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SISÄLLYS – INNEHÅLL – CONTENTS

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Articles	
Radoslaw Grabowski, Bjørnar Olsen, Þóra Pétursdóttir & Christopher Witmore Teillager 6 Sværholt: the Archaeology of a World War II Prisoner of War Camp in Finnmark, Arctic Norway	3
Kristiina Mannermaa, Pirkko Ukkonen & Suvi Viranta Prehistory and Early History of Dogs in Finland	25
Ingela Bergman, Olle Zackrisson & Lars Östlund Travelling in Boreal Forests: Routes of Communication in Pre-industrial Northern Sweden	45
Krista Vajanto & Maarten R. van Bommel Dyed Textiles from Late Iron Age Finland	61
Tytti Juhola, Heli Etu-Sihvola, Tuomas Näreoja & Juha Ruohonen Starch Analysis Reveals Starchy Foods and Food Processing from Finnish Archaeological Artefacts	79
<i>Johanna Enqvist</i> The New Heritage: a Missing Link between Finnish Archaeology and Contemporary Society?	101
Discussion <i>Teemu Mökkönen</i> Archaeological Radiocarbon Dates as a Population Proxy: a Skeptical View	125
Miikka Tallavaara, Petro Pesonen, Markku Oinonen & Heikki Seppä The Mere Possibility of Biases Does Not Invalidate Archaeological Population Proxies – Response to Teemu Mökkönen	135
Håkan Rydving Do We All Have to Be Archaeologists?	141
Notes & News Petr Sorokin & Stanislav Belskiy The Fortress of Kivennapa on the Karelian Isthmus: the Results of an Archaeological Investigation	147
В оок Reviews Peter Holmblad & Mikael Herrgård, Luolamiehistä talonpojiksi. Pohjanmaan muinaisuus sanoin ja kuvin. Reviewed by <i>Jari-Matti Kuusela.</i>	155
Eero Ehanti, Johanna Aartomaa, Irma Lounatvuori & Erik Tirkkonen (eds.), Lost at Sea, Rediscovered. Reviewed by <i>Marcus Hjulhammar.</i>	159
Trond Lødøen & Gro Mandt, Vingen – et naturens kolossalmuseum for helleristninger. Reviewed by <i>Antti Lahelma.</i>	161

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Tytti Juhola, Heli Etu-Sihvola, Tuomas Näreoja & Juha Ruohonen STARCH ANALYSIS REVEALS STARCHY FOODS AND FOOD PROCESSING FROM FINNISH ARCHAEOLOGICAL ARTEFACTS

Abstract

Starch analysis has increasingly been used to study ancient plant cultivation and consumption in many parts of the world, especially in America, Asia and the Near East. In this article we present the first evidence of fossil starch granules found on Finnish archaeological artefacts. Samples taken from three artefacts, a Late Stone Age pottery sherd from the Kiukainen culture, an Iron Age grinding stone from the 7th century AD and an 18th century AD tooth, yielded an abundance of plant starch granules and also possible evidence of food processing. The granules were studied using the latest microscopy techniques and compared against a database constructed of modern starch counterparts in order to identify the origins of the granules. The results indicate that a variety of starchy grain foods were already available by the Late Stone Age. They also suggest that cereal grains and other plant foods had been processed with the Iron Age grinding stone. Starch analysis of the 18th century tooth reveals traces of early potato consumption.

Keywords: starch analysis, cereal, millet, buckwheat family, potato, Finland

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INTRODUCTION

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Human nutrition depends on carbohydrates, which are usually more readily available from plant sources than from animal sources (Englyst & Hudson 2000: 61). Nutritionists divide plant carbohydrates into three categories: sugars, starches and cellulose. Because plant products rich in starch, such as cereals, legumes and root vegetables, are naturally easy to store for long durations, they have become important in most food cultures as staple foods. Starch analysis is especially suitable for detecting these kinds of starch-rich foods from archaeological residues, and in addition many non-cultivars can be studied with this method.

The function, formation, structure, morphology, degradation and gelatinization of starch have been studied extensively since the late 20th century, and the results have been reviewed in detail by many authors (French 1984; Wang et al. 1998; Eliasson 2004; Tester et al. 2004; Copeland et al. 2009). Within their seeds, tubers and roots as well as some fruit and vegetative tissues, plants build up carbohydrate storage units in the form of starch granules, mainly composed of polysaccharides amylopectin and amylose, which construct a complicated semi-crystalline structure with crystalline and amorphous regions within the granule (Bertoft 2004: 57; Jane 2007: 31; Copeland et al. 2009: 1528). Crystalline structures are composed of double helical polysaccharides, either amylopectin or amylose chains, and these are chiral entities that turn polarized light, creating bright quadrants separated by a dark birefringence cross, also called an extinction cross (Tester et al. 2004: 159). Furthermore, these structures



have also been shown to produce specific patterns visible in confocal laser-scanning microscope (CLSM) (van de Velde et al. 2002: 1530–32). Alternating crystalline and amorphous formations are layered on top of each other and around the centre, or the hilum, of the granule and form so-called growth rings (French 1984: 208; Donald 2004: 157–8; Tester et al. 2004: 161; Copeland et al. 2009: 1528).

The structure of starch, its formation process, the size and shape of the granule, the unimodal or bimodal size distribution, the ratio of amylopectin and amylose, the type of crystallinity and other properties such as the tendency to produce single or compound granules, are directed primarily by the botanical origin (Jane 2006: 205; 2007: 31; Tester et al. 2004: 152; Copeland et al. 2009: 1528). In addition, environmental and other factors may affect the features and qualities of starch granules. Different growing environments may affect the granule size and size distribution to some degree, and the water content of the granule may affect the crystallinity and birefringence as well as the visibility of growth rings (Goering et al. 1973: 297; French 1984: 208; Oliveira et al. 1994: 1176; Tester et al. 2004: 161).

The efforts of starch researchers have also benefited archaeologists, as this research has provided tools for working in the reverse direction, investigating the botanical origin of ancient food remains by evaluating the morphology of fossil starch granules. Researchers working with archaeological deposits argue that the family, genus and possibly also species of plants can be identified from fossil starch granules retrieved in good condition (Messner et al. 2008: 112). Taxa have been identified by comparing the morphology and size of fossil specimens to modern starch collections (Loy 1994: 87; Piperno & Holst 1998: 766; Piperno et al. 2000: 896; Messner et al. 2008: 112, 114; Zarrillo et al. 2008: 5007; Duncan et al. 2009: 13205; and many others). Starch granules have been successfully retrieved and analysed from food residues on many types of ancient artefacts such as pottery vessels, vessels carved from gourd or squash shells, grinding stones, stone knives and microliths, as well as from dental calculus on human teeth (Piperno & Holst 1998; Piperno et al. 2000; Perry 2004; Zarrillo & Kooyman 2006; Perry et al. 2007; Messner et al. 2008; Henry & Piperno 2008; Zarrillo et al. 2008; Duncan et al. 2009; Hardy et al. 2009; 2012; Henry et al. 2011; Saul et al. 2012; Li et al. 2013). Starch has also been analysed from soil samples (Loy 1994; Zarrillo et al. 2008; Saul et al. 2012). Researchers also argue that processing activities, such as cooking, grinding and malting, can sometimes be identified by inspecting signs of damage on starch granules (Dronzek et al. 1972; Samuel 1996; Piperno et al. 2004; Zarrillo et al. 2008: 5007; Henry et al. 2009).

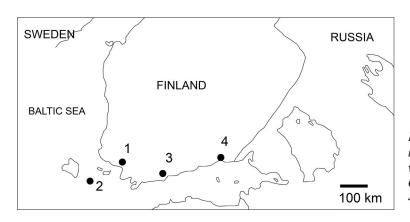
Starch analysis has been successfully employed in America, Asia and the Near East to research plant consumption, domestication and the cultivation history of important crops such as maize, chili peppers, yams, manioc, millet and cereals, as well as to solve the function of ancient tools (Piperno & Holst 1998; Piperno et al. 2000; Perry 2004; Zarrillo & Kooyman 2006; Perry et al. 2007; Henry & Piperno 2008; Messner et al. 2008; Zarrillo et al. 2008; Li et al. 2013). The method has also been suitable for studying the utilization of wild plants in Early Neolithic Germany and in pre-European Canada (Zarrillo & Kooyman 2006; Saul et al. 2012). As starch can in certain environments be extremely age-resistant, starch analysis has been employed for investigating Neanderthal diet in Spain, Belgium and Iraq (Henry et al. 2011; Hardy et al. 2012). To our knowledge, prior to our pilot project, archaeological starch analysis had not been employed in Finland or in any Scandinavian countries. In this article we report the construction of a starch collection extracted from edible northern European plant species to serve as a reference database. We also report on our use of light microscopy to study archaeological starch and present some results from case studies of our starch analysis of certain recently found Finnish artefacts.

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MATERIALS AND METHODS

Archaeological artefacts and site descriptions

In this project three artefacts were chosen for starch analysis for their availability, for being recently excavated from the ground, and for their potential for containing fossilized food remains. The first two artefacts, a sherd from a Late Stone Age Kiukainen culture pottery (TYA 863: 9; radiocarbon dating in process) and an Iron Age grinding stone, a so-called cubic stone (TYA 894:



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Fig. 1. Locations mentioned in the text: 1. Kaarina Ravattula Ristimäki, 2. Kökar Otterböte, 3. Kirkkonummi, 4. Hamina.

10), from the 7th century AD, had recently been excavated at Kaarina Ravattula Ristimäki multiperiodic site in southwestern Finland by project teams from the University of Turku (excavation manager Juha Ruohonen). The inside surface of the large pottery sherd was heavily encrusted with food residue. The grinding stone in turn had been found during an archaeological inventory course, and had not been handled with bare hands or cleaned, but placed in a plastic bag straight from the ground to minimize contamination. The third artefact was a tooth (documentation in process), which had been excavated from an 18th century mass grave in the city of Hamina (Sw. Fredrikshamn) and brought to the University of Turku for analysis by osteoarchaeologist Anne-Mari Liira. The tooth had a massive dental calculus deposit. All of these artefacts were considered promising candidates for starch analysis (Fig. 1).

Sampling

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Many scholars underline the importance of preventing modern starch contamination of samples from hands, surfaces, equipment, air and water during all phases of the process, from handling the artefacts at the excavation site to sampling and preparation at the laboratory (Loy 1994: 96; Piperno & Holst 1998: 768; Messner et al. 2008: 115; Li et al. 2013: 1668). While the excavation phase of these artefacts was not controlled by this pilot project, during all other phases special attention was paid to protecting the samples from contamination. The pottery sherd had originally been washed in order to remove any soil particles. We rinsed the surface of the food crust with sterile water to remove possible contamination before sampling although we were aware that there were microscopic cracks on the food crust which we were not able to clean. Soil particles were not removed from the surface of the cubic stone because we were concerned this would remove any fossil starch as well. The calculus seemed very fragile and was not brushed or cleaned in any way before sample preparation because it received a thorough wash in the decalcifying detergent, as described below.

The samples were prepared at the Department of Geology and Geography laboratory at the University of Turku. Disposable vinyl powder-free gloves, laboratory overcoats and clean equipment were used at all times. Respirator masks were used during the preparation of dental calculus samples. Sterile disposable scalpels and disposable pipettes were used, the dental picks were washed, sterilized by boiling and rinsed with sterile water, the decanters and other equipment were washed and rinsed with sterile water. The samples on microscope slides were dried in petri dishes with closed lids to avoid contamination from the air. Based on the experiences during this project, better procedures are being developed and in future projects even more emphasis will be placed on contamination prevention.

It is also important to take soil samples at archaeological excavations from the same layers as the artefact (Loy 1994: 96; Zarrillo et al. 2008: 5007; Saul et al. 2012: 3485). In this way possible contamination from the soil to the artefact can be controlled. Unfortunately, there were no soil samples available in this pilot study, but this will be addressed in future projects.

Scientific name	English name	Sample	Source
Acer platanoides	Norway maple	Seed	Collected
Avena sativa	Oat	Seed	Commercial
Chenopodium album	Common lamb's-quarters	Seed	Collected
Elymus caninus	Bearded couch-grass	Seed	Vendor
Fagopyrum esculentum	Common buckwheat	Seed	Commercial
Fallopia convolvulus	Black bindweed	Seed	Collected
Glyceria fluitans	Floating sweet-grass	Seed	Vendor
Hordeum vulgare var. nudum	Barley (naked)	Seed	Commercial
Hordeum vulgare var. vulgare	Barley (hulled)	Seed	Commercial
Lens culinaris	Lentil	Seed	Commercial
Leymus arenarius	Blue lyme grass	Seed	Vendor
Nuphar lutea	Yellow pond lily	Seed	Collected
Panicum miliaceum	Broomcorn millet	Seed	Commercial
Pisum sativum var. sativum	Garden pea	Seed	Commercial
Polygonum aviculare	Common knotgrass	Seed	Collected
Quercus ssp.	Oak	Seed	Collected
Rumex longifolius	Northern dock	Seed	Collected
Sagittaria sagittifolia	Arrowhead	Root	Collected
Secale cerealia	Rye	Seed	Commercial
Setaria italica	Foxtail millet	Seed	LUOMUS
Setaria viridis	Green foxtail	Seed	LUOMUS
Solanum tuberosum	Potato	Tuber	Commercial
Triticum aestivum	Common wheat	Seed	Commercial
Triticum dicoccon	Emmer wheat	Seed	Commercial
Triticum monococcum	Einkorn wheat	Seed	Commercial
Triticum spelta	Spelt wheat	Seed	Commercial
Vicia faba	Broad bean	Seed	Commercial
Zea mays	Maize	Seed	Commercial

Table 1. Modern reference samples used in this study.

Samples from the pottery sherd were extracted following the guidelines set by Messner et al. (2008: 115). In laboratory conditions, several samples from the food crust on the sherd were scraped into test tubes, using new disposable scalpels and clean test tubes for each sample. The weight of the crust samples was small, approximately 20–50 mg.

A method for sampling stone tools, described as 'spot sampling', is to rinse the surface using a pipette and sterile water and to gather the water for analysis into a test tube (Perry 2004: 1074; Messner et al. 2008: 115; Li et al. 2013: 1668; see also Loy 1994: 98). A second method is to carefully pick out residues from cracks and indentations on the tool surface (Piperno & Holst 1998: 768). A third method is to collect the residue from the artefact by submitting it into an ultrasonic bath (Perry 2004: 1075; Messner et al. 2008: 115; Zarrillo et al. 2008: 5010). The first two methods were used experimentally on the grinding tool resulting in two separate samples. The ultrasonic method was not used because we treated only half of the grinding surface of the stone, leaving the other half untouched. We also suspected that ultrasonic treatment would separate possible starch clusters in the samples. ۲

The samples extracted from the pottery sherd and the grinding stone were submitted to heavy liquid separation, as instructed by Perry (2004: 1075) and Zarrillo et al. (2008: 5010), with some modifications. The starch granules with specific gravity close to 1.5 were separated from other organic and inorganic material in the following

way: Solutions with a specific gravity of 1.3 and 2.0 were prepared using LST Fastfloat and sterile water. First 5 ml of the 1.3 solution was added to a sample test tube (size 15 ml), agitated and centrifuged at 2500 rpm for 10 minutes. The supernatant contained the material under 1.3 specific gravity, and it was removed to a second test tube. Next, 5 ml of the 2.0 solution was added to the first test tube, agitated and centrifuged, and the supernatant, which was of 1.3-2.0 specific gravity, was poured into a third test tube. This step was repeated. All three tubes were filled with sterile water and left to set overnight. Next day the samples were washed four times by removing 75% of the liquid for recycling, filling the tubes with sterile water, agitating and centrifuging. The supernatant was removed and the small amount of residue left on the bottom of the tubes was pipetted onto a series of microscope slides placed in petri dishes. The lids were closed on the petri dishes and the slides were left to dry at room temperature, then mounted with a 1:1 mixture of glycerol and sterile water, covered with coverslip and sealed with nail polish using disposable brushes.

The dental calculus was sampled in the following way, as instructed by Henry & Piperno (2008: 1943). Some calculus was scraped off from the tooth using a sterilized dental curette. The calculus was collected onto a piece of aluminum foil, from which it was poured into a glass test tube. Because the decalcifying liquid Calgon, recommended by Henry & Piperno (2008), was not available, a decalcifying liquid containing citric acid was added to the test tube and left to rest for 24 hours, after which the mixture was subjected to an ultrasonic bath for 5 minutes and centrifuged for 10 min at 2500 rpm. The supernatant was removed and the sample was rinsed eight times with sterile water and centrifuging, until there were no more bubbles from the decalcifying liquid. Then 10% HCl was added to the tube and left to break down the calculus for 12 hours. The sample was rinsed twice as explained above removing the supernatant and the remaining residue was pipetted onto microscope slides. The slides were dried in the same way as the previous samples, but this time they were mounted in immunofluorescence mounting medium (Mowiol 4-88) instead of glycerol. This method of sample preparation improved CLSM scanning quality, as it decreased interference with

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the starch reflection and diffraction patterns, and also enhanced preservation of the samples.

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Modern plants were collected to form a reference starch collection for the use of this project. The reference collection consisted of many northern wild species which may have been used as food in prehistoric times according to ethnological observations and recent archaeobotanical studies, as well as several cultivated crop species. Potato was included as a potential 18th century crop, and also maize was included, as it can be present as a modern contaminant. Some of the samples were acquired from wild plant seed vendors, some were collected by the authors from forests and meadows, and some from commercial cereal, fruit and vegetable packages. As these sources may present some problems regarding the exact authenticity of the species, plans have been made to build a large verified collection in co-operation with experts at the Finnish Museum of Natural History (LUOMUS).

The reference species were sampled in the following way: Seeds, roots and fruit of over 50 different species were ground in mortars into very fine flour, which was then mounted on microscope slides using either the glycerol and water mixture or the immunofluorescence mounting medium. To improve visibility, some of the key samples were also sonicated in sterile water before being mounted on microscope slides. Some of the samples, such as linseeds and wild berries, proved not to contain visible starch granules at all and only 28 of the samples had large enough starch granules to be studied within the resources and schedule of this project. These species are listed in Table 1 (scientific naming based on Hämet-Ahti et al. 1998).

Analysis and identification

The prepared microscope slides were analysed under a light microscope Olympus BX50 equipped with a cross polarized light filter and imaged using an Olympus DP10 microscope camera at the Department of Geology and Geography laboratory. Some key samples were also documented using scanning electron microscopy (SEM) at the Åbo Akademi Process Chemistry Centre and a confocal laser-scanning microscope (CLSM) Leica SP5 STED/MP (Leica Microsystems GmbH, Mannheim, Germany and MaiTai HP -laser, Spectra-Physics, US) at the Turku Bioimaging Centre.

The CLSM, a point scanning light microscope, acquires optical sections with 2 µm thickness through the starch grain by recording transmitted, scattered and diffracted laser light with a combination of photomultiplier tubes and an objective with very high magnification. This allowed us to reliably detect low-intensity scattered light from the internal and surface structures of starch granules. An oil immersion objective lens (N.A.1.4 100x Oil, Leica) was used in the collection of high-magnification data. The polarization filter at 45° was applied when wide field data was collected. The confocal reflection was excited at 488 nm and detected in 480-560 nm by GaAs-hybrid photomultiplier tubes. The transmission light was collected by a photomultiplier tube set to detect over the whole visible spectrum.

All of the fossil starch finds were imaged and measured, and particularly interesting finds were also scanned using CLSM. The fossil starch finds could not be scanned using SEM because of the method of preparation. Although our experiments with CLSM were very preliminary, they suggested that species-specific differences in internal morphology of starch granules may be documented using CLSM, as already demonstrated by nutritional and industrial food research (Dürrenberger et al. 2001; van de Velde et al. 2002; Jane 2006; 2007). Some examples of CLSM scans of wheat, barley and millet starch granules as well as fossil granules are presented in Figs. 11–13.

Identification of plant species was experimented by comparing the morphology of fossil starch granules with examples from our modern starch collection (Table 1) as well as with images and observations by other researchers (Perry 2004: 1075-6; Messner et al. 2008: 112, 114; Zarrillo et al. 2008: 5007). The identification was based on the following distinct features pointed out by scholars working with archaeological residues: the size (Table 2) and general shape of the starch granule, the surface features, the shape and location of the hilum, the shape and location of internal fissures, visible growth rings and the shape of the birefringence cross (Loy 1994: 87-8; Piperno & Holst 1998: 766, 768; Piperno et al. 2000: 896-7; Perry 2004: 1075-6; Messner et al. 2008: 112).

RESULTS AND DISCUSSION

Reference samples

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The key reference samples for this case study consisted mainly of species belonging either to the cereal and wild grass group, the millet species group, including wild millet, or the buckwheat family group. Fifty to one hundred starch granules from each key sample were imaged and measured, and the maximum measurements were compared with the literature (Table 2). According to this research, in all of these three groups the sizes of the cultivated species were significantly larger than the wild species. Out of the wild grasses in the cereal and wild grass group, the blue lyme grass (Leymus arenarius), bearded couch-grass (Elymus caninus) and floating sweetgrass (Glyceria fluitans), only blue lyme grass presented rather large, roundish granules which could easily be confused with cereals.

However, there are differences in morphology and in the visibility of growth rings within this group (Figs. 5&6). According to the literature, crater-like depressions on the surface are more common and larger with wheat starch than with barley starch (Henry et al. 2011: 487; Li et al. 2013: 1669). Barley starch often has clearly visible growth rings unlike any wheat starch (see also Piperno et al. 2004: 671). On the other hand, we observed no craters on the blue lyme grass and bearded couch-grass reference samples, and visible growth rings were extremely rare in these. Cereals, wheat, barley and rye, can also be identified by their bimodal size distribution, in which two size groups of starch granules are observed, large and small (Copeland et al. 2009: 1529). We also observed possible bimodal populations in the blue lyme grass and bearded couch-grass reference samples.

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The reference samples of broomcorn and foxtail millet produced roundish starch granules with rather smooth outlines, central hilums, and distinctive round central depressions, and it appears that only the size difference sets these species apart (Fig. 8). The green foxtail also differs in shape and surface structure to the cultivated millet species. The reference samples of the buckwheat family (Polygonaceae), common buckwheat (*Fagopyrum esculentum*), common knotgrass (*Polygonum aviculare*), black bindweed (*Fallopia convolvulus*) and northern dock (*Rumex longifolius*)

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Scientific name	English name	Max. L µm (a)	Max. W µm (a)	Range µm (d)	L Range µm (b)	W Range µm (b)
Elymus caninus	Bearded couch-grass	17	16	-	-	_
Fagopyrum esculentum	Common buckwheat	14	14	-	-	-
Fallopia convolvulus	Black bindweed	11	10	-	-	-
Glyceria fluitans	Floating sweet-grass	6	6	-	-	-
Hordeum vulgare	Barley	33	27	10.1-22.4	6.9-26.8	6.2-23.0
H. vulgare var. nudum	Barley (naked)	31	31	-	-	-
Leymus arenarius	Blue lyme grass	23	20	-	-	-
Panicum miliaceum	Broomcorn millet	14	11	5.0-11.9	3.3-11.1	3.0-10.8
Polygonum aviculare	Common knotgrass	10	8	-	-	-
Rumex longifolius	Northern dock	9	9	-	-	-
Secale cereale	Rye	44	44	12.4-45.2	-	-
Setaria italica	Foxtail millet	17	16	5.1-19.3	4.8-20.3	3.7-17.2
Setaria viridis	Green foxtail	11	11	5.0-14.0	-	-
Triticum aestivum	Common wheat	28	28	10.0-39.4	5.7-39.4	5.1-35.0
Triticum dicoccon	Emmer wheat	34	31	8.0-30.0 (c)	-	-
Triticum monococcum	Einkorn wheat	36	34	-	-	-

(a) Reference collection, measured by Juhola

(b) Li et al. 2013: 167-71

(c) Piperno et al. 2004:672

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(d) Yang & Perry 2013: 3171

Table 2. Starch granule size variation. Preliminary statistics on the maximum length and width of starch granules in some key samples from the reference collection, compared with the literature.

presented rather small starch granules with uneven surfaces, the common buckwheat having the largest granules.

The CLSM scans of the reference starch granules demonstrated patterns, which are likely to reflect the internal crystal and lamellar structures of the granules. Figs. 11–13 show images processed by using transmission light, polarized light and reflected light. The diffraction signal produced a more robust ring-like pattern with the reference sample of barley than with emmer wheat, and the blue lyme grass did not present any ring-like diffraction at all. Scanning of millet produced a distinctive spot of reflected light.

As discussed above, it is important to note that the size and other features of starch granules may vary within species for genetic and environmental reasons, which leads to the fact that the reference collection should be expanded to include as many samples as possible from each species from different locations and environments, in order to acquire a better understanding on the possible variations. For this reason, the results in this article are only preliminary, and more research is necessary in order to build reliable parameters and statistics for species identification.

Food residues on Stone Age pottery sherd

The Kiukainen culture sherd yielded an abundance of starch granules. The sample reported here consisted of 41 starch granules out of which 12 could be given preliminary identifications (Fig. 2). Compared with descriptions and images in the literature as well as with our modern starch collection (see examples in Figs. 5–8, 10), the size and morphology of the fossil starch granules resemble most closely those of the species listed in Table 3. The discovery of various types of starch may indicate a diet combining gathered starch sources with cultivated crops or the use of non-monoculture crops.

Cereal starch

The large, almost identical starch granules in Figs. 2A and 2B were identified as belonging to the grass family by their smooth roundish or

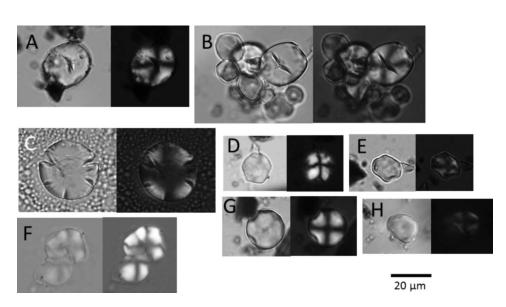


Fig. 2. Some starch granules on the Kiukainen culture pottery sherd and their possible identifications. Light microscope images on the left and polarized on the right. A: Cereal starch, possible wheat (Triticum sp.). B: The largest granule, cereal starch, possibly wheat (Triticum sp.) with potential bimodal distribution of starch grains characteristic for cereals. C: Large starch granule from cereal, which shows signs of degradation or processing. D and E: Buckwheat family (Polygonaceae). F: Unidentified. G and H: Foxtail millet Setaria italica.

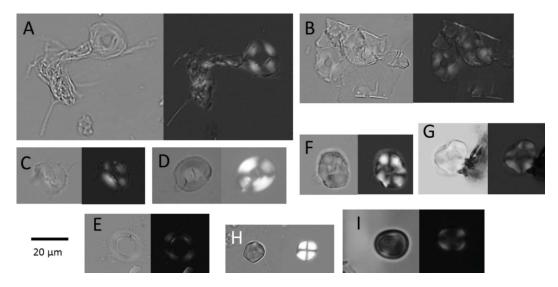


Fig. 3. Starch granules with signs of degradation or processing (possibly malting) on the Iron Age cubic stone. The granules are uniform in size and correlate both in size as well as in form to cereal malt, although wild grasses cannot be ruled out. The damage inflicted on the granules may indicate the process of malting: germination and heating. F and G: Possible pseudo-compound starch granules resembling pea (Pisum sativum) and other legumes. H: Possible millet starch. I: Degraded starch.

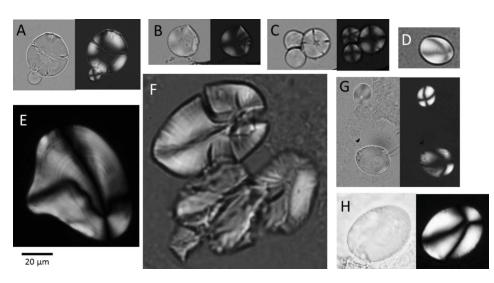


Fig. 4. Starch granules on the 18th century dental calculus. A: Cereal starch showing typical bimodal distribution of granules and signs of processing or digestion. B and C: Starch granules from millet. The size and morphology correlates with foxtail millet (Setaria italica) and the central depression is visible in many of the granules. D–H: Starch granules which show characteristics typical of potato (Solanum tuberosum). The granules are extremely large, the hilum is off-centred and very distinct lamellae are visible.

oval shape, central hilum, central fissure and size (Messner et al. 2008: 119-20; see also Table 2). The approximate size of the granule in 2A was 25 x 20 µm, which falls nicely into the category of cultivated cereals, being larger than any of the wild grasses in our reference collection (Table 2). The size of the large granule in 2B was 23 x 20 µm, which is just on the upper limit of blue lyme grass, but clearly larger than the other two wild grasses. Starch in Fig. 2B has the typical bimodal size distribution of cereals. The fossil starch granules in question were dotted with many large crater-like surface depressions which are common in wheat species, and especially common in emmer wheat (Triticum dicoccon) (Piperno et al. 2004: 671). Interestingly, the CLSM scan from the granule in Fig. 2A showed thin ring-like diffraction and a surface reflection that more closely resembled our experimental scans from emmer wheat than those from other cereals.

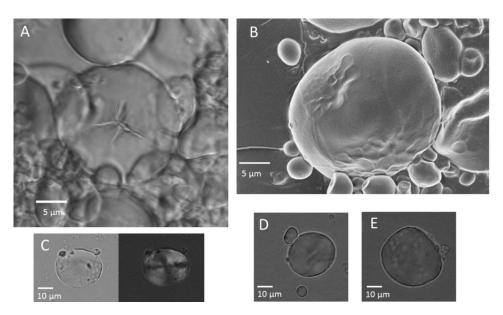
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According to previous research, emmer wheat, einkorn wheat and barley were among the first domesticated crops in Europe, and emmer wheat was the most important crop for the Linear Pottery culture (LBK) (in Poland and Germany, the 5th millennium BC) and also continued to be the most common wheat in northern Europe through the Neolithic and Bronze Ages (Zohary & Hopf 2000: 34–5, 42–8, 59, 230–1, 234, 236–7). In Sweden, emmer wheat was cultivated from the beginning of the Early Neolithic (3900–3500 BC), together with common wheat, club wheat, einkorn wheat, naked barley and hulled barley (Welinder et al. 1998: 50, 71–5).

The oldest macrofossil finds of emmer wheat in Finland date back to the early Iron Age (Rousi 1997: 67). The Kiukainen culture population was familiar with cereal farming, as is evident from the naked barley (*Hordeum vulgare* var. *nudum*) finds at the Turku Niuskala site (Rousi 1997: 61; Zohary & Hopf 2000: 237), located less than 2 km from the Ristimäki site. Emmer wheat as well as other wheat species had been cultivated for thousands of years in neighboring countries, so it is very likely that different wheat species were known in Finland as well, through either cultivation or trade.

Millet

The food crust on the Kiukainen culture sherd also consisted of several starch granules with the typical features of millet starch (Fig. 2G&2H). Comparing the size of the fossil granules to Table



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Fig. 5. Starch granules from modern emmer wheat (Triticum dicoccon) reference samples. A: The CLSM scan shows central crevices on some of the granules. B: The SEM picture highlights the typical multiple depressions on the surface. C–E: Emmer wheat morphology.

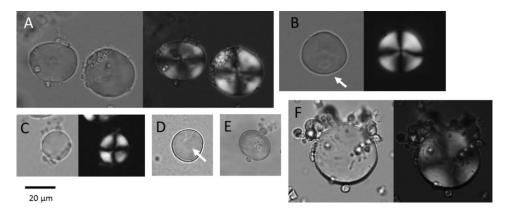
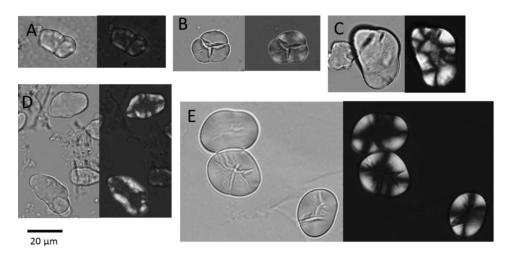


Fig. 6. Starch granules from modern reference samples. A and B: Common wheat (Triticum aestivum). The granules are roundish in shape and the hilum is centric. Some surface depressions are visible (see arrow). C–E: Barley (Hordeum vulgare). The arrow points to visible growth rings (D is variety nudum). Surface depressions are often grouped. F: Rye (Secale cereale) may have very large granules.

2, the only possible millet species would be foxtail millet (Setaria italica), as the granules were very large, 19 x 19 μ m and 17 x 13 μ m respectively. Broomcorn millet (*Panicum miliaceum*) as well as the wild millet, green foxtail (*Setaria viridis*), must be excluded because they have smaller granules. The fossil starch in Fig. 2G was also scanned using CLSM, and a large spot of reflected light was observed in the centre of the granule, correlating with the reference samples of the foxtail and broomcorn millet species (Fig. 11). ۲

According to researchers both foxtail and broomcorn millet were domesticated in China, broomcorn millet by the 9th millennium BC and foxtail millet by the 7th millennium BC (Lu et al. 2009: 7367). Some researchers suggest that broomcorn



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Fig. 7. Pseudo-compound starch granules from modern legumes. A–C Garden pea (Pisum sativum var. sativum). D: Broad bean (Vicia faba). E: Lentil (Lens culinaris).

Identification	Number of starch granules
Cereal, possibly wheat (Triticum sp.)	2
Degraded or processed starch, probably cereal	1
Polygonaceae family, possibly buckwheat (Fagopyrum)	2
Millet, probably foxtail millet (Setaria italica)	3
Grass family (Poaceae), species not identified	3
Possible pseudo-compound granule, unidentified	1
Unidentified	29

Table 3. Starch granules on the Stone Age pottery sample.

millet was domesticated separately in Europe, as it appeared in Europe in the 7th millennium BC (Crawford 2009: 7271), or even that these two millet species were domesticated in eastern Europe alone, from where the cultivation then spread into Asia (Hunt et al. 2008: 55).

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Broomcorn millet cultivation was adapted by the LBK culture and it continued in many parts of Europe through the Late Neolithic and Bronze Age, but became rarer in later ages (Zohary & Hopf 2000: 83). In the Scandinavian Bronze Age (1800–500 BC), millet became an important crop, because of its tolerance to drought during the warmer climate phase (Welinder et al. 1998: 50, 71, 74). Millet can be cultivated in poor quality soils without fertilizers and irrigation, and it yields crop in less than three months, which makes it suitable for the short growth period.

According to researchers millet cultivation together with horses, sheep, carts, looms, bronze equipment, and farming tools, belonged to a completely new Bronze Age farming lifestyle in Sweden (Welinder 2011: 43). In addition, foxtail millet (Setaria italica), appeared in central Europe in the 2nd millennium BC (Zohary & Hopf 2000: 86). Millet imprints on pottery from the 2nd millennium BC have been found at a seal hunting camp site at Kökar, in the Finnish archipelago, less than 100 km from the Ristimäki site – the pottery bore a strong resemblance to Lausitz culture pottery from Poland (Gustavsson 1997: 95, 100, 108-10). Researcher Lars-König Königsson has suggested that the pollen records of barley from Novgorod (Neolithic Stone Age, since the 4th millennium BC) may also include broomcorn or foxtail millet, because it was impossible to distinguish between millet and barley pollen (Königsson et al. 1997: 366). The distance from Novgorod to the Ristimäki site is only 550 km. Today, broomcorn, foxtail and green foxtail millet species can occasionally

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be found growing wild in southern and central Finland (Hämet-Ahti et al. 1998: 617–8).

Populations cultivating millet existed in neighboring areas at the time of the Kiukainen culture, and millet could have been cultivated in Finland as well. Although broomcorn millet would have been a more feasible candidate than foxtail millet for the fossil starch on the Kiukainen sherd, it would have yielded somewhat smaller starch granules. Further research will be necessary to investigate the extent of millet cultivation and choice of millet species in Finnish prehistory.

Buckwheat family

The starch granules in Figs. 2D (14 x 13 µm) and 2E (14 x 11 µm) were possibly from the buckwheat family (Polygonaceae), which consists of several edible wild plants, such as common knotgrass (Polygonum aviculare) and curly dock (Rumex crispus) (Aaltonen & Corander 1997: 44-8), as well as cultivated common buckwheat (Fagopyrum esculentum). Compared to the reference collection samples of common knotgrass, northern dock (Rumex longifolius), black bindweed (Fallopia convolvulus) and common buckwheat, as well as to other reference samples, the fossil starch showed a close resemblance to common buckwheat starch in size and form. The starch granules in the wild plants were smaller in size as well as different in shape compared to the starch in common buckwheat (Table 2). Researcher Liliana Janik also lists other species of cultivated buckwheat, such as tartary buckwheat F. tataricum, Kashmir buckwheat F. kashmirianum and F. cymosum, which should be added to the reference collection and compared with this possible buckwheat find (Janik 2002: 301).

Although these results are preliminary and more research into wild plant starch morphology is necessary in order to produce reliable conclusions, it is very possible that buckwheat will be found at Kiukainen culture sites. Buckwheat pollen has been found in central Finland from 5260 calBC (Alenius et al. 2013: 12) and lately also in Kirkkonummi, southern Finland, from sediment (as yet undated) that could be connected to the Kiukainen culture (pers. comm. Teija Alenius). The distance between Kirkkonummi and the Ristimäki site is less than 140 km. Buckwheat lipids have also been found on pottery at the Kökar site (the 2nd millennium BC) (Gustavsson 1997: 95). Buckwheat tolerates poor soil and for that reason was very suitable for the end phase of crop rotation in the swidden agriculture of eastern Finland, where buckwheat was cultivated extensively in the 18th and 19th centuries AD (Rousi 1997: 112). In addition, Stone Age fields must have had very poor soil, as scholars have suggested that fertilizers were not used before the 1st millennium BC or Iron Age (Pedersen & Widgren 2011: 52; Welinder 2011: 19).

At the time of the Kiukainen culture, buckwheat was cultivated on the coasts of the Baltic Sea. Janik has presented several buckwheat pollen finds from Poland, Latvia and Denmark, dated from the 4th to the 2nd millennium BC (Janik 2002: 300–1). The Danish sample was of the tartary buckwheat species, discovered inside a vessel. Janik reports that buckwheat disappeared from pollen records during the Bronze Age and reappeared later in the Middle Ages (Janik 2002: 299, 302). Today, common buckwheat and tartary buckwheat grow occasionally as wild weeds in southern and central Finland, and common buckwheat is also found in Enontekiö and its vicinity, northwestern Finland (Hämet-Ahti et al. 1998: 146).

Degraded or processed starch

Researchers have documented changes in starch structure that may indicate food processing, such as cooking, parching, baking, grinding or malting (Samuel 1996; Piperno et al. 2004; Zarrillo et al. 2008: 5007; Henry et al. 2009). When starch is cooked in hot water the granules begin to gelatinize, swell and distort, lose their birefringence crosses and may fuse into each other until they are unrecognizable (Copeland et al. 2009: 1529–30; Henry et al. 2009: 916–7).

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The fossil granule in Fig. 2C was either a very damaged granule or one showing evidence of advanced food processing, such as dehusking grain by heating with hot stones and pounding the grain to remove the outer coating. The growth rings had become visible in the centre, which could result from intense heating, and there was cracking on the outer edges of the granule (Hardy et al. 2012: 623). Such treatment together with pounding the grain with stones would have been necessary to remove the inedible husks from emmer wheat and einkorn wheat (Rousi 1997: 67), as well as from hulled barley. In addition, there was also a slight hint of gelatinizing on the top edge of the granule which may indicate cooking in water, as in food preparation.

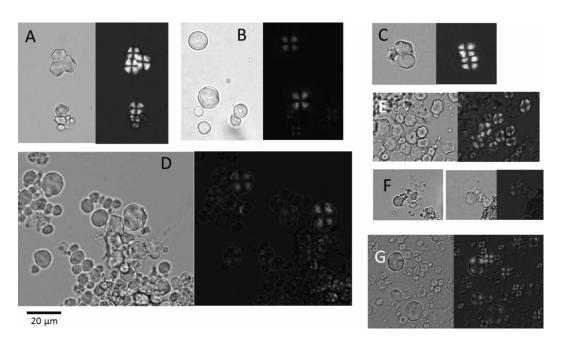


Fig. 8. Starch granules from modern reference samples. A: Broomcorn millet (Panicum miliaceum). B: Foxtail millet (Setaria italica). C: Green foxtail millet (Setaria viridis). D: Common buckwheat (Fagopyrum esculentum). E: Common knotgrass (Polygonum aviculare). F: Northern dock (Rumex longifolius). G: Black bindweed (Fallopia convolvulus).

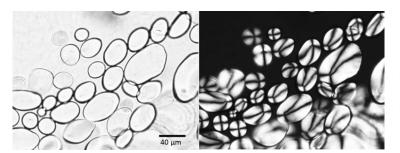


Fig. 9. Potato (Solanum tuberosum) starch granules from a modern reference sample. The granules are very large and vary in form, the hilum is acentric, and the lamellae are very distinct. ۲

According to the morphology and very large size $(30 \times 28 \ \mu\text{m})$ of the granule in Fig. 2C, it was probably a cereal grain, but because of the damage inflicted to the starch granule it was difficult to examine. The size may also be enlargened by processing.

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Residues on Iron Age grinding stone

Cubic stones, small, cube-shaped or roundish hand-fitting stones with worn facets, are a common artefact type from the Bronze Age to the Viking Age, and scholars have suggested that they were used for grinding grain or for dehusking hulled grain by pounding, or even for grinding cremated human bones (Huurre 2003: 62; Kaliff 1997). Macrofossils of hulled barley have been found at many sites in Finland dating from the 6th century BC onwards, and both emmer wheat and hulled barley have been found in Laitila from a Merovingian Period cairn (AD 575–800) (Rousi 1997: 61; Huurre 2003: 48–9). This starch analysis of an Iron Age cubic stone found in a 7th century cairn was an interesting opportunity to study the function of the stone artefact. The fact that the tool was found at a cremation burial site could imply that it was connected to the deceased or to the functal practices. It is our understanding that there was no evidence that the stone had suffered heat damage from cremation fires.

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The hypotheses about bone crushing could not be solved with this analysis but there were several small pieces of cremated and crushed human bones in the same test pit with the cubic stone.

The sample extracted from the stone yielded 19 starch granules, of which six were probably processed cereal starch, four were starch from the grass family, two were pseudo-compound granules typical to legumes, one was a probable millet starch granule and six were degraded starch granules (Table 4, Figs. 3, 7, 10).

Processed cereal and eroded starch

Six starch granules (Fig. 3) from the cubic stone sample were identified as degraded or processed starch, probably cereal. The round granules with centric hilums were uniform in size (largest approximately 23 x 19 µm) which correlates with cereals and blue lyme grass in our reference collection. The granules had very large holes in their centres which was possible evidence that the grain had germinated (Dronzek et al. 1972: 237; Samuel 1996: 488-9). The doughnut-like swollen and slightly distorted granules conformed with the results of our malting experiments, in which moist germinated grain was subjected to mild heat (Fig. 10B). Figs. 10A-D present starch from correctly germinated malt, in which the sprout is only a few millimetres long, and Fig. 10E displays more substantial deformation from overly germinated malt. Malt is germinated grain, which has been dried in the sun or parched with heat, and then pounded into coarse flour. Numerous macrofossils of hulled barley have been found in Finland starting from the 6th century BC (Rousi 1997: 61) and malt can also be processed from other grains, such as wheat and rye.

Another six degraded starch granules were extracted from the cubic stone, possibly deriving from cereal grain because of their form and size (largest diameter approximately $31 \mu m$). These granules were very eroded with pronounced

growth rings and were difficult to study because of their deteriorated condition and their position on the microscope slide.

A pounding tool such as a cubic stone could have been used for different kinds of food processing activities such as grinding malt or dehusking grain. It is important to point out that this starch could also derive from grain which was germinated spontaneously or corroded in other ways. As we were not able to take any control soil samples it was not possible to verify that the starch on the stone tool had not originated from the soil.

Pseudo-compound granules

Two possible so-called pseudo-compound starch granules were found on the cubic stone sample. Pseudo-compound granules have the appearance of separate granules fused together, and this structure is typical of some starches, for instance the pea (French 1984: 184, 186) (see also Fig. 3). Compared to legume reference samples and examples in literature they resembled pea (Pisum sativum) starch, as described by Thomas C. Hart (2011: 3250). The pea sample in our reference collection was not adequate, as our sample was from modern P. sativum var. sativum and not from P. sativum var. arvense, which was the ancient pea variety (Rousi 1997: 122). The centres of the fossil starch granules seemed to be damaged, and it is possible that such damage was caused by milling, as is in the case of maize starch experiments reported by Zarrillo et al. (2008: 5007-8).

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It is claimed that legumes such as peas and lentils (*Lens culinaris*) were domesticated and cultivated together with grains in almost every agricultural community because of their high protein content as well as their ability to return nitrogen to the soil (Rousi 1997: 116–7; Zohary & Hopf 2000: 92, 98). There have been many finds of Neolithic lentils and peas in Europe, for instance peas in Rhine Valley LBK sites (4400–

Identification	Number of starch granules
Cereal starch granules, possibly processed	6
Degraded starch granules, possibly cereal	6
Grass family (Poaceae), species not identified	4
Pseudo-compound granules, possibly from legume	2
Probable millet (for instance Panicum / Setaria), species not identified	1

Table 4. Starch granules found on the Iron Age cubic stone sample.

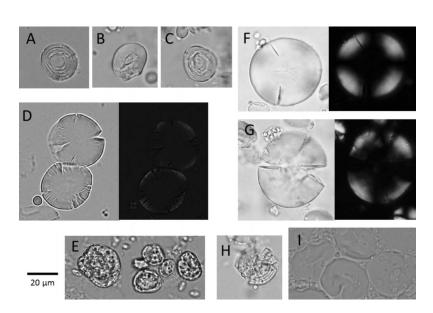


Fig. 10. Starch granules from modern reference samples of processed grain. A–C: Common wheat malt (Triticum aestivum). The hole in the centre of the granule indicates germination of the seed. D: Barley malt (Hordeum vulgare). A hole in the centre is beginning to form due to germination. Cracking of the outer edge indicates dry heating. The extinction cross has nearly disappeared. E: Overly germinated barley malt. Granules are pricked with holes which do not appear to malt germinated the traditional way. F–H: Rye malt (Secale cereale). Germination is visible in the centre of the granules. I: Cooked barley. The granules have swollen and partly gelatinized in boiling water. The extinction crosses have vanished.

4000 BC) and in Poland from Funnel Beaker culture (2825–2665 BC) (Zohary & Hopf 2000: 106–7). In Sweden, peas and broad beans (*Vicia faba*) have been present since the Early Neo-lithic (Welinder et al. 1998: 74). Legumes became less frequent in European sites during the Bronze Age, only to increase again during the Iron Age (Zohary & Hopf 2000: 106–7).

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In Finland, pollen from common pea has been found in Niuskala, dating from 500 BC, and macrofossils of common pea and broad bean have been recovered from a cairn in Laitila from the Merovingian Period (AD 575–800). In Vammala, an imprint of a lentil seed has been detected on a ceramic sherd from the Migration Period (AD 400–575) (Huurre 2003: 48–9).

At the time of the cubic stone, legumes were cultivated in Finland and probably had an established place in the food culture. The fact that the possible legume starch was found on the grinding stone is not unusual. In many countries, legume seeds are ground to flour to provide traditional meals.

Millet

The sample from the cubic stone yielded a small faceted starch granule (approximately 11 μ m in diameter) which correlates with our reference samples from broomcorn, foxtail and green foxtail millet species. Also millet grains require dehusking by pounding before cooking for food. In central Europe millet was known by the Romans and cultivated widely in Europe in the Middle Ages (5th – 15th centuries AD) (Klemettilä 2007: 13, 43, 47).

Starch residues in 18th century dental calculus

A tooth from a mass grave dated to the 1740s was selected for this starch analysis because of its large calculus deposit. The tooth was a single find and it is not known to which skull it was formerly attached. The mass grave was situated in a military area in the centre of Hamina, next to the fortress built in the 1720s during Swedish rule, and is one of the mass graves associated

with the Russo–Swedish War (AD 1741–1743) (pers. comm. Anne-Mari Liira; pers. comm. Riku Kauhanen). In 1743, Hamina became the Russian city Friedrichshafen. It has been suggested that the deceased belonged to the Swedish navy or army, but to our knowledge no artefacts such as crucifixes or other evidence marking nationality or denomination were found in the graves (pers. comm. Anne-Mari Liira).

The dental calculus sample removed from the 18th century tooth yielded 24 starch granules, of which 22 were given preliminary identifications by comparing them to modern reference samples and descriptions in the literature (Table 5, Figs. 4, 6, 8–10). Twelve of the starch granules were from potato (*Solanum tuberosum*), eight were from millet species (*Setaria italica* and possibly others), one granule was an unidentified grass species (including cereals) and one granule was possibly from processed cereal grain.

Potato

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An impressive number of potato starch granules were retrieved from the calculus sample, interestingly, since potato is considered a very rare food in the 18th century AD in Finland, Sweden and Russia. In Sweden, potato did not become common before the 1770s AD (Gadd 2011: 148), and in Russia it was necessary for Catherine the Great (reign AD 1762–1796) to command her people to eat potatoes (Paalo 2007: 12). Despite the efforts of certain enthusiasts from the 1720s onward, potato was long considered 'the devil's plant' in Finland and was not completely accepted before the end of the 19th century AD (Paalo 2007: 19).

Potato starch granules were very large, in these samples up to four times as large as any grain starch granule (Figs. 4&9). The starch in root vegetables also differs in morphology to starch in seeds. Both hilum and growth rings are offcentred and the shape is usually oblong or oval (Messner et al. 2008: 121).

Millet

Several possible millet starch granules were found in the calculus sample and were identified as foxtail millet because of their large size. Smaller granules, on the other hand, could also be defined as broomcorn millet. To our knowledge millet was not cultivated in Sweden in the 18th century AD (Gadd 2011: 118–64), but we assume that it was a common grain in Russia. It should be taken into account that although millet has often been regarded as nonexistent in historical Finland, this is not the case. Millet flatbread and milletstuffed pies have been documented as part of the Finnish Karelian food culture (Uusivirta 1982: 172–3, 198). Hamina is located in southeastern Finland close to the historical Karelian county.

Cereal starch

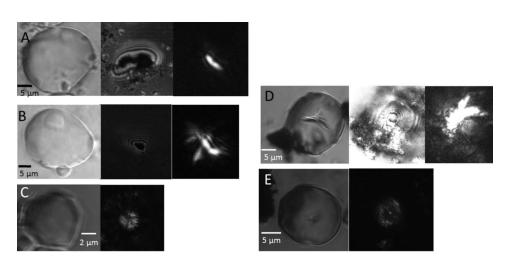
One large cereal starch granule (Fig. 4A, size approximately 27 x 25 µm), which showed apparent signs of degradation, gelatinization or processing, was found in the calculus sample. The granule showed possible signs of germination in the centre of the granule, cracking on the edges which may be produced by intense heating in dry conditions, as well as slight gelatinization on the top edge, which could be evidence of cooking, suggesting consumption of malted products, as malt is cooked in the process of brewing beer or cooking malt porridge. The small granule next to the large granule indicated bimodal size distribution, typical to cereal starch. However, it is also possible that this cereal grain was not malted, but that the digestive enzyme in saliva and the physical process of chewing food has affected the starch granule (see Englyst & Hudson 2000: 67-8).

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It is generally known that beer played a major

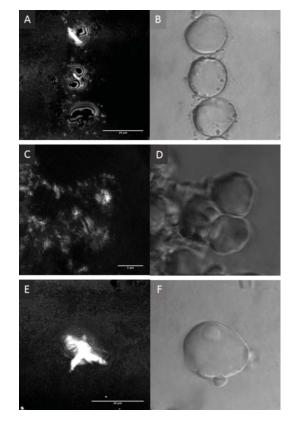
Identification	Number of starch granules
Cereal starch granule, possibly processed	1
Grass family (Poaceae), species not identified	1
Foxtail millet (Setaria italica)	3
Millet (for instance Panicum / Setaria), species unidentified	5
Potato (Solanum tuberosum)	12
Unidentified	2

Table 5. Starch granules found on the dental calculus sample from the 18th century AD.



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Fig. 11. CLSM scans of reference and fossil starch granules recorded while using transmission light (left), polarized light (middle) and reflected light (right). A: Barley (Hordeum vulgare). When conducting a set of optical cross-sections through the granule, the diffraction signal produced a robust ring-like pattern and the reflection signal a surface reflection. B: Emmer wheat (Triticum dicoccon). There is a more delicate ring-like diffraction and also a surface reflection. C: Broomcorn millet (Panicum miliaceum). Scanning produced a distinctive central spot of reflected light but no ring-like diffraction or surface reflection. D: Scans from the fossil granule in Figure 2A. E: Scans from the fossil granule in Figure 2G.

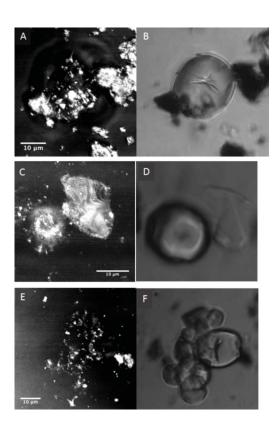


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Fig. 12. CLSM scans of reference starch granules with left column showing maximum projections of reflected polarized light and right column the transmission signal. A and B: Barley (Hordeum vulgare). C and D: Broomcorn millet (Panicum miliaceum). E and F: Emmer wheat (Triticum dicoccon).

part in 18th century cuisine and provided necessary calories. Beer was made from barley or rye and used as a beverage as well as in soups in Sweden (Morell 2011: 188) and Finland.

Although starch analysis of the 18th century dental calculus did not give a definite answer as to the nationality of the deceased, it does give an interesting insight into the early 18th century diet. Strong evidence of beer consumption was to be expected, but the presence of potato starch was a surprise. Millet starch, on the other hand, gave an eastern tendency to the result, hinting that the deceased may have been Russian or Karelian. But the situation may be more complicated, because according to researchers, dental calculus may trap starch granules over a long period of time, possibly over the whole lifetime ۲



of the person (Henry & Piperno 2008: 1944; Piperno & Dillehay 2008: 19622; Hardy et al. 2009: 250). Soldiers may have eaten local foods wherever they were stationed, which may have affected the starch deposits in their calculus.

CONCLUSIONS

This pilot project presented some examples of the possibilities that starch research can bring to archaeology in Finland and other northern countries. Numerous starch granules were retrieved from all three artefacts from different ages, proving that starch can survive for thousands of years on artefacts. The samples were selected from three different artefact types, ceramic, stone tool and tooth. However, many other artefact types, for instance stone blades, grinding and milling stones, cooking stones, and wooden vessels, could also be sampled for starch analysis.

By utilizing starch analysis we were able to track development of agriculture, non-monoculture crops and sources of dietary carbohydrates during Late Stone Age. In the Iron Age sample we saw evidence of processing starchy plant Fig. 13. CLSM scans of fossil starch granules with left column showing maximum projections of reflected polarized light and right column the transmission signal. A and B: Fossil starch in Figure 2A. C and D: Scans from the fossil granule in Figure 2G. E and F: Scans from the fossil starch in Figure 2B.

foods. New information on the origins of the deceased in a mass grave was gained when starch from the calculus sample was compared to the typical diets of the period.

The key to reliable results is a comprehensive reference collection which has been studied and documented in order to aid recognition of the different variations in each species. We have plans to build such a collection of wild plants which grow in Finland today or have grown in ancient times and which could have been used as food in prehistoric times. In this preliminary study, the focus was on identifying cultivated grain species, but in the future a large collection of wild roots and cultivated root crops should also be added to the reference material.

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A major risk in starch analysis is contamination with modern starch, which may occur through contact with hands, tools, surfaces, and even air and water. This risk should be minimized by adopting preventive measures at the excavation site, during the journey from the excavation site to the laboratory, and finally in the laboratory as well. The development of clean procedures is in progress, and in the future the results will become more reliable as the contamination risk decreases. Control soil samples from the context of the artefacts should be analysed in order to test for contamination. Also it may be necessary to conduct research to discover best methods and procedures. Special attention should be directed to soil removal from the artefacts before sampling. It is however challenging to find a method which would remove all the microscopic soil particles without, for example, shifting them into the cracks and crevices of the food crust. Finding a method for cleaning stone tools without removing fossil starch from the surfaces seems very problematic, if not impossible. As the use of HCl has since been criticized, we have investigated more gentle preparation methods for the future analysis of dental calculus.

Despite the fact that this starch project was the

very first of its kind in Finland, it succeeded in providing insight into new possibilities in the microscopic analysis of cultural remains. Combining starch analysis with other lines of microscopic or chemical research, such as macrofossil, pollen, lipid, and isotope research, will eventually produce a network of evidence for nutrition and subsistence studies. As new analysis methods have become available, the role and concept of an archaeological artefact is rapidly changing. An artefact is no longer only a museum specimen, a piece to look at, but is rather an archive or a treasure chest of chemical and biomolecular information.

NOTE

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The Kiukainen culture pottery sherd was radiocarbon dated to 2880±30 BP (Poz-66814), i.e. 1131–973 calBC (88.8%).

A cavity containing an imprint of a cereal grain (Cerealia), possibly barley (cf. *Hordeum vulgare*), was detected on the surface of this ceramic sherd. A cast of the impression was made with dental silicone Xantopren Comfort Light. The cast was studied with a stereo-microscope and compared to modern cereal grains. The identification is preliminary and will be confirmed with SEM-pictures, but the cast has been shown to Professor Karin Viklund and Professor Emeritus Roger Engelmark (University of Umeå), who agree with the identification (pers. comm. Santeri Vanhanen).

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