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Research paper

Characteristics of 13th-century mortars from the tower at Lublin Castle

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Abstract: The tower at Lublin Castle, known as the donjon, is the only monument of Romanesque art on the eastern side of the Vistula River. The cylindrical, brick building is part of the Lublin Castle complex. During contemporary restoration work, the 13th-century walls were uncovered, making it possible to retrieve the original materials. The article presents the mineralogical, chemical and granulometric characteristics. The analytical methodology included: qualitative mineralogical analysis of the whole sample by X-ray diffraction (XRD); morphological studies with elemental evaluation and microanalysis of the binder by scanning electron microscopy and energy-dispersive X-ray spectroscopy (SEM-EDS). Residue determination of hydrochloric acid-insoluble parts and their granulometric analysis were performed. This methodology enabled to determine the composition of the mortars. It was found that the tested mortars have a similar mineralogical structure, but slightly differ in the proportion of individual components, primarily aggregate fractions. The samples consist mainly of quartz, calcite, and silicates. Moreover, the lime binder of the mortars was shown to be microcrystalline in nature. The aggregate used in historical mortars predominantly consisted of quartz sand and minerals of the feldspar and silicate group. The condition of the mortars requires conservation interventions. The performed characterization of the historic mortars was important for designing compatible restoration mortars.

Keywords: historical mortars from the 13th century, research, Lublin castle tower

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1. Introduction

The tower on the Castle Hill in Lublin is the only known monument of the late Romanesque period in the historic Lublin province. The tower was erected around the middle of the 13th century in the form of a cylindrical, homogeneous building showing the features of the Late Romanesque style [1, 2], in a typical manner for 13th-century fortresses, i.e. inside the defensive ring, as the main center of the fortification (Fig. 1A).

Fig. 1. A) Castle tower in the view of Lublin in Civitates Orbis Terrarum, by A. Hogenberg, G. Braun, Cologne 1618, B) Cylindrical tower blended into the south wing of the castle, now the Lublin Museum

The tower, which is a building on a circular plan, has never been rebuilt on a larger scale. It stands in the courtyard of the castle, partially blending into its southern wing with half of its perimeter. It has three stories. A crenellation placed on a lunette frieze is located on its top. The lower parts of the tower were built from broken limestone and brick, whereas the upper parts – from brick. The building has a basement, with three underground floors, and is covered with a timber-framed roof. On the roof, there is a wooden belvedere with a gallery. Inside there is a vaulted spiral staircase, running inside the wall from the lowest basement floor to the present attic. All floors have domed vaults.

There are three types of windows: narrow, rectangular windows with a glyph opening inward, rectangular windows with embrasures, and half-circle windows with a narrowing inside the wall. On the top floor, there is a biforium with a preserved square stone column with chamfered corners, passing to a trapezoidal capitol supporting a pair of brick arcades. The entrance to the tower leads through a doorway in the ground floor, from where a spiral staircase inside wall can be used to access the basement or the upper floors of the tower (Fig. 1B).

Along with the castle chapel, the tower was the only part that survived after the destruction of the castle during the wars in the 17th century [3]. Since 1826, the tower and the castle served as a prison. Since 1957, it has housed the exhibition rooms of the Lublin Museum.

However, the significance of the object for the history of the castle and the city has not translated proportionately into the scope of the research undertaken. This is because no

planned and systematic architectural or preservation research has been carried out so far, which would be aimed at clarifying the basic issues related to the object: the workshop and the building material used.

The objective of this paper was to characterize the microstructure and mineral composition tests of mortars collected from the walls of thirteenth century from the tower at Lublin Castle.

A combination of techniques was used, which included X-ray diffraction, scanning electron microscopy with micro-area elemental composition analysis and granulometric characteristics.

2. Materials and methods

The study and characterization of historic mortars is an important tool for identifying the origin of components, and is also used to obtain the information about the historical evolution of the monument [4–7].

The technical properties of mortars depend on the raw materials [8]. The composition of mortars (the nature of the ingredients) and texture (which depends on the composition of the mass and grain size distribution) are the most important features in terms of technical compatibility between the historical material and the newly designed material [9].

The production of mortars is a complex process, involving the knowledge of materials and technology [4]. Therefore, in order to gain the necessary information about historical mortars, studies are performed using chemical, mineralogical, and petrographic methods [5, 6].

The results obtained can be used to acquire the information on the characteristics of the binder, aggregate and possible additives. In many cases, the number of available samples and their size is not sufficient for physical-mechanical testing [4, 5]. Sampling follows the principle of minimal damage to the historic substance; it usually involves using a chisel and small hammer, rather than core drilling [10].

As part of the ongoing research, six representative samples were taken from various locations of the wall, dated to the 13th century by architectural researchers and art historians (Fig. 2).

The sampling procedures are carried out with great care under the supervision of conservation personnel, so that the samples taken will satisfy the needs of the planned analytical studies (Fig. 3).

Macroscopic observation of the samples (Fig. 3) showed homogeneous texture and composition. To obtain a complete set of information, the samples were examined using various instrumental methods and techniques. The first step was chemical analysis involving attack with diluted hot hydrochloric acid to determine the amount of residues insoluble in hydrochloric acid [11, 12].

For sieve analysis, the samples were initially crushed in an agate mortar, weighed and dried to a constant mass at 105°C. Then, they were digested for two minutes with hydrochloric acid. After washing and obtaining neutral pH, they were dried at 60°C to

Fig. 2. A) Stone part of the cylindrical tower in the first floor of the south wing of the Lublin Museum building, B) Stone arrangement in the face of the cylindrical tower visible from the first floor of the south wing of the Lublin Museum building

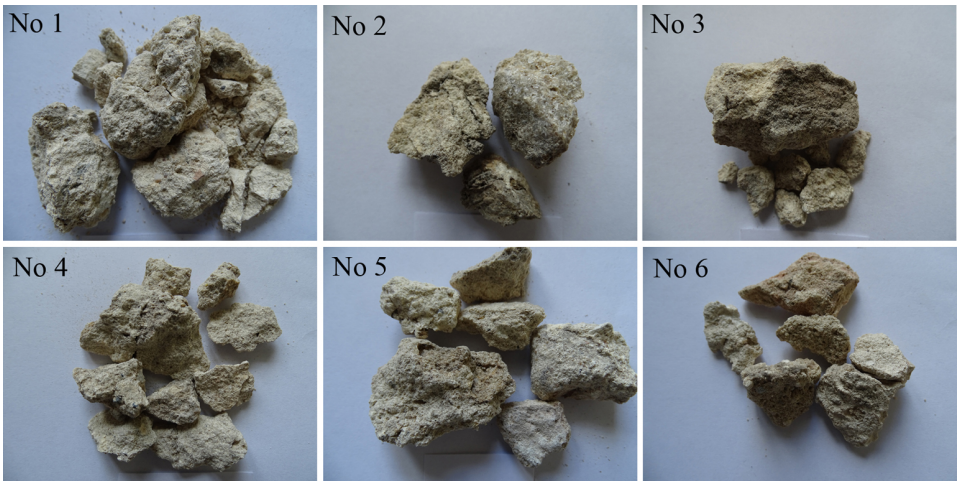

Fig. 3. Mortar samples of the tower inside the first floor of the south wing of the Lublin Museum building

a constant weight. The samples were weighed and sieved on analytical sieves (< 0.020, 0.020 0.050, 0.100, 0.250, 0.500, 1.000, > 2.000 mm); the percentages of each fraction were calculated.

Prior to X-ray diffraction (XRD) analyses, the samples were dried at 60°C; then they were crushed and pulverized in an agate mill. XRD allows detection of the crystalline phases present in mortars when their concentration is sufficiently high, usually below 3–5% [11, 13–15].

X-ray diffraction was performed using a Philips X'PRO type PW 3040/60 diffractometer, which was equipped with a copper anticathode X-ray tube. The operating conditions were as follows: step – 0.01, count time – 1.2 s, measuring range 5°–76°, 2Theta.

Scanning electron analysis is used to evaluate particle shape and size, morphology and interrelationships between mortar components, the presence of inclusions, cracks, void filling and pore shape. A dispersive (X-ray) spectrometer (EDS), combined with a scanning electron microscope, allows conducting rapid analyses of elemental composition. The representativeness of the sample is crucial for interpreting the microstructure observed on a very small scale [16].

Scanning electron microscopy (SEM) observations and energy-dispersive X-ray microanalysis with electron microprobe (SEM-EDS) were carried with non-sputtered cross sections. The determination was performed using a LINK-ISIS electron microprobe coupled to a JSM-6300 electron microscope from JEOL (accelerating voltage: 20 kV beam current: 10–9 A, duration of analyses: spot – 100 s, micro-area – 15 min). All analyses were carried out in the laboratory of Faculty of Civil Engineering and Architecture of Lublin University of Technology.

3. Test results

3.1. Determination of residues insoluble in hydrochloric acid

As indicated via macroscopic analysis, all mortars show some similarity in terms of binder/aggregate ratio and particle size distribution. All samples have relatively high percentages of insoluble residues, ranging from 63.8% to 67.7% by weight, following dissolution with hydrochloric acid (Table 1).

Table 1. Ratio of insoluble residues and their granulometric distribution

Sample	Binder: aggregate ratio from HCl dissolution test [weight]	> 2.00 mm	1.00 mm	0.50 mm	0.25 mm	0.10 mm	0.05 mm	0.02 mm	< 0.02 mm
No. 1	1:2.6	0.51	11.96	50.78	29.53	4.51	1.59	0.75	0.00
No. 2	1:2.6	0.53	12.85	49.54	31.22	4.68	0.52	0.21	0.00
No. 3	1:2.2	0.00	1.12	14.04	43.35	40.15	0.71	0.26	0.02
No. 4	1:2.6	0.90	8.94	47.54	31.70	7.24	0.55	0.26	0.00
No. 5	1:2.2	0.39	5.22	51.99	31.88	6.89	1.75	0.57	0.05
No. 6	1:2.4	0.90	4.29	17.76	39.63	34.97	0.51	0.47	0.02

The mortar samples subjected to grain size analysis show that filler mainly comprises sand fraction (grain size 2–0.05 mm). The grains with a grain size of 0.50–0.25 mm dominate in samples No. 1, No. 2, No. 4, No. 5 and account for about 80% of the weight of the samples. On the other hand, aggregate sizes of 0.25–0.10 mm predominate in samples

No. 3 and No. 6, accounting for 75–83% of their weight. The samples contain no dust fraction (grains less than 0.05 mm) or negligible amounts there of (Table 1) and (Fig. 4).

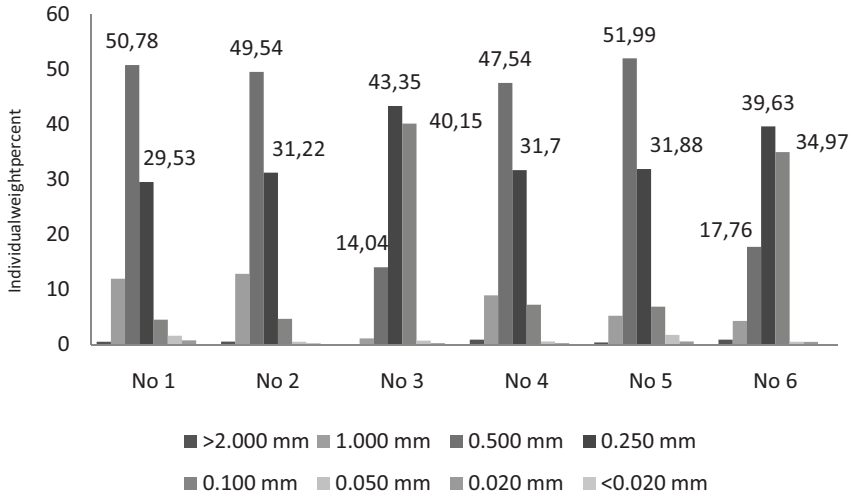

Fig. 4. Grain size distribution of analyzed samples

The X-ray diffraction technique was used to determine the mineralogical composition of the binder and filler, i.e. mineralogical components or crystalline phases. The X-ray diffraction patterns of the analyzed samples reveal that the mortars are mineralogically homogeneous (Fig. 5). Quartz is the ubiquitous, dominant phase, present in all samples and constitutes the filler grains. Calcite is another important component of the mortars and is a component of the binder. Subordinate components of mortars are minerals of the feldspar and silicate groups. These are mainly represented by microcline, as well as the less common albite, muscovite and clinochlorite. These components, like quartz, constitute the filler grains (Table 2).

Table 2. Summary of the results of XRD analysis of the tested samples

Sample	Cal	Qtz	Mc	Ab	Kn	Ms
No. 1	++	+++	t	t	t	+
No. 2	++	+++	t	t	t	+
No. 3	++	+++	+	+	t	+
No. 4	++	+++	t	t	t	+
No. 5	++	+++	t	+	–	+
No. 6	++	+++	t	t	t	+

Cal: calcite; Qtz: quartz; Mc: microcline; Ab: albite; Kn: kaolinite; Ms: muscovite
+++ : dominant; ++: present; +: small amounts; t: traces; –: not detected

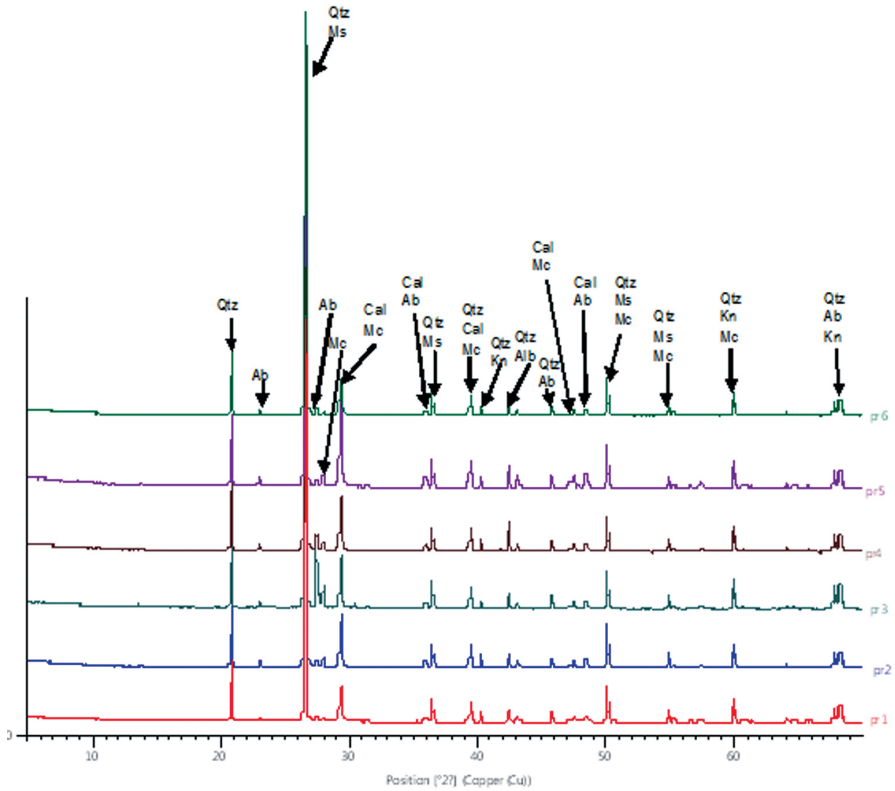

Fig. 5. Representative XRD patterns. Cal: calcite; Qtz: quartz; Mc: microcline; Ab: albite; Kn: kaolinite; Ms: muscovite

3.2. Scanning microscope (SEM) and Energy-dispersive X-ray spectroscopy (EDS) analyses

SEM analysis provided valuable information about the components of the mortar: it determined the relationship between the binder and aggregate, and enabled to observe their forms, sizes, textures and distribution in the mortar.

SEM photomicrographs were recorded at magnifications of 1000, 2000, 5000 and are shown below (Fig. 6A–6D).

The tested mortars consist of a binder, which is in the form of microcrystalline calcite (micrite), heavily penetrated by fungal colonies.

The results of scanning microscope (SEM) observations with an X-ray microanalysis (EDS) attachment show that the mortars tested contain a lime binder with filler. All mortars have a compact microstructure, typical of lime mortar, with aggregates well-embedded in the mass. Using an X-ray attachment, mainly calcite (Fig. 7A), well-bedded quartz (Fig. 7C), and finer grains of aluminosilicates (Fig. 7B) were identified.

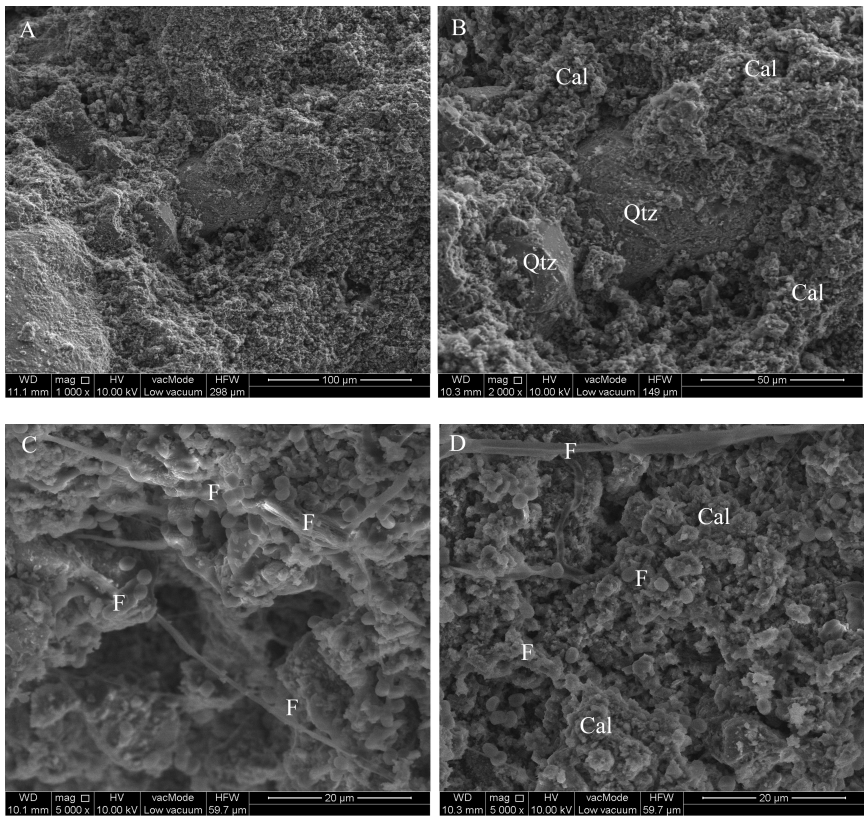

Fig. 6. SEM images of mortar samples. A), B) binder composed of micritic calcite (Cal), with quartz grains (Qtz) in the center. C), D) mortar binder heavily penetrated by fungal colonies (F). Magnification: ×1000, over ×2000, over ×5000

As shown by scanning tests, the mortar binder is made up of micrite occurring as isomeric grains of 0.5–1.0 μm in size. They form a kind of crystalline grout. The results of chemical composition tests obtained with a microprobe (EDS) showed that they are accompanied by small amounts of silica (SiO_2), as well as aluminum, sodium, potassium and phosphorus (Fig. 8).

The presence of admixtures may be due to two reasons, the first, probably the primary reason, was the type of raw material used to make lime, the second may have been clay admixtures from the lime pit.

The material from lime (CaO) was fired was probably chalky or marly limestone mined in the Lublin area, which is easy to mine and available nearby. These limestones are characterized by the presence of opal silica ($SiO_2 \cdot nH_2O$) and chalcedony, hence the small but constant admixture of these components within the carbonates contained in the mortars. In addition, these same limestones contained clay; thus, the elevated aluminum and potassium content. The amounts of other admixtures are also typical of these rocks.

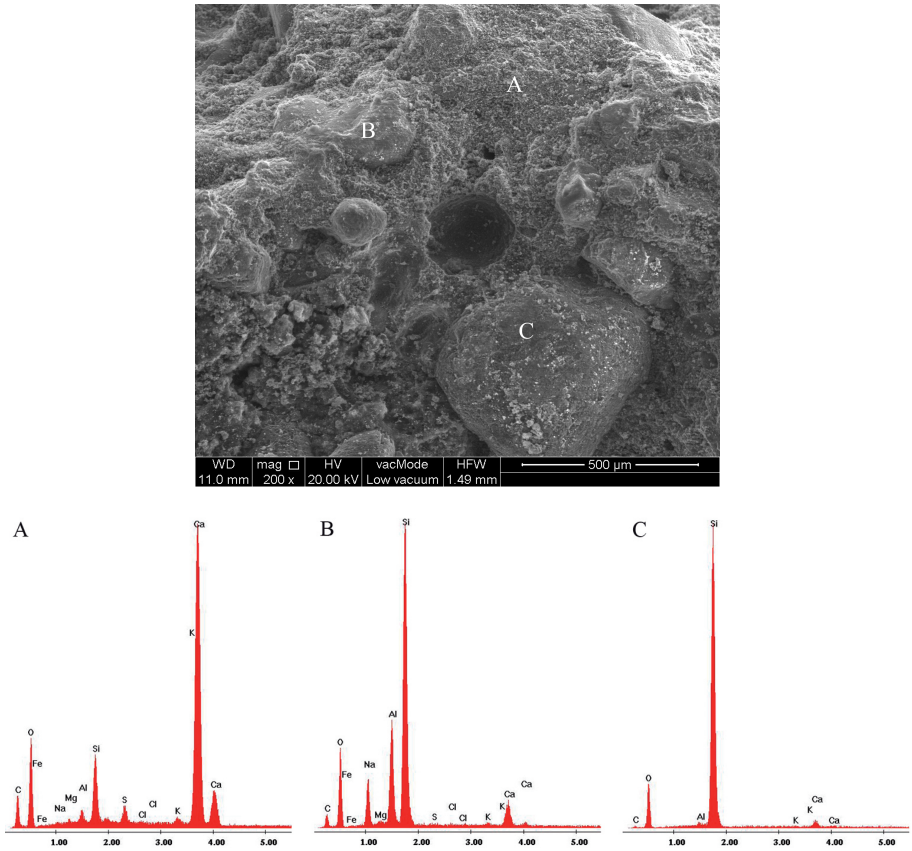

Fig. 7. SEM image of sample NR 2 and EDS spectra taken from points corresponding to: (A) – binder: calcite, (B) – aggregate: aluminosilicate, (C) – aggregate: quartz. Magnification: ×200

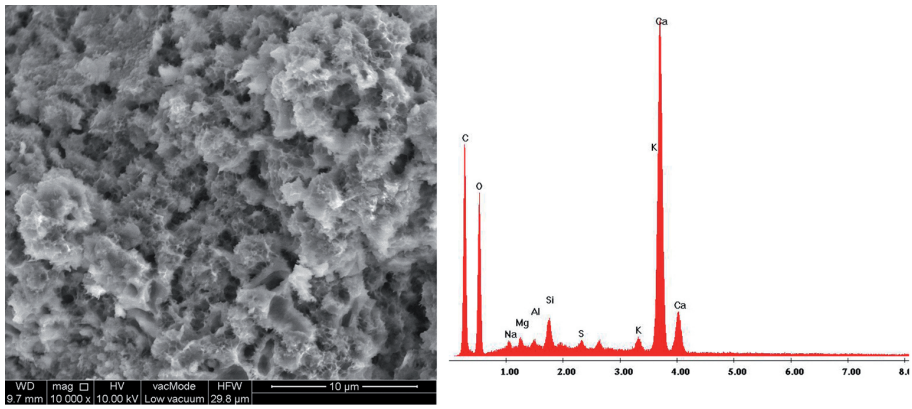

Fig. 8. The mortar binder is made up of micritic calcite. SEM-EDS analysis, magnification: over ×10, 000

4. Discussion

On the basis of the tests performed and the analysis of the observed microscopic features of the mortars from the basalt, the differences between the samples relate only to the size of the aggregate grains.

The aggregate composition was dominated by quartz grains by volume, mainly in the form of well-rounded grains. The size of quartz grains ranged from 0.10 to 0.50 mm, while in terms of volume, the dominant size was 0.25 mm to 0.50 mm. Aluminosilicates were somewhat semi-rounded, their size ranged from 0.25 mm to 0.10 mm. The variation in the nature and morphology of the aggregate grains indicated that the material came from the weathering of locally occurring metamorphic rocks in the bedrock. This was indicated by their similar petrographic composition and slight heaving resulting from possible short-distance transport. Such variability would indicate some variation in mortar preparation technology, resulting from greater or lesser involvement of the construction workshop in the proper preparation of mortars. The aggregate used as mortar filler was thoroughly sieved.

The binder was calcium hydroxide, observed as calcium carbonate as a result of carbonation homogeneous. This may have been due to the good firing of the carbonate rock, but also good homogenization with the filler, and may have been related to the relatively long seasoning of the lime. The lime used as a binder for the mixture was fired at the right temperature and slaked for a long time, as it contained very small amounts of micrite clusters.

5. Conclusions

Testing of the materials taken from historic buildings is of paramount importance for future restoration. It determines the need to ensure full compatibility between the original material and the restoration mortar. The results of mineralogical testing of mortars lead to the conclusion that all mortars have similar manufacturing technology, evidenced by comparable silica aggregate showing related bimodal grain size and the same type of binder.

Considering the usefulness of mortar analysis for historical research, it should be noted that it is primarily an attempt to objectify the accuracy of the researcher's observations, which are usually made traditionally, i.e. with an eye, rather than a microscope. Contrary to the belief that all lime mortars are the same, it enables to see some ordering of samples in terms of:

1. the volume ratios of the main components, namely the binder and composition of grains;
2. the distribution of aggregate grains, which indicates the fact of sieving or non- sieving of the added sand;
3. the low or high frequency of micrite clusters in the binder, which in turn indicates the quality of the lime firing and curing process;
4. the variation of the group of accessory components.

If the research is extended to other sites, answering points 2 and 3 above will enable to isolate and identify the construction workshops operating in Lublin Castle in the second half of the 13th and first half of the 14th century; however, it is still too early to do so.

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Charakterystyka XIII-wiecznych zapraw z baszty na Zamku Lubelskim

Słowa kluczowe: historical mortars from the 13th century, research, Lublin castle tower

Streszczenie:

Baszta na Zamku Lubelskim, zwana donżonem, to jedyny po wschodniej stronie Wisły zabytek sztuki romańskiej. Cylindryczna, murowana budowla wchodzi w skład kompleksu Zamku Lubelskiego. W Basztę wzniesiono około połowy XIII w. w formie cylindrycznej, jednorodnej budowli wykazującej cechy stylu późnoromańskiego [1, 2]. Budynek został wzniesiony w typowy sposób dla XIII-wiecznych warowni – wewnątrz pierścienia obronnego, jako główne centrum umocnień (Fig. 1A). Od tamtego czasu baszta nie była przebudowywana na większą w skalę. Baszta to cylindryczna wieża na planie koła. Stoi na dziedzińcu zamku, częściowo wtopiona w jego południowe skrzydło połową swego obwodu. Posiada trzy kondygnacje. Zwieńczona jest krenelażem umieszczonym na lunetowym fryzie. Dolne partie wieży zostały zbudowane z wapienia łamanego i cegły, górne z cegły.

W trakcie współcześnie wykonywanych prac restauratorskich odkryto XIII-wieczne mury, co umożliwiło pobranie oryginalnych materiałów. W artkule przedstawiono charakterystykę mineralogiczną, chemiczną i granulometryczną. Wieża na Wzgórzu Zamkowym w Lublinie jest jedynym znanym zabytkiem późnego okresu romańskiego na terenie historycznego województwa lubelskiego. Znaczenie tego obiektu dla dziejów zamku i miasta nie przełożyło się jednak proporcjonalnie na zakres podejmowanych badań. W istocie bowiem nie dokonano, jak dotychczas, planowych i systematycznych badań architektoniczno-konserwatorskich programowo mających na celu wyjaśnienie podstawowych kwestii związanych z obiektem: warsztatem i zastosowanym materiałem budowlanym.

Badania i charakterystyka zabytkowych zapraw jest istotnym narzędziem identyfikacji pochodzenia składników, służy również do pozyskiwania informacji o historycznej ewolucji zabytku [4–7]. Właściwości techniczne zapraw są zależne od surowców [8]. Skład zapraw (charakter składników) i tekstura (zależna od składu masy i rozkładu uziarnienia są to najbardziej istotne cechy w kwestiach kompatybilności technicznej, pomiędzy materiałem historycznym, a nowo projektowanym [9].

Produkcja zapraw jest złożonym procesem, obejmującym znajomość materiałów i technologii [4]. Dlatego aby zdobyć niezbędne informacje o historycznych zaprawach wykonujemy badania, w których mają zastosowanie metody chemiczne, mineralogiczne, petrograficzne [5, 6].

Makroskopowo wszystkie zaprawy wykazują pewne podobieństwo pod względem stosunku spoiwo/kruszywo oraz pod względem rozkładu wielkości cząstek. Wszystkie próbki charakteryzują się stosunkowo wysokimi odsetkami nierozpuszczalnych pozostałości, w zakresie od 63.8–67.7% wag., po rozpuszczaniu kwasem solnym (Tabela 1).

Próbki zapraw poddane analizie uziarnienia zawierają jako wypełniacz, głównie frakcję piaskową (wielkość ziaren 2–0,05 mm). Ziarna o wielkości 0,50–0,25 mm dominują w próbkach Nr P 1, P 2, P 4, P 5 i stanowią około 80% masy próbek. Natomiast w próbkach Nr P 3 i P 6 przeważa kruszywo o rozmiarach 0,25–0,10 mm, co stanowi 75–83% ich masy. Próbki nie zawierają frakcji pylastej (ziaren poniżej 0,05 mm) lub zawierają jej znikome ilości (Tabela 1).

Do określenia składu mineralogicznego spoiwa i wypełniacza – składników mineralogicznych lub faz krystalicznych, zastosowano technikę dyfrakcji rentgenowskiej. Rentgenowskie wzorce dyfrakcji analizowanych próbek ujawniają, że zaprawy są jednorodne mineralogicznie (Fig. 4). Kwarc

jest fazą wszechobecną, dominującą, występuje we wszystkich próbkach i reprezentuje ziarna wypełniacza. Kalcyt jest kolejnym ważnym składnikiem zapraw i stanowi składnik spoiwa. Podrzędnymi komponentami zapraw są minerały z grupy skaleniowej i krzemianowej.

Wyniki obserwacji w mikroskopie skaningowym (SEM) z przystawką do mikroanalizy rentgenowskiej (EDS) pozwalają stwierdzić, że zaprawy poddane badaniom zawierają spoiwo wapienne z wypełniaczem. Wszystkie zaprawy mają zwartą mikrostrukturę, typową dla zaprawy wapiennej, z dobrze osadzonymi kruszywami w masie. Stosując przystawkę do analizy rentgenowskiej zidentyfikowano głównie z kalcyt (Fig. 6A) i kwarc o ziarnach dobrze obtoczonych (Fig. 6C) i drobniejsze ziarna glinokrzemianów (Fig. 6B).

Na podstawie wykonanych badań i analiz obserwowanych cech mikroskopowych zapraw z baszty, różnice pomiędzy próbkami dotyczą jedynie wielkości okruchów kruszywa.

W przypadku wszystkich zapraw, w składzie kruszywa objętościowo dominowały ziarna kwarcu, głównie w postaci dobrze obtoczonych ziaren. Wielkość ziaren kwarcu wacha się od 0,10 mm do 0,50 mm, natomiast pod względem objętości dominowała wielkość 0,25 mm do 0,50 mm. Glinokrzemiany nieco półobtoczone ich wielkość wacha się od 0,25 mm do 0,10 mm

We wszystkich próbkach spoiwo miało charakter mikrytowy (węglanowy) było jednorodne. Mogło to wynikać z dobrego wypału skały węglanowej, ale także z dobrej homogenizacji z wypełniaczem, być może też wiązało się z relatywnie długotrwałym sezonowaniem wapna. Zróżnicowanie w charakterze i morfologii ziaren kruszywa świadczyło o tym, że materiał pochodził ze zwietrzelin lokalnie występujących w podłożu skał metamorficznych. Wskazywał na to zbliżony do nich skład petrograficzny oraz nieznaczne obtoczenie wynikające z ewentualnego transportu na niewielkie odległości. Taka zmienność wskazywałaby na pewne zróżnicowanie w technologii przygotowywania zapraw, wynikające z większego lub mniejszego zaangażowania warsztatu budowlanego w odpowiednie ich przygotowywanie.. Kruszywo stanowiące wypełniacz zaprawy zostało dokładnie przesiane. Także wapno użyte jako spoiwo mieszanki wypalono w odpowiedniej temperaturze i długo lasowano, zawiera ono bowiem bardzo niewielkie ilości skupin mikrytowych.

Badanie materiałów pobranych z zabytkowych budynków mają pierwszorzędne znaczenie dla przyszłej renowacji. Określa konieczność zapewnienia pełnej kompatybilności między oryginalnym materiałem i zaprawą renowacyjną. Wyniki badań mineralogicznych zapraw prowadzą do wniosku, że wszystkie zaprawy mają podobną technologie wykonania, świadczy o tym porównywalne kruszywo krzemionkowe wykazujące pokrewną bimodalną wielkości ziarna i taki sam typ spoiwa.

Rozszerzenie badań zapraw na inne obiekty znajdujące się na wzgórzu zamkowym, pozwoli prawdopodobnie wyodrębnić cechy charakterystyczne zapraw stosowane przez warsztaty budowlane działające na zamku Lubelskim w drugiej połowie XIII i pierwszej połowie XIV wieku.

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