KAUNAS CASTLE FROM ANOTHER PERSPECTIVE: THE CHEMICAL COMPOSITION OF IRON CONSTRUCTION ELEMENTS

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Abstract

The chemical composition of iron construction elements is not usually a topic of interest in Medieval archaeology. This article focuses attention on the chemical composition of iron elements such as nails. For compositional analysis, the XRF method was used. A comparison was made between XRF results and physical measurements. The aim of this article was to identify the amount of iron in metal elements that were possibly used in constructing castles, and to find a connection between the chemical composition of objects and their physical measurements.

Key words: Portable XRF, chemical composition, iron, metallurgy, Kaunas castle, Medieval period.

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Introduction

It is widely accepted that with the introduction of brick and stone castles in the 14th century in the current territory of Lithuania, iron started to be used in construction (Kuncevičius 2005, 163; Zabiela 1995, 118). There is no evidence that large amounts of iron were used in building before this period, even despite the fact that in the territory of Lithuania, during the first and the early second millennium AD, wooden castles were built on prominent hills, usually referred to as hill-forts (Zabiela 1995, 117).

In some cases, large amounts of iron objects are found during excavations of the sites of former stone castles, and in other cases less. Iron construction elements, such as nails, hinges, hangers and castings, show the technical skill and economic development. There is not yet enough research to identify these artefact types and their chronology. It is also important to know what kind of material the object is made from. This gives more information about trade routes, natural resources and technical progress.

In collaboration with the M.K. Čiurlionis National Museum of Art in Kaunas, it was decided to conduct an XRF analysis to find out the chemical composition of metal building elements from Kaunas Castle. The main aim of the research was to identify the amount of iron in the metal that was possibly used in the construction of the castle. Also, the adaptability, challenges and prospects of the method were encountered during the research, and that will be discussed here as well.

Historical context

Kaunas Castle is built in a strategically convenient place, at the confluence of the rivers Nemunas and Neris. It is an established fact in historiography that the Crusaders destroyed the first Kaunas Castle in 1362 (Mekas 1971b, 153; Žalnierius 2004, 205) which was built in the mid-14th century (Žalnierius 2002, 8). According to archaeological, historical and architectural research, the first castle was an enclosed castle, made of old and large boulders (Mekas 1971a, 8-9; Abramauskas 2012, 64). The second Kaunas Castle, built on the foundations of the first, is already mentioned in 1368 (Mekas 1971b, 153). The castle was a reconstruction of the enclosure type in the Gothic style, with towers at the corners (Abramauskas 1987, 95). The cultural layer formed during the existence of the second castle dates back to the turn of the of the 14th and 15th centuries up to the 17th century (Žalnierius 2002, 8). After the wars with the Teutonic Knights ended, the castle lost its military importance. Later, around the end of the 15th century and the beginning of the 16th century, the castle became a regional population centre (Sirutavičius 2000, 159). According to archaeological data, the decline of the second castle dates from the mid-17th century AD (Žalnierius 2002, 8).

The first archaeological excavations of Kaunas Castle started in 1930, due to the concern on the city council for the city's oldest architectural monument (Mekas 1971a, 3). The excavations were supervised by E. Volteris and K. Mekas. They lasted for several years, were discontinued, and resumed in 1938; but after the war began, the work stopped and only resumed in 1954 (Mekas 1971a, 3). Archaeological research was carried out in 1954-1958 by P. Tarasenka and R. Rimantienė, after 1955 by K. Mekas, and after 1958 by E. Milčiūtė (Mekas 1971a, 3). Since 1989 (Žalnierius 1990), 1991 (Žalnierius 1996), 1994 (Žalnierius 1996), 1996 (Žalnierius 1998), 1998 (Žalnierius 2002b), 2000 (Žalnierius 2002b), 2005 (Žalnierius 2006), 2007 (Žalnierius 2008), 2009 (Žalnierius 2010) and 2011 (Žalnierius 2012; Žalnierius et al. 2012), excavations were performed by A. Žalnierius.

Material

Thirteen samples (ten nails and three other items: a hook, door or gate flap, and a connecting piece) from the depositories of the M.K. Čiurlionis National Museum of Art in Kaunas were selected for the research. The inventory book states that all the investigated objects were found during excavations of the eastern oval of the tower in 1954.[1]

1. Hook-nail (Inv. No. Tt–4400; Fig. 1). The hook-nail was hammered next to a door or a jamb. Pins were attached to the eyelets, which, when fitted with a wooden or metal bar with a shaped opening at one end, had to fill the resulting opening and prevent the door from opening (Kaminskaitė 2017, 86). The eyelet selected for the test is about 13 centimetres long, curved at 11 centimetres, stem thickness 0.8 x 0.8 cm, eyelet hole diameter 0.5 centimetres, weight 46 grams.
2. Nail (Inv. No. Tt–4403; Fig. 2). The object is classified as Type A, nails with circular heads (Rimkienė 2019). These types of nails were used universally, depending on the need. The total length of the nail is 19 centimetres, stem thickness 1.5 x 1.5 centimetres, head diameter 2.5 centimetres, weight 130 grams. Based on the measurements, it can be concluded that the nail was used in the construction of the castle.
3. Nail (Inv. No. Tt–4404; Fig. 3). The object is attributable to Type K nails with square heads (Rimkienė 2019). This type of nail could have been used as a decoration, but at the same time it had a protective function against mechanical damage, as well as for fastening purposes like sheet metal, wood and leather. The nail head in question has a more rectangular shape with two forging marks clearly visible on the two sides, creating a figure of eight or infinity sign shape. The total length of the nail is 15 centimetres, stem thickness 1.2 x 1.2, head size 3 x 1.5 centimetres, weight 44 grams.
4. Nail (Inv. No. Tt–4405; Fig. 4). The object is attributable to the KTB type, headless nails (Rimkienė 2019). This type of nail could have been used for fastening and joining wooden structures. The total length of the nail is 17 centimetres, stem thickness 1.5 x 1.5 centimetres, weight 74 grams.
5. Nail (Inv. No. Tt–4406; Fig. 5). The object is attributable to the KTB type, headless nails (Rimkiene 2019). As before, this type of nail could have been used for fastening and joining wooden structures. The total length of the nail is 20 centimetres, stem thickness 1.5 x 1.5 centimetres, weight 130 grams.
6. Nail (Inv. No. Tt–4407; Fig. 6). The shape of the nail head is rather obscure. Basically, it could be classified as PA type nails with semi-circular heads (Rimkienė 2019). Usually, this type of nail head is a combination of quadrilateral and circular, but in this case it is a mixture of tapered oval and quadrilateral. This type of nail is considered to be a decorative element, serving a protective function against mechanical damage. The total length of the nail is 14.3 centimetres, stem thickness 1.5 x 1.5, head size 4 x 3 centimetres, weight 52 grams.
7. Nail (Inv. No. Tt–4408; Fig. 7). The object is attributable to the KTB type, headless nails (Rimkienė 2019). As mentioned above, this type of nail could have been used for fastening and joining wooden structures. The total length of the nail is 15 centimetres, stem thickness 1 x 1 centimetres, weight 44 grams.
8. Nail (Inv. No. Tt–4409; Fig. 8). The object is attributable to the KTB type, headless nail (Rimkienė 2019). The total length of the nail is 14.5 centimetres, stem thickness 1 x 1 centimetres, weight 26 grams.
9. Nail (Inv. No. Tt–4410; Fig. 9). The subject is classified as a PA type nail with semi-circular head (Rimkienė 2019). This type of nail is considered to be a decorative element serving a protective function against mechanical damage. The total length of the nail is 12.6 centimetres, stem thickness 0.5 x 0.5, head size 1.2 x 0.8 centimetres, weight 14 grams.
10. Nail (Inv. No. Tt–4413; Fig. 10). The object could possibly be categorised as a nail, because it has the characteristics of a nail, a head and a stem. However, the unusual shape of the head raises some doubt as to whether it is actually a nail. Although

[1] Museum inventory book: Tt–5. No. 4099–5129. M.K. Čiurlionis National Museum of Art. Department of Applied Arts.

Fig.1. Hook-nail (M.K. Čiurlionis National Museum of Art, Inv. No. Tt–4400; photograph by E. Rimkienė).

Fig.2. Nail (M.K. Čiurlionis National Museum of Art, Inv. No. Tt–4403; photograph by E. Rimkienė).

Fig.3. Nail (M.K. Čiurlionis National Museum of Art, Inv. No. Tt–4404; photograph by E. Rimkienė).

Fig.4. Nail (M.K. Čiurlionis National Museum of Art, Inv. No. Tt–4405; photograph by E. Rimkienė).

Fig.5. Nail (M.K. Čiurlionis National Museum of Art, Inv. No. Tt–4406; photograph by E. Rimkienė).

Fig.6. Nail (M.K. Čiurlionis National Museum of Art, Inv. No. Tt–4407; photograph by E. Rimkienė).

Fig.7. Nail (M.K. Čiurlionis National Museum of Art, Inv. No. Tt–4408; photograph by E. Rimkienė).

Fig.8. Nail (M.K. Čiurlionis National Museum of Art, Inv. No. Tt–4409; photograph by E. Rimkienė).

Fig.9. Nail (M.K. Čiurlionis National Museum of Art, Inv. No. Tt–4410; photograph by E. Rimkienė).

Fig.10. Nail (M.K. Čiurlionis National Museum of Art, Inv. No. Tt–4413; photograph by E. Rimkienė).

Fig.11. Structural detail (M.K. Čiurlionis National Museum of Art, Inv. No. Tt–4414; photograph by E. Rimkienė).

Fig.12. Nail (M.K. Čiurlionis National Museum of Art, Inv. No. Tt–4415; photograph by E. Rimkienė).

Fig.13. Structural detail (M.K. Čiurlionis National Museum of Art, Inv. No. Tt–4423; photograph by A. Kapčius).

similar to the trapezoidal form, it differs from those found in the Crusaders' castles (e.g., Veliuona unnamed castles, KT type (Rimkienė 2019). The head appears to be made from the same rod as the stem. The connection line at the border of the stem and the head was also overlooked, which would be an unusual practice in the castle's lifetime. The total length of the nail is 12 centimetres, stem thickness: 1 x1 centimetres, head size 3.5 x 2.5 centimetres, head height 0.5 centimetres, weight 86 grams.

11. Structural detail (Inv. No. Tt–4414; Fig. 11). This object is chronologically questionable. The object consists of a stem and an intervention plate attached to it. The overall length is 16 centimetres, stem thickness 1 x 1 centimetres, plate length four centimetres and width two centimetres, thickness 0.7 centimetres.
12. Nail (Inv. No. Tt–4415; Fig. 12). The object is attributable to the KTB type, headless nail (Rimkienė 2019). However, the shape of the head is uncertain. The nail could be either KTB type, or, for some reason, a specially shaped head from the stem, since the bending angle is two centimetres from the beginning of the nail. This practice seems to have been applied in the castles of the Grand Duchy of Lithuania, since nails of a similar kind were found in the castle on Trakai Island.[2] The total length of the nail is 25 centimetres, stem thickness 1.5 x 1.5 centimetres, weight 206 grams.
13. Structural detail (Inv. No. Tt–4423; Fig. 13). The object was possibly used as a structural element where rebuilding was required. It is oblong, rounded at one end, and severed or broken at the other. A cylinder ten centimetres long and four centimetres in diameter is attached to the widest plane of the part. The piece is eight centimetres wide at its widest point, 15 centimetres long, and two centimetres thick.

Method

XRF is a very fast and non-destructive method for analysing material that does not damage the sample. The chemical composition of a sample is determined by measuring the characteristic X-ray spectrum emitted by different chemical elements present in the sample when exposed to high-energy photons (X-rays). These photons are emitted from a small X-ray tube in the analyser. Secondary characteristic X-rays occur when a photon of sufficient energy strikes the atom (present in the specimen), knocking out the electron from the atom's inner orbital layer. An atom returns to a steady state when the void left is filled by an electron from the atom's higher energy level ('falls' from a higher energy level to a lower energy level). This electron transitions to a lower energy level by emitting a secondary X-ray photon, whose energy (usually measured in electron volts, eV) is equal to the difference in energy between the two quantum states of the 'fallen' electron. When a

[2] Author has found similar examples during the visit in the Trakai History Museum depositories, the material is unpublished.

sample is analysed by fluorescence X-ray, each chemical element in the sample emits its own specific spectrum of secondary X-rays. By measuring a wide range of fluorescent X-rays emitted by different chemical elements in a sample, the NITON analyser quickly determines the concentration of chemical elements in the sample.[3]

The research was conducted by a private company, Independent Research. All samples were measured by portable Niton XL2 PLUS technical equipment.

Table 1. The pros and cons of portable XRF method

Plus	Minus
Non-destructive	Limit on sample size
Minimal preparation	The research yields a limited number of items.
Fast	XRF cannot characterise fine elements
Easy to use	

The method applied here is non-destructive, which in itself is a plus, when working with material kept in museums. At the same time, a trained person can quickly and efficiently scale vast museum collections (Roxburgh et al. 2019, 57; Shackley 2011, 9). Data collected by this method can provide information on the nature of craft production (Roxburgh, van Os 2018, 306), as well as the place of origin of the raw material.

Despite all the pros, the method, and in particular the results and their interpretation, receives considerable criticism. In most cases, surface preparation of objects is either unnecessary or minimal, but this does not apply to metals, where chemical conditions can alter the surface composition, and can affect the results (Shackley 2011, 9). Degradation and/or contamination of metal surfaces, and the way in which they are preserved, can alter the composition of the surface to avoid the need for cleaning, for articles previously treated with preservative chemicals containing zinc or other metals (Tykot 2016, 50). Unfortunately, no surface cleaning procedure was performed in this study. Thus, from a perspective point of view, it would be useful to carry out a comparative analysis of the article to determine the chemical composition before and after the surface was cleaned.

Outside factors that may influence the results:

1. Soil
2. Preservatives
3. Storage conditions of articles and oxidative processes
4. Incorrect sample preparation
5. Device error
6. The device captures only one point on the item

Results and discussion

The results of the XRF method applied to research of iron finds from Kaunas Castle are shown in Table 2. The data presented indicates that the iron content in over 12 objects exceeds 94% of the composition of the product. One group of objects here is nails, whose iron content is only 76% (Tt-4409). Iron and copper were found in all the studied objects. Lead was found in ten at concentrations up to 1%, but the object Tt-4409 was found, even 20% of the product composition.

Another interesting aspect of the study could be the relationship between the metal composition and the weight and dimensions of the objects. Nails were chosen as a control group, because they formed the main and the largest group in terms of the number of objects examined. Nails nos Tt-4409 and Tt-4408 are noteworthy, belonging to the same type, nails without heads. They both have similar lengths, but vary in weight by almost two times. The chemical composition analysis showed that nail no Tt-4408 99% is composed of iron, and nail no Tt-4409 only 76%; also, 20% lead was found in it. The strangest thing is that lead is a heavier metal, but the nail that has it is light. Another case that drew our attention is nails nos Tt-4406 and Tt-4415, which are of the headless type. Nail no Tt-4406 has an overall length and weight less than nail no Tt-4415. However, after some elementary mathematical calculations, nail no Tt-4415 should weigh 44 grams less. A look at the chemical composition of both objects shows that iron accounts for 98% of nail no Tt-4415, and 97% of nail no Tt-4406. One per cent could possibly affect the weight, all the more so because both nails had a similar chemical composition. However, nail no Tt-4415 has a titanium content of 0.095% by weight in the composition.

After the results obtained, the question arises about the raw material of the products. The chemical composition of the articles is varied, and, as is mentioned above, iron and copper are the main constituents. However, the abundance of other elements is questionable. This study was conducted as an experiment to test the feasibility of the method within the confines of the topic being studied. Obviously, the number of specimens is too small for broader conclusions. However, studies by other researchers have shown that the degree of uniformity of composition found in large groups of objects

[3] Information taken from technical leaflet.

Table 2. The results of XRF analysis of chemical composition

Nr.	Object	Inv. nr.	Mo (%)	V (%)	Mn (%)	Fe (%)	Cu (%)	Pb (%)	Co (%)	Ti (%)	Nb (%)	Zn (%)	Zr (%)	Cd (%)	Sn (%)
1.	Hook-nail	Tt–4400				98.44	0.51	0.03			0.03	0.70			
2.	Nail	Tt–4403			0.49	96.80	1.55	0.02	0.30			0.72			0.02
3.	Nail	Tt–4404				97.35	0.84	0.09			0.06	1.17			
4.	Nail	Tt–4405			0.08	97.85	1.01	0.05				0.61			0.05
5.	Nail	Tt–4406				97.97	0.75	0.05				1.01			
6.	Nail	Tt–4407				97.66	0.89	0.10		0.15		0.85			
7.	Nail	Tt–4408				99.10	0.28			0.12	0.91	0.32	0.02		
8.	Nail	Tt–4409				76.87	0.34	20.45	0.54			1.67			
9.	Nail	Tt–4410				99.51	0.24					0.23			
10.	Nail/ Décor element	Tt–4413	0.09		0.43	97.78	0.11	0.02	0.77			0.62	0.02		
11.	Con-struction detail	Tt–4414	0.16	0.11	0.52	94.80	2.03	0.21			0.31	1.25	0.10	0.05	
12.	Nail	Tt–4415				98.54	0.47	0.08		0.09		0.71			
13.	Con-struction detail	Tt–4423				97.29	1.15	0.64	0.33		1.03				

is likely to indicate something more about the organisation of the work, the transfer of technical knowledge, and interaction between different crafts. A high degree of uniformity over a wide geographical area may indicate that the raw materials were also supplied centrally, or that the artefacts were mass-produced at a central workshop. In contrast, significant changes in composition may indicate differences in production methods, and differences in the supply of raw materials, including differences in processing (Roxburgh et al. 2019, 57).

Further, the theoretical possibilities of supplying raw material for ironwork and the construction of Kaunas Castle can be explored. According to historical data, in the 14th and 15th centuries, metal raw materials were transported by the Vilnius-Kaunas- Königsberg-Danzig inland and waterways route (Kiaupienė, Petrauskas 2009, 110). After the opening of the Hanseatic League's office in Kaunas in 1440-1441, one of the most active merchants was a Danzig merchant, who, among other things, also sold iron (Kiaupienė, Petrauskas 2009, 113). However, Kaunas Castle was built at the end of the 14th century. So, where did the iron for the construction come from? Of course, there were two options: either it was made from imported raw material, or from domestic wetland ore. The latter is quite widespread in Lithuania, it is found in wetlands and wet forests at a depth of 30 to 80 centimetres. Local ore is porous, has less iron, but is easily reduced to iron (Navasaitis 2004, 20). According to A. Endzinas, the ore found in 1960 during excavations of Kaunas castle was brought from the surrounding ore (Endzinas 1964, 195). The author has published a publication on the development and geography of iron production in Lithuania (Endzinas 1964). In this publication, he correlates archaeological research data with place names. This identifies the probable locations for iron extraction and processing. Of course, associating place names with potential locations for production and extraction can be a challenging and confusing factor, as we often do not know how long a given place name has existed for. However, the work has attracted attention, because other authors seem to be inclined to trust the results of the study (Navasaitis 2004; Malinauskas, Linčius 1999), but there are also critics (Salatkienė 2009). According to Endzinas, as craftsmen developed their skills, the process of smelting iron was moved from the field to smithies (Endzinas 1964, 195). Blacksmiths and smithies were also to be found near castles, because iron was expensive, and Lithuanian nobles tried to concentrate iron resources in their own hands, so that in the event of an attack, they could be defended, in order not to fall into the enemies' hands. Looking at the maps made by Endzinas, several locations around Kaunas can be seen. However, it is difficult today to determine the distance from which raw materials or products could have been supplied. Because the ore was mined in different locations, the resource was transported, and the life of one deposit could be from one to a hundred years. The more permanent locations were the smithies and blacksmiths that were built near the ore deposits, and after their exhaustion continued to serve their purpose or replace it.

During the Middle Ages, much of the local raw material was consumed by warfare and for noble luxury items (Malinauskas, Linčius 1999, 114). J. Stankus, using the method of metallographic analysis, researched the technology of ironwork manufacturing in Lithuanian cities from the 14th to the 16th centuries, and found that the hot-forged metal forging technique was used for forging nails (Stankus 1999, 185). The author also found that the nails were forged from a monolith, while claiming that iron of various structures was used for the forged articles (Stankus 1999). Although metallographic analysis is a more accurate method, the number of nails investigated in this study does not allow for broader conclusions regarding the product equivalence typologically.

Conclusions

The purpose of this study was to find out the amount of iron in structural parts used in the construction of Kaunas Castle, and at the same time to look at the theoretical possibilities to find where the raw material could be sourced, and to test the XRF method in the structural details of artefacts. Based on the results of the study, it can be concluded that the 12 samples tested had an XRF iron level above 90%, and one sample only had an iron level above 70%. The relationship between the chemical composition and the nail typology could not be determined, due to the insufficient sample size. However, this is a starting point for future research. The application of the XRF method could only be partially justified in this case, since factors such as preservatives, article storage conditions, oxidation processes, improper sample preparation and device error could have had a direct impact on the distortion of the chemical composition. However, it is theoretically possible to locate the raw materials of articles from which they were supplied for the construction of Kaunas Castle, but relying solely on the surrounding place names would be too naïve, especially knowing that other places were searched for after a source of ore was exhausted. The exact period of existence of these place names is also unknown.

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KAUNO PILIS IŠ KITOS PERSPEKTYVOS – GELEŽINIŲ KONSTRUKCIJOS DETALIŲ CHEMINĖ KOMPOZICIJA

EGLĖ RIMKIENĖ

Santrauka

Kauno pilis buvo viena pirmųjų mūrinių pilių, statytų Lietuvoje XIV amžiuje. Dalis archeologinių tyrimų metu surinktos statybinės geležies artefaktų yra saugomi Nacionalinio M. K. Čiurlionio dailės muziejaus fonduose. Tyrimo tikslas buvo sužinoti geležinių artefaktų, naudotų statybose, cheminę kompoziciją. Šiam tikslui įgyvendinti buvo pasirinktas nedestruktyvus metodas – XRF analizė, kurią atliko UAB „Nepriklausomi tyrimai“ su Niton XL2 PLUS. Tyrimui buvo atrinkta 10 vinių (2–10, 12 pav.), kilpvinė (1 pav.), varstomoji detalė (11 pav.), jungiamoji detalė (13 pav.).

Atsižvelgus į tyrimo rezultatus, darytinos išvados, kad dvylikoje XRF metodu ištirtų mėginių geležies koncentracija viršija 90 proc. ribą, viename mėginyje geležies koncentracija viršijo tik per 70 proc. ribą. Nustatyti ryšio tarp cheminės kompozicijos ir vinių tipologijos nepavyko dėl per mažo bandinių kiekio. Tačiau tai yra startas ateities tyrimams.

XRF metodo taikymas šiuo atveju galėjo pasiteisinti tik iš dalies, kadangi tokie veiksniai kaip konservavimo medžiagos, dirbinių laikymo sąlygos, oksidaciniai procesai, netinkamas bandinio paruošimas, prietaiso paklaida galėjo turėti tiesioginę įtaką cheminės kompozicijos rezultatų iškraipymui.

Teoriškai nustatyti gaminių žaliavos vietas, iš kur ji buvo tiekiama Kauno pilies statybai, yra įmanoma, tačiau remtis vien aplinkiniais vietovardžiais būtų per daug naivu. Ypač žinant, kad pasibaigus balų rūdos ištekliams, buvo ieškoma kitų vietų. Taip pat nežinomas ir tikslus tų vietovių pavadinimų egzistavimo periodas.