr$ ratios are between 0.7078 and 0.7098 which stem primarily from the western part of Denmark, whereas the higher values $^{87}Sr/^{86}Sr$ ratios are between 0.7089 and 0.7108 and stem from the eastern part of Denmark, east of the Lillebælt (Little Belt) (Frei and Price 2012). Thus, the presently available baseline data combined with the rather homogeneous geology of Denmark provide a good point of departure for future strontium isotope tracing studies of various kinds, for example, to evaluate which individuals seem to be non-locals or to investigate the raw material to produce textiles. However, the available data values from Bornholm (Frei and Frei 2011) are insufficient to define the highly complex geology of the island. Therefore, we are presently conducting a detailed investigation based on soil and water samples from the different lithologies present in Bornholm (Frei and Frei, in prep).

Strontium isotopes in archaeological textiles

The phenomenon of travel, transmission, and trade in the field of textiles and fiber material has often been investigated through comparative studies (e.g. Barber 1991).

However, until recently, there were no absolute terms for determining the origin of textile raw material. New developments have aimed at filling this gap by the means of strontium isotope analysis. Thus today, strontium isotope analyses are also applied to other organic materials such as wool, plant fibers from ancient textiles (Figures 4 and 5), and plant fibers from basketry and carbonized seeds. These investigations have been possible due to further development of new geochemical protocols (Benson *et al*. 2006, Frei *et al*. 2009a, Heier *et al*. 2009, Frei 2010, Frei *et al*. 2010).

Denmark possesses a unique collection of well-preserved prehistoric garments from the Bronze and Iron Ages, and thus, this collection constituted the perfect point of departure to address the question of whether

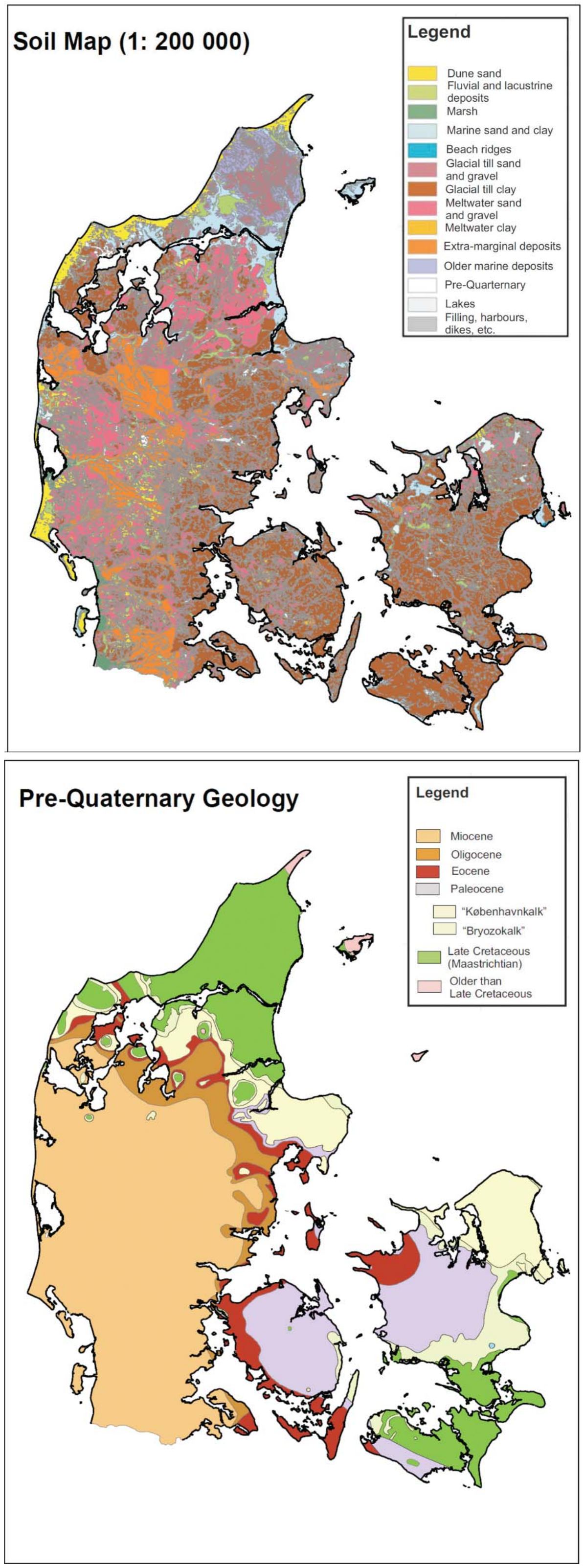

Figure 2. Map (upper) of Denmark delineating the different soil types within a1-meter depth, from GEUS Denmark and the Greenland Geological Survey. The lower map depicts geological pre-quaternary rocks, from GEUS Denmark and the Greenland Geological Survey (GEUS 2004). The sedimentary strata get generally younger from the north-east towards the south-west, because the Danish north-eastern area has experienced an uplift with a consequently higher degree of erosion.

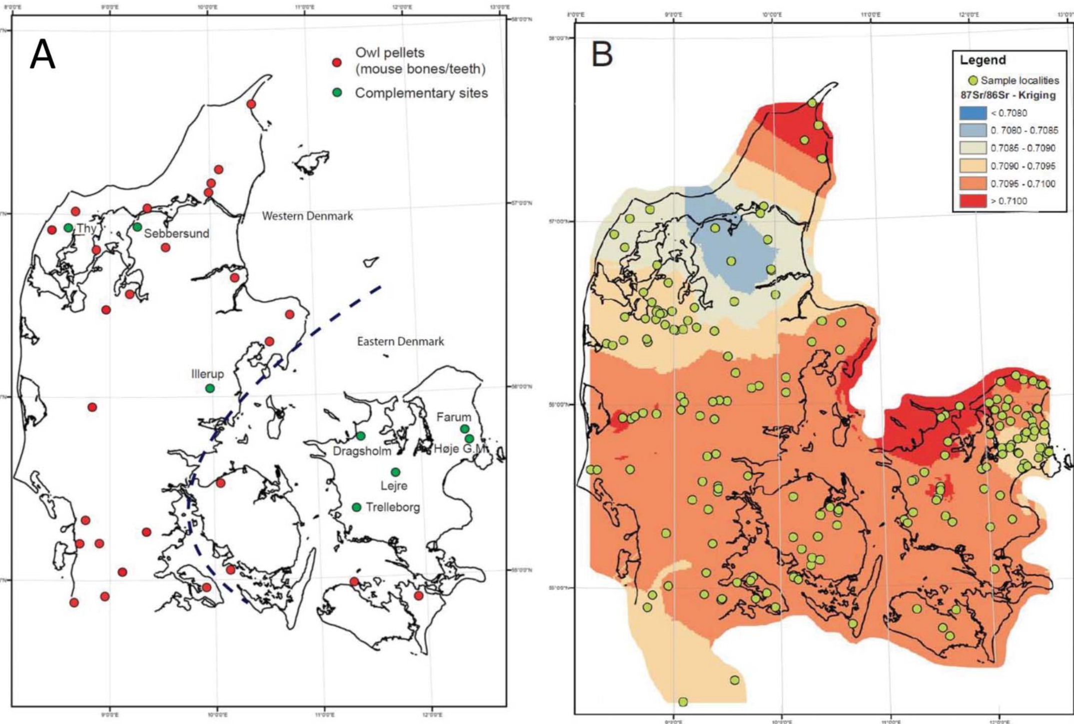

Figure 3. A. Localities where fauna samples were taken for baseline measurements, the stipple lines depict the boundary between eastern and western Denmark, after Frei and Price (2012); B. Depicts the countered distribution of the strontium isotope ratios of surface water samples, after Frei and Frei (2011).

textile raw materials had been traded in Danish prehistory. There are several ways in which archaeological textiles are investigated, for example, by textile tools, fiber counting/recognition techniques, ^{14}C-dating techniques, context analyses, dye analyses (HPLC-analyses), visual quality analyses, weaving techniques, and function analysis (Andersson Strand *et al*. 2010). However, the important research question of the provenance of textile raw material was usually investigated by means of comparative analyses. Thus, the recent developments provide a novel method to acquire information on the provenance of archaeological textile raw material (Frei 2010). In the following section, a brief description of the new methodology for textiles (Frei 2010), as well as a short explanation of the differences between skeletal tissues as compared to that of wool is provided.

Concentrations of strontium in bone tissue are often between ~50 and 1000 ppm (Bentley 2006) whereas in hair ranges between 0.05 and 15 ppm (Frei *et al*. 2009a) (Figure 1). Thus, wool fibers have much lower strontium concentrations than, for example, tooth enamel (Morita *et al*. 1986, Attar *et al*. 1990, Kohn *et al*. 1999, Rosborg *et al*. 2003, Frei 2010). Consequently, the precleaning protocol, the strontium separation (chromatography), and the final analytical measurements (mass spectrometry) are considerably more difficult in wool/hair than in teeth, as they present a greater methodological and analytical challenge than those found in tooth enamel.

Furthermore, the issue of contamination by soil and percolating water is more pertinent when dealing with textiles than with teeth due to the differences in resistance to contamination of these two very different materials (the tooth enamel being the hardest tissue in the body). Moreover, dyestuffs, from , for example, plants, can also be a source of contamination if these dyestuffs have a different origin than that of the textile's raw material. Thus, the main focus of the development of this new methodology has been on decontamination procedures to ensure the recovery of the primary ^{87}Sr/^{86}Sr ratios, which could otherwise be masked by the above-mentioned processes. The methodology is a multistep leaching procedure that includes a 20% cold hydrofluoric acid (HF) wash, a hydrochloric acid (HCl) wash, and an organic dye-removal step consisting of a strong oxidative procedure using ammonium peroxodisulfate ($(NH_4)_2 \cdot S_2O_8$) (Frei *et al*. 2009a, 2010). In between all these steps, several rinses with deionized water (Milli-Rho-Milli-Q; Millipore) should be included. Afterwards, the textile residue is dissolved and ion chromatographic procedures are followed. Finally, the ^{87}Sr/^{86}Sr ratio is measured on a thermal ionization mass spectrometer (TIMS).

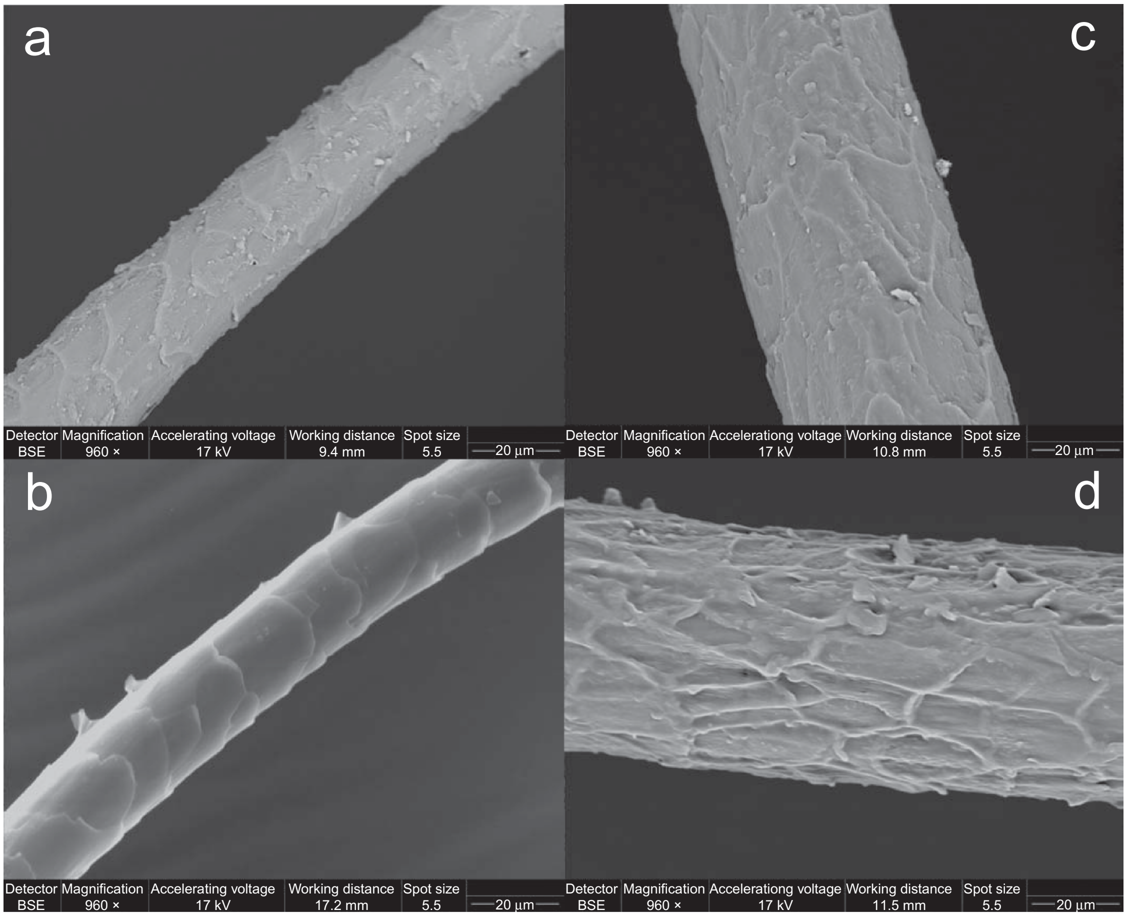

Figure 4. SEM images of individual single wool fibers. The fibers in ‘a’ and ‘c’ were dyed with organic dyestuff (red and blue respectively), the dyed wool fibers indicate that the organic dyestuff coats the hair scales (typical of animal hair) with a thin layer. Small individual particles, identified as mordant salt crystals, occur at the surface. The fibers in ‘b’ and ‘d’ changed to red and blue (respectively) wool fibers after exposure to APDS (ammonium peroxidisulfate)-HF(hydrofluoric acid) treatment. The deterioration is only minor (although stronger in the case of the blue sample ‘d’) as the fiber cuticle remains largely preserved (Frei *et al*. 2010).

In spite of the difficulties that textile raw material presents (Frei 2010), the chemical protocols developed by Frei have shown that it is possible to analyze minute pieces of thread from ancient textiles often in the order of only 10–20 mg per sample equalling a few cm long thread (Frei *et al*. 2009a).

Case studies of Danish archaeological textiles

The development of this new methodology in strontium isotope analysis of archaeological textiles has already provided new crucial information from Southern Scandinavia (Frei 2009, Frei *et al*. 2009b) as well as new insights into the network and trade of textiles during the Danish Bronze and Iron Ages (Bergfjord *et al*. 2012, Frei 2010).

The case studies presented herein were unearthed from peat-bog contexts and all belong to the Danish Iron Age. In general, the Danish textile bog finds have been divided into two main groups by Hald (1950), named after two textile finds: the first is the ‘Huldremose group’ and the second the ‘Corselitze group’. Later, Bender Jørgensen (1986) further developed Hald’s categories into the Huldremose type, the Haraldskær type, the Virring type, and the Spin-pattern type. In this investigation, textiles belonging to the two groups proposed by Hald were chosen.

(1) *Textiles from the Huldremose group*

Some of the first textiles to be investigated by this new methodology were some of the best preserved and complete garments from the Danish pre-Roman Iron Age (500 BC– AD 1). Archaeological textiles have been retrieved twice from the peat-bog site of Huldremose in Northeastern Jutland (Huldremose I and Huldremose II). Typologically, both belonged to Hald’s “Huldremose

Figure 5. Preparing samples at the laboratories of the Danish Center of Isotope Geology (DCIG) at the University of Copenhagen. Photo by Karin M. Frei.

group" of textiles (Hald 1950). This group is defined by textiles usually woven/spun as 2/2 twills and S/S spun and by the twisting of every other warp pair and a low thread count of 7–8 threads per centimeter (Hald 1950).

The textiles from the Huldremose I find were made in connection with the discovery of a peat-bog body of a woman (Figure 6). She was wearing several pieces of clothing: a chequered skirt, a chequered scarf, and two skin capes; today, she is on display at the permanent exhibition of prehistory in the National Museum of Denmark. Wool fibers from the yarn belonging to the chequered scarf were analyzed for strontium isotopes (Figure 7). In addition, three pieces of plant fibers adhering to the bog body were discovered during the process of this study (Figure 8) and analyzed (Frei *et al*. 2009b). The existence of this plant fiber textile was not known prior to this discovery. It is thought that this additional textile could have been a kind of undergarment (Frei 2010, Mannering *et al*. 2011).

Moreover, peat samples still adhering to the bog body were also studied to check for potential contamination. The results of the Huldremose I (Figure 9) demonstrate that the plant textile samples had radiogenic strontium isotope ratios (elevated) which can be expected from, for example,. old geological terrains as those present in Norway and Sweden and hence were nonlocal. On the other hand, the wool yarn from the chequered scarf (C 3474) showed a local strontium isotopic value. Thus, the Huldremose woman was wearing garments made from materials that stemmed from abroad as well as from Denmark (Bornholm excluded). It is therefore possible to assume that she had either herself been travelling outside Denmark and traded or was presented with her plant fiber garment while she was abroad, or she acquired the textile of imported plant fibers material in Denmark itself, either through trade or as a gift.

The Huldremose II find (Figure 10) is a single deposition find, composed of a large single tubular textile. From this textile, a total of 11 samples were analyzed (Frei *et al*. 2009b).

The results from the Huldremose II find (Figure 11) demonstrate that the 11 wool samples taken randomly from all over the garment have a highly interesting pattern. The 11 samples cluster somewhat into three groups. The first six seem to be of local provenance. However, the two other groups have too radiogenic strontium isotopic values to be of Danish provenance, (the island of Bornholm excluded). Hence they are non-local.

However, the Huldremose II textile has previously been considered typologically as being of local provenance as was the type of spinning which is also in itself impressively homogeneous (Irene Skals, National Museum of Denmark, personal communication). Therefore, these facts suggest that the wool was gathered from different places (one within Denmark and probably two from outside Denmark) prior to

Figure 6. Author sampling small peat-bog remains directly from the body of the Huldremose woman bog mummy at the conservation department of the National Museum of Denmark at Brede. Photo by Karin M. Frei.

Figure 7. Wool scarf (sample C 3474) from the Huldremose woman, Huldremose I find; the red circle shows where the sample was taken at the conservation department of the National Museum of Denmark at Brede. Photo by Karin M. Frei.

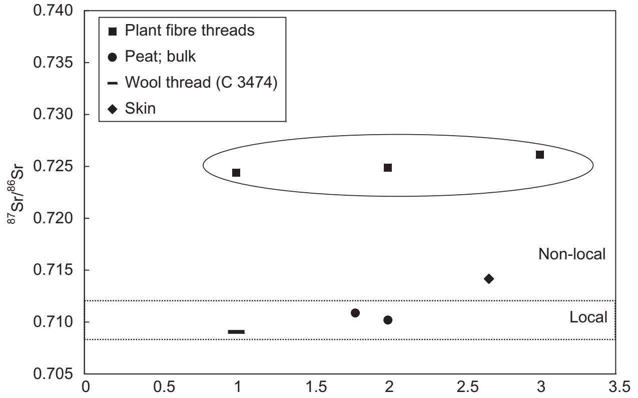

Figure 9. $^{87}Sr/^{86}Sr$ ratios of bulk peat, and residues of skin, plant, and wool fibers from the Huldremose I find. Plant fibers define a group (encircled by an ellipse) that is characterized by non-local $^{87}Sr/^{86}Sr$ ratios. The skin sample also lies in the non-local $^{87}Sr/^{86}Sr$ range. Peat and wool yarn fibers from the scarf of the Huldremose I find (sample C 3474) lie within the range of $^{87}Sr/^{86}Sr$ ratios that are considered local (after Frei *et al*. 2009b).

Figure 8. Magnified picture (binocular microscope) of one of the plant fiber thread samples found still adhering to the body of the Huldremose woman find. Photo by Karin M. Frei.

Figure 10. Sampling the large wool tubular textile find Huldremose II, at the conservation department of the National Museum of Denmark at Brede, Irene Skals and author. Photo by Karin M. Frei.

spinning the yarn, although, the spinning could still have occurred locally. Thus, this new information shows the complexity that can be concealed within one single large garment. Moreover, it sheds new light into the societies of the Danish pre-Roman Iron Age, as the Huldremose II garment was undoubtedly crafted by a highly knowledgeable and skilled person (or people) (Mannering *et al*. 2011) and as very similar wool qualities had been chosen, but from sheep that were very far apart, that is, from raw materials derived from different sources.

(2) *Textiles from the Corselitze group*

Two different textiles belonging to Hald's Corselitze group were analyzed: Corselitze (7325 a, Figure 12) and Haraldskær (3707 C1, Figure 13) (Frei *et al*. 2009a). The Corselitze find is the only one from the Iron Age period, besides the find from Horreby Lyng, which was retrieved outside the Jutland Peninsula. Both textile finds were instead recovered on the island of Falster. The cloth was wrapped around a woman's body fastened by a wool cord and a woven band (Hald 1980). A bronze fibula by the woman's neck dates the find to around AD 300. The results of the sample taken from the Corselitze textile (7325 a) has a strontium isotope ratio that falls within the Danish bio-available strontium isotopic range; thus, this textile most probably was made of wool from sheep

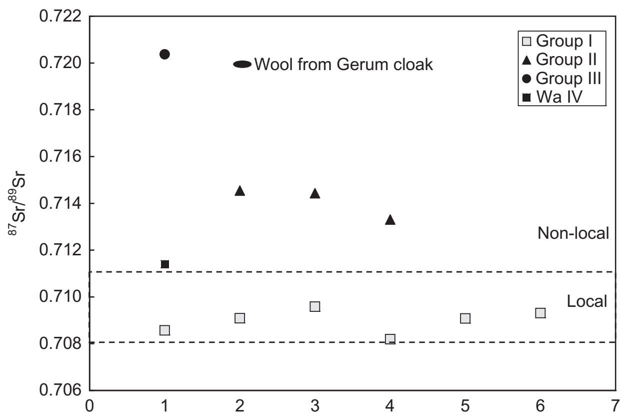

Figure 11. $^{87}Sr/^{86}Sr$ ratios of wool residue samples from a single large archaeological garment (Huldremose II find). Three provenance groups can be discerned: Group I wool threads indicate a local source of strontium. Groups II and III comprise wool threads with elevated $^{87}Sr/^{86}Sr$ ratios implying a non-local provenance. Group III sample shows a $^{87}Sr/^{86}Sr$ ratio similar to wool from a textile find in Sweden (Gerum cloak; (Frei *et al*. 2009a)). Sample Wa IV plots between group I and II.

grazing on Danish soil (Bornholm excluded) (Frei *et al*. 2009a).

The final textile is from a well-known find, the Haraldskær woman who was recovered in a bog near Vejle. Her remains are exhibited at Vejle Cathedral. She was discovered with the remains of three wool textiles, a skin cape and a finely braided wool hairnet. Of the three wool textiles, the one chosen to be analyzed by the strontium isotopic method was the textile with fringed edges and fine stripes in both directions (3707 C1). The results of the strontium isotope analysis on this sample demonstrate that this textile also falls in the Danish bio-available strontium isotope range; thus, this textile most probably was made of wool from sheep grazing on Danish soil (Bornholm excluded) (Frei *et al*. 2009a).

Conclusions

The strontium isotope tracing system has been proved to be an important tool within archaeology in the fields of migration and trade. Yet, very few such investigations have been conducted within Denmark. However, the very recent construction of two different types of baselines (Bornholm excluded) now provides the necessary prerequisite to enable strontium isotopic tracing studies to be conducted in Denmark.

Moreover, recent developments within the field of textile research have made it possible to investigate the provenance of archaeological textiles by following a multi-step pre-cleaning method.

Thus, these two achievements provide new possibilities to investigate human and animal migration and textile trade throughout Danish prehistory by applying the strontium isotopic tracing system. It is, nevertheless, important to mention that all methods have their limitations, and this one is unfortunately no exception. The success of the strontium isotope analyses depends on the archaeological context, the preservation, diagenetic processes (contamination), as well as on how well defined the bio-available strontium isotope range of the retrieval site and the potential place of origin are. However, the numerous worldwide publications prove that there is a great potential in tracing studies applying the strontium isotope system. In Denmark, it is even more pertinent due to the fact that we now have a well-defined baseline

Figure 12. Sampling the wool textile fragment belonging to the Corselitze find (7325 a) at the conservation department of the National Museum of Denmark at Brede, Irene Skals and author. Photo by Karin M. Frei.

Figure 13. Sampling the wool textile fragment belonging to the Haraldskær find (3707 C1) at the conservation department of the National Museum of Denmark at Brede, Irene Skals and author. Photo by Karin M. Frei.

of the bio-available strontium isotopic range, (and soon we will also have one from Bornholm) which will provide researchers the necessary tools to identify potential migrations and trade.

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