**5_1_5_1_Building_material_analyses**

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Introduction

In this section are presented the results of the investigation of the contents, structures and properties of the building materials used on the Trypillia mega-site of Nebelivka and the changes these materials have undergone due to chemical, physical and mechanical activities. The approach can be summarised as a systematic typological analysis, using classifications rooted in geology and chemistry and based upon the technical-technological traits of the building materials. These traits provide proxy information on technological processes for different products (e.g., pottery-making, house-building). However, it should be underlined that taphonomic changes, whether depositional or post-depositional, may mask the proxy information we are searching for on technology. It has therefore been decided that simple classifications would be most likely to adhere to the general principles and produce satisfactory results. We cannot escape the fact that we often deal with only partial information. Compensation for this problem lies in the realms of inter-disciplinary research, in which geo-archaeology and archaeo-mineralogy are the most significant elements.

The building of houses with timber, clay and reeds has been at the centre of Trypillia archaeology since the earliest excavations (Hvojko 19xx; for summary, see Videiko 2016). However, it is surprising to note that no results of *ploshchadka* research after 100 years so far have focussed on physical-chemical analyses of the building materials, presumably because of poor preservation thanks to the effects of burning and ploughing. Thus, no 'original' template has been preserved from which to derive past practices. It is accepted that house-models are a problematic form of evidence that may not replicate original house forms and building techniques.

The burning of a Trypillia house preserves the burnt daub mass (the '*ploshchadka*') in plan form but has destroyed much, often leaving only shapeless daub remains. However, close attention to this building material allows the recovery of much useful technological information. We should also be aware that house spaces were not only 'functional' areas for living and shelter but also reflected social, economic, ritual and other practices. Two long-running debates for Trypillia houses concern the question of one- or two-storey houses and the use of fire as constructive as well as destructive elements (for discussion and an experimental approach, see below, Section 6).

The issues not so far discussed in the literature on Trypillia houses include: (1) the absence of a basic catalogue of building materials; (2) the absence of a technical-typological classification; (3) the consequence that current architectural typologies are not based upon any technical - typological traits; (4) the absence of any analysis of house-building technologies in the round; (5) the failure of the construction of local groups based upon architectural styles consequent upon issues (2) - (4); and (6) the absence of analysis of the raw materials for building materials, which prevents resource / economic analysis, the analysis of labour investment and related social questions.

The reasons for house-burning are multiple and should be separately investigated, in part through emphasis on taphonomy (e.g., the surface deposits on building materials and pottery). It is important to recall that the nature of the clay affects the secondary changes to these materials during house-burning.

For these reasons, this analysis begins with the most important task - the creation of a technical-typological analysis of an important Trypillia structure - namely the mega-structure excavated at Nebelivka in 2012. This analysis starts with a systematic classification of technical - typolological traits. The second part of the analysis is devoted to an investigation of the ways that heat impacted on the different clays used in the construction of the mega-structure, including a suite of methods used to determine the temperature to which daub was fired during the burning of the mega-structure.

## Clay materials used in building

Five different clays were used at Nebelivka (Photo 1). During preliminary fieldwork in the village territory, the project team was able to identify four sources of clay. This indicated that the distances required to bring clay to the mega-site rarely exceeded 2km.

The five clay sources could be distinguished initially by colour and, later, by physical-chemical analysis. The colour of the greenish clays was due to ferrous oxide ($Fe_20$), while red clays have a strong component of ferric oxide ($Fe_20_3$). Colour variations in the clays is due to the ratio of ferrous oxide to ferric oxide. Other impurities affecting the colour of clays include manganese (Mn), magnesium (Mg), chromium (Cr), phosphorus (Ph) and calcium. But even with the presence of such impurities, it is the iron elements that were predominant in determining firing temperatures. At temperatures of $700^0$C in an oxidising atmosphere, $Fe_20$ is oxidised to $Fe_2O_3$, with consequent colour changes (e.g., the range 'greenish-brown - pale yellow' changes to the range 'bright orange - red - dark reddish-brown') without visible changes towards vitrification. These colours show the temperature range of $700^0$C - $900^0$C.

## 4.6.3 Methods and materials

Preliminary work on the building materials discovered during the excavation of House A9 in 2009 (see Section 5.2.1) allowed the establishment of the full range of building materials and pottery. The site visit to the excavations of the mega-structure in 2012 led to the creation of a sampling strategy for building materials, with archaeological advice and eventual archaeological classification of the different construction elements in the mega-structure. The samples of building materials collected from the 2012 excavation enabled a preliminary classification of this body of material.

Laboratory methods were designed to fulfil three goals: (1) the establishment of the technical - typological characteristics of the samples, including their physical-chemical properties, their composition, their structure, their context and their recipes; (2) the determination of the function of the different building materials; and (3) the classification of building materials based on Goal (1).

The sampling strategy took 12 factors into account: (1) impressions on the base of the building material; (2) mechanical surface treatment of the building material; (3) texture of the face of the building material and the nature of its fracture; (4) the macro-texture of the building material (layers, unevenness); (5) porosity of the building material; (6) material composition of the building material; (7) origins and relation to known sources; (8) structure and stratigraphy of the building material; (9) ratio of elements of clay mixture; (10) orientation of the building material structure; (11) colour; and (12) hardness, density and strength of the building material.

A combination of macro- and micro-level methods was used, including morphological analyses, qualitative analyses (composition), quantitative analyses (ratios between different elements in clay mixture), metric analysis (fractions of content, thickness of layers of building material) and textural-structural analyses. The specific methods utilised are listed below (Table 1). Technical analyses usually related to contemporary standing buildings were not completed (resistance, plasticity). These traits were registered but not analysed further.

Table 1 Analytical methods utilised

| METHOD | FIELD OF INFORMATION |
|---|---|
| Technical-typological analysis | clay recipes, methods of preparation and function |
| Optical microscopy | ratios between elements in the clay mixture, especially with crystalline ingredients |
| Optical microscopy | Determination of fine temper and clay minerals |
| Stereoscopic and optical microscopy | mineral temper, ratio of elements in mixture and structure of fine-grained vitrified materials (preliminary results) |
| stereo-microscopy (direct and side-light) | Clay morphology |
| polarising, non-reflective stereo-microscopy | Definition of elements in the clay mix |
| Micro-chemical analysis | Determination of ionic content and acidity |
| Chemical analysis | added precision to the determination of the clay composition |
| XRF analysis | Determination of the clay elements and crstalline structure |
| electro-micro-probe analysis | Determination of the clay elements |
| Stratigraphic analysis | Determination of the number of layers and the order |

| | |
|---|---|
| | in which they were laid down in the clay, their thickness and degree of adhesion |
| Thermo-chemical analysis | Definition of the range of firing temperatures of the clay |
| Colorimetric analysis | Definition of the range of firing temperatures of the clay |
| Analysis of sintering | Determination of the porosity of the clay |
| Quantitative thin-section analysis | Evaluation of porosity |

A total of 155 samples were collected, comprising building material (n = 91 samples), pottery (n = 22 samples) and grinding stones and unworked stone (n = 15 samples) in accordance with the Ukrainian excavation grid and the mega-structure contexts, as well as materials from clay sources (n = 16 samples) and rock sources (n = 11 samples) (Section 5_1_5_3). A total of 89 photographs was taken of these samples (Section 5_1_5_2).

Samples were manually cleaned before analysis (before cleaning: Photos 2 - 4; before, during and after cleaning: Photo 5; during cleaning: Photo 6; after cleaning: Photo 7).

A total of the 80 most characteristic samples was selected for further laboratory analysis. Visual inspection by magnifying glass, supplemented by stereo-microscopic investigation, confirmed the preliminary field classifications of morphology, mineral content and stratigraphic structure. Thin-sections were made for polarising microscopy to recover more specific information on mineral contents, structural traits, ratios of elements in the clay mixture and mineral temper. Further, micro-probe analysis was used for the clay minerals in some of the samples. Polished sections (Russian: *Anschliff*) were studied in reflected side light to gain further stratigraphic information.

## 4.6.4 Results

### Macro-textural investigation of impressions

Impressions of the following materials were found on daub samples: 1) wood; (2) wood shavings; (3) straw; (4) chaff; (5) grain; (6) steppe grasses (e.g., *Stipa*); (7) woven stems (? twine); and (8) leaves. There were no obvious examples of reed impressions. The commonest impressions on the underside of daub were regularly-cut, flat wooden beams with fibres (Photos 8 - 11). There were fewer samples with smooth impressions of thin, circular rods of diameter 7 - 10cm (perhaps hazel withies), probably because of the filing of timber or the process of de-barking (Photos 12 - 13). Even more rare are impressions of branches of diameter 3 - 5cm (Photos 14 - 15). Some multi-layered impressions were found, some with finger impressions producing 'barbotine'-like roughening, especially on examples of highly plastic clays) (Photos 16 - 17). Cereal grain, chaff and straw impressions were very common throughout the daub samples (Photos 18 - 19).

Petrographic investigations of building materials using SEM and electro-probe

Reflected-side light and penetrating polarised light were used at various levels of magnification (x20, x100, x200 and x600). A thin-section and polished section (*Anschliff*) were made for each of the 10 samples analysed.

*Context 29 (Sample 1b)*: decorated daub from podium: silty polymineral, weakly carbonised clay with >50% chaff. Surface slip applied in 2 layers - lower not compacted, upper (8mm thick) compacted through careful smoothing (cf. 1mm-thick slip for red-ochre painting; different clay = light, pale yellow marl clay, more acidic). Partially isotropic matrix, and colour show firing temperature of above $800^0$C (Photos 20 - 21)

*Context 29 (Sample 54)*: the clay was a silty, poly-mineral carbonate marl from the podium. It lacked a sand fraction and a natural calcitic temper. No firing temperature was determined (Photos 22 - 23). The polished section showed a polymineral clay, with mostly calcite and some quartz-feldspar, olivrite and mica minerals (Photos 24 - 25).

*Context 257 (Sample 76):* lower layer of platform. The clay was a silty, poly-mineral carbonate marl, lacking a natural calcitic temper; an anisotropic clay matrix, with partial optical preservation of the clay minerals, indicating that the daub was burnt at not more than 800 - $850^o$C.

*Context 114 (Sample 80)*: daub scatter. The daub showed many pores, with an anisotropic matrix and colour showing firing temperature of 200 - $300^0$C. The clay was a silty, polymineral, weakly carbonate clay + >50% chaff. (Photos 26 - 27).

*Context 112 (Sample 36a)*: South wall near the podium. Red daub in multi-layered daub (red-white-pink). Silty polymineral clay with natural temper of quartz-feldspar and ferrous materials, + >60% chaff. Lighter clay found only at junction with white clay. The colour shows a firing temperature of $>900^0$C (Photos 28 - 29).

*Context 112 (Sample 36b:* South wall near the podium. White daub in multi-layered daub (red-white-pink). Clay carbonate marl with lot of calcium carbonate and quartz-feldspar and little ferrous inclusions; chaff up to 60%. Anisotropic matrix and colour showing firing temperature of $850^0$C. (Photos 30 - 31).

*Context 112 (Sample 36c)*: South wall near the podium. Pink daub in multi-layered daub (red-white-pink). No firing temperature was ascertained (Photos 32 - 33).

*Context 112 (Sample 36d)*: South wall near the podium. Daub made by poor mixing of two polymineral clays with chaff - a light, marl clay with ferrous minerals and a greasier clay with a high ferrous content. Clay is isotropic and start of vitrification shows firing temperature of >1000$^0$C (Photos 34 - 35).

*Context 118 (Sample 119)*: part of the South East wall. Careful mixing of two clays and water - a light carbonate marl with ferrous particles and a greasier clay with low ferrous content. Anisotropic nature of clay means a firing temperature not exceeding 800$^0$C (Photos 36 - 37).

*Context 149 (Sample 37)*: 'pithos' above the platform Context 29. The clay matrix is anisotropic, typical for temperatures that did not exceed 800 - 850$^0$C. Shaped in two levels of clay - a lower level of olivritic clay marl with 50% chaff (which dries faster); an upper level consisting of a mixture of two clays - a polymineral clay and a marly clay with 30% chaff (to reduce shrinkage / cracking during drying). A slip was applied to smooth the outside surface, perhaps to achieve a decorative effect (Photos 38 - 39).

The results of these analyses were confirmed by the X-ray electro-probe investigation of 36 plasters.

Daub firing temperatures

The basic outline of the interaction of clay with heat shows that, when dried at 20$^0$C - 90$^0$C, clay loses water from its pores to become more durable but this is reversible below 90$^0$C. Above 100$^0$C, capillary water is irreversibly lost and the clay becomes a friable ceramic with the original colour and texture (200$^0$C - 400$^0$C). At temperatures of 400$^0$C - 800$^0$C, the ceramic loses crystallization water and. at the higher temperature, there is some sintering with increased strength, changes in colour and preserved granularity. The colour of poly-mineral clays (pale yellow, brownish, greenish-brown) exhibit more complex changes.

Some samples contained unburnt carbonaceous particles, showing pale yellow to dark grey and pink shades; this showed a low firing temperature of c. 200$^0$C achieved with a weak draught (e.g., Photos 40 - 41). The low sintering is shown by these samples being very crumbly (Photo 42 with no organic content).

The majority of daub samples underwent strong colour changes (fawn or light brown to red, pink or reddish-brown; light-coloured clays to yellow or pinkish-yellow colours), whether the daub contained organic temper or not (the organic temper burnt out at temperatures exceeding 300$^0$C) (Photos 43 - 44).

Vitrified daub showed a surface with swollen distortions with loss of original material (Photo 45). Differing kinds of vitrification depended on the clay composition and its refractory properties; these differences can be seen in the same sample! (e.g., light clay can be vitrified while red clays maintained their granularity) (Photo 46).

Three groups of daub were selected for more detailed analysis: (1) daubs with weak transformations; (2) daubs with strong transformations; and (3) vitrified daubs. Group (1) samples were fired in a muffle kiln to act as a reference collection for Groups (2) & (3), using XRD methods (reflected light microscopy). Light-coloured clays were excluded from this experiment because their colour changes were not diagnostic. Group (1) consisted of Samples 27a and b (Context 29 - pithos), 56a and b (? Context) & 61 (Context 49). The original colour was partly maintained and unburnt organic particles survived, indicating a low temperature and a weak draught; the samples were brittle, with low sintering. Group (2) consisted of Samples 2 (Context 29) and 29 (Context 107). Despite radical changes in the samples, some structural features were still maintained; this indicates a very high firing temperature between $700^0$C and $900^0$C. Group (3) consisted of Samples 6 (Context 107) and 8 (Context 146). Complete changes had occurred from the initial characteristics of the clays at temperatures exceeding $1000^0$C.

Thermal charts are presented for each Group (1) sample in JPEG forming two variants: Variant 1 - short-term firing with a uniform increase by $100^0$C every hour: temperature changes every $100^0$C (Fig. 47); Variant 2 - long-term firing in a stable temperature regime, with five 8-hour firings: temperature changes every $200^0$C (Figs. 48 - 49).

### 4.6.5 Discussion and conclusions

The conclusions made on the basis of the SEM investigations were as follows:
(1) building materials were often made by mixing different clays, sometimes forming lumpy mixtures of uneven colour and many cracks and pores (uneven shrinking with drying);
(2) by contrast, high-quality mixtures lead to uniform surface colours and consistent textures, with an even distribution of cracks;
(3) three types of mixture were observed: (a) chaff-free clay made of a mixture of two clays (Podium, Platform); (b) red clay or marl with chaff, turning red or yellow after firing (smoothing layers); (c) a mixture of two clays and chaff, turning pink after firing (smoothing layer).
(4) daubs contained natural minerals, including (a) clastic quartz-feldspar; (b) calcium carbonate, making the clay more plastic; and (c) ferrous minerals;
(5) the main artificial temper was chaff (40 - 60%);
(6) the different size of porosities in the daub can be classified as (a) burnt-out chaff ; (b) gaps between minerals formed in drying; and (c) expansion of minerals through high firing and subsequent cooling. These types can be found in one daub sample;
(7) the striking absence of sand-grade temper (possibly removed in clay purification);
(8) no chaff was used in the application of clay to horizontal surfaces (e.g., Platforms). These were dense clays - stronger than chaff-tempered daub;
(9) the surface smoothing layer was usually applied in several coats, with chaff temper in the lower part, but with different clays used in different cases;
(10) a light, carbonate-rich clay layer, free of chaff, was applied in a 1-mm-thick layer as a basis for painted decoration;

(11) red paint was made from red ochre;
(12) the clay materials of the podium and the lower level of platform 257 were the same;
(13) different clay materials were used to construct the different layers of Platform 257, suggesting either different times of making or different functions; and
(14) the firing temperatures of most daub was found to be no more than 800 - 850$^0$C, with one very low temperature (200 - 300$^0$C, Sample 80) and one vitrified sample, fired at over 1000$^0$C (Sample 36d).

The conclusions made from the daub temperature analyses were as follows:
(15) all samples showed similar tendencies in colour change based upon firing temperature increases.
(16) Very different firing temperatures were reached in different parts of the mega-structure. These temperatures can be grouped into low (200 - 400$^0$C), medium (400 - 900$^0$C) and high (>900$^0$C). Concentrations of vitrified daub ('high') may indicate the starting-point for the fires; concentrations of unburnt daub ('low') may have been the most remote from the fire or were 'protected' under collapsed walls and/or as lowest parts of the design (e.g., the podium, the Platforms).
(17) In the case of four contexts (the platform Context 6; the podium Context 29); and two daub scatters Contexts 107 and 112), contrasting daub temperatures were recorded for different samples from the same context. The higher (highest) of the temperatures is taken to represent the principal impact of the fire, while the lower (lowest) indicates areas protected from the main conflagration.
(18) 'High' temperatures were found in only three Contexts (the platform Context 6 and the daub scatters Contexts 107 and 112). 'Medium' temperatures were found in four contexts (the podium Context 29; the platform Context 257; the West wall Context 118 and the pithos (Context 149) above platform Context 29). Finally, 'low' temperatures were found in five contexts (the fired clay wall slots Context 159; the North wall Context 173; and daub scatters Contexts 45, 110 and 120).

Another way to interpret the daub firing temperature evidence is a consideration of its spatial distribution. The plotting of all contexts with examples of vitrified daub in Phase 3 Lower, 3 Upper and 3 combined shows markedly different distributions in the two phases. In Phase 3 Upper, the principal clustering of vitrified daub was in the South-West corner, with a spread along the podium and the South wall; there were hardly any clusters of vitrified daub in the Eastern rooms. However, in Phase 3 Lower, much more vitrified daub was found in the Eastern rooms, with less in the Western wall and less in the South-West corner. The combined picture suggests at least two starting-points for the fires that were lit to burn down the mega-structure: a dominant cluster in the South-West corner and a less marked concentration in the North-East corner. The daub firing temperature results do not contradict this proposal.