PREPARATION AND COMPOSITION OF CLAY PASTE IN BRONZE AGE POTTERY FROM NORTH-WESTERN CROATIA: THE ROLE OF EXPERIMENTS

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Introduction

This paper presents the preliminary results of two sets of experiments conducted as part of research into Bronze Age pottery production technology in north-western Croatia. A study was carried out on 7891 pottery fragments recovered from five Bronze Age settlements situated in the river valleys along small streams in the Turopolje and Podravina regions (Figure 1). The pottery was attributed to the early phase of the Central European Urnfield culture, dating to the period between the 15th and 13th c. BC (Kudelić 2015, 2016). The results presented in this paper consider the research conducted on the manufacturing processes of the clay paste and firing conditions of the Bronze Age ceramics and experimental briquettes. The experiments were aimed towards finding a recipe similar to that used for the Bronze Age pottery. Clay materials were collected from the vicinity of Bronze Age settlements and were used to produce 82 experimental briquettes according to various recipes (organic, grog and clay pellet temper) and firing conditions. The recipes were documented using a digital microscope (magnification x20-60) observing fresh fracture surfaces of the briquettes and Bronze Age pottery.

Technological characteristics of Bronze Age pottery from north-western Croatia

Pottery production in the north-western part of Croatia was abundant during the end of the Middle and the beginning of the Late Bronze Age, which resulted in a huge number of ceramic fragments being recovered from archaeological sites. The wide variety of vessel forms pointed towards a low degree of form standardisation (Figure 2). According to macroscopic observation, the pottery was hand-made and of low quality (highly porous and low-fired), which meant that the recovered fragments were poorly preserved. Potters used several forming techniques, such as modelling a clay lump by drawing and pinching, although the main technique was coiling and slabbing (Figure 3). Horizontal traces from using rotation in the vessels' final forming stage were recorded on the rim and the base areas of several fragments. The surface was smoothed (over 60% of the overall pottery sherds), and occasionally burnished (1–4%) during the vessels' final processing stages. Traces of slip coating have also been recorded from macroscopic observation of the pottery and these show high colour contrast between core and surface of the fresh fractures and peeling of layers from the surface. Vessels were usually fired under a reducing atmosphere, or in an incomplete oxidising atmosphere, which resulted in a predominantly dark grey colour of the core, while the surface retained a dark brown, greyish-brown or yellowish-brown colour.

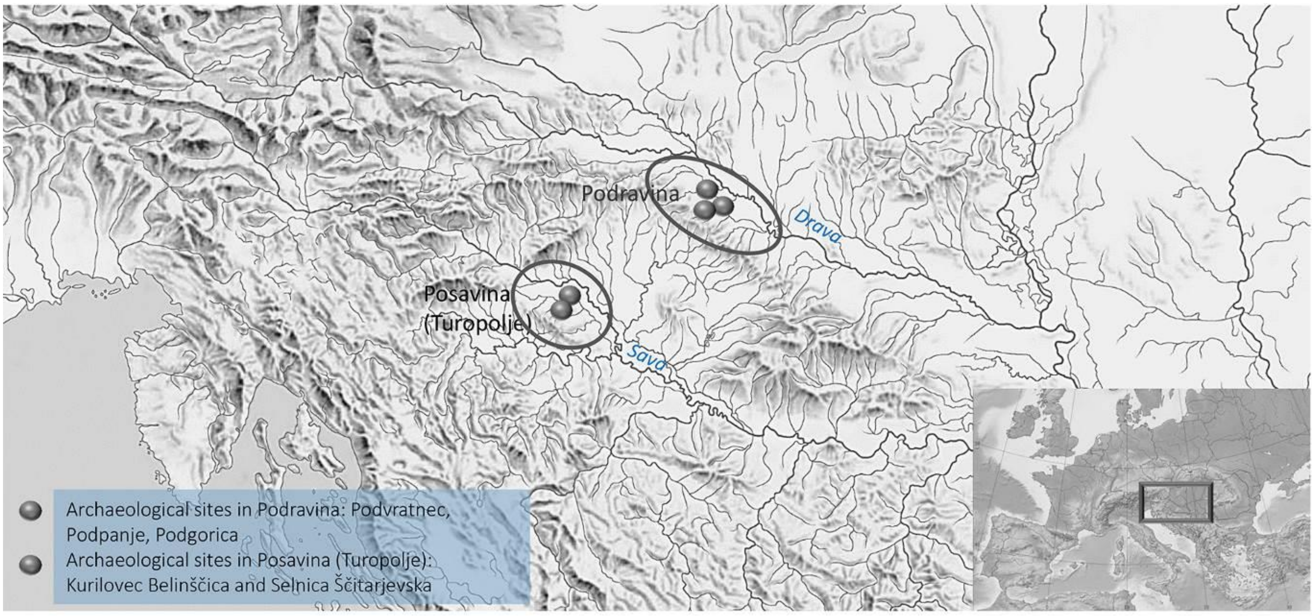


Figure 1. Location of the Bronze Age sites in north-western Croatia.

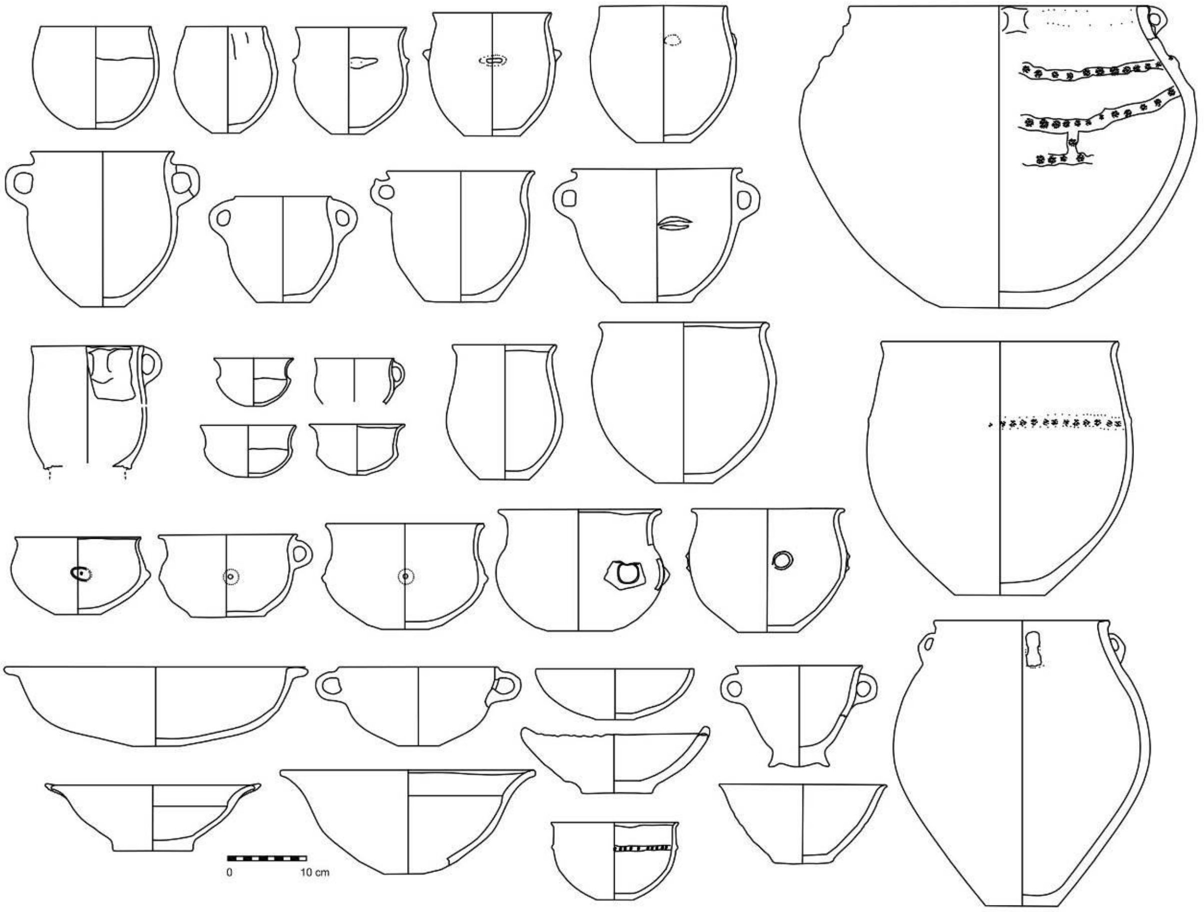


Figure 2. Basic vessel forms dating between the end of the Middle and the beginning of the Late Bronze Age in north-western Croatia (drawing by A. Kudelić).

Figure 3. Bronze Age site of Kurilovec-Belinščica: traces of forming techniques indicating coiling and slab building on the interior surface of the vessels (arrowed) (photographed and drawn by H. Jambrek and A. Kudelić).

Archaeometric analyses (XRPD and thin section petrography) of the clay samples and ceramics showed that the raw material used for the Bronze Age pottery was collected from the vicinity of the settlements, as the local alluvial clays (clayey silt and silty clay) were of sufficient quality for use in pottery shaping (Kudelić *et al.* 2017). Petrographic analysis showed that potters prepared the clay pastes by adding crushed pottery (grog), and probably an organic temper (Figure 4; Kudelić 2015; Kudelić *et al.* 2017). From the high number of tiny pores and voids present in the microstructures of the pottery, it is assumed that the organic temper was very fine. Grog was added to the clay material of all types of ceramic vessels in different amounts, and this was determined by macroscopic analysis to be between 5% and 40%.

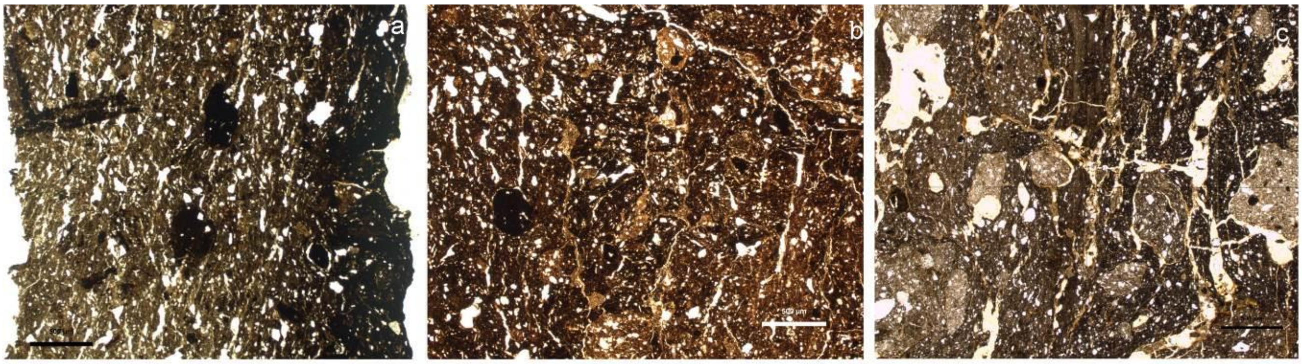


Figure 4. Photomicrographs of thin sections of Bronze Age pottery (scale bar = 0.5 mm): a) and b) pottery from the site of Podgorica; c) pot from the site of Kurilovec-Belinšćica (by M. Mileusnić, modified by A. Kudelić).

According to macroscopic analyses, the Bronze Age samples were divided into six main fabric groups. Pottery fabrics were determined by type, size, and frequency of the temper (grog grains), where the grains were identified using a digital microscope and analysing the images of the fresh fracture surfaces of the pottery. 99% of the 182 analysed pottery samples contain grog (Kudelić 2015). However, the results of the microscopic analysis did not provide sufficient evidence to fully characterise the preparation of this paste - such as the amount of grog or types of organic temper. Experimental research was therefore undertaken to provide answers to these questions.

First set of experiments

Collecting clay material

Field survey has shown that easily accessible clay material of good quality is located in the immediate vicinity of the archaeological sites. Clay samples were collected from these areas as potential sources for the raw material used in the Bronze Age. These were used for archeometric analyses (Kudelić *et al.* 2017) and to produce experimental briquettes. Three clay samples were selected from different deposits located near the remains of a Bronze Age settlement in Turopolje (Table 1). The first deposit is located in a brickyard, four kilometres from the archaeological site of Kurilovec-Belinšćica. The sample acquired at this site was a mixture of clay types (sample M, Table 1). Due to the size of the clay mine, and the depth of the exploitation pit, the well-stratified clay layers within the profile provide valuable insight for geologists, and assure a precise clay deposit analysis. The other two deposits were found by chance. The first deposit is located 600 m to the north-east of the archaeological site (labelled G, Table 1) and the sample was collected from the depth of more than one metre from the surface. The second deposit is located 500 m to the south-east of the site (sample K, Table 1) and the sample was collected from a much shallower depth of 40 cm from the surface and it was used in the second set of experiments. Clay material was also collected in the Podravina region, in the river Drava valley (samples P.Pv and P.Pg, Table 1) in the immediate vicinity of the remains of two Bronze Age settlements, and used in the second set of experiments.

The clay material was extracted by drilling 30-70 cm deep cores from the deposits. Sampling locations were recorded using GPS (Table 1). Collected clay samples were manually cleaned of larger inclusions such as gravel, leaves or similar unwanted organic material. Physical properties of each clay sample were observed: plasticity and its effects on shaping, drying time, and ease of manufacture in relation to the addition of different types and quantity of temper.

This first set of experiments was initiated before archaeometric analysis was undertaken.

Grog and organic tempered briquettes

Thirty-five briquettes were produced using different recipes in order to better understand preparation processes to be compared with the freshly fractured archaeological ceramics (Figure 5). In order to make a range of briquettes with different characteristics, grog was prepared using different available materials (brick, briquette and contemporary ceramics), and ground using a hammer (first a stone hammer, then a metal one) on a stone base which was used as a grindstone. It was noticed that grinding produced an extremely small amount of grog in relation to the initial quantity of raw material. Sieving was necessary to separate the grog grains from the powder obtained by grinding, because the dust severely dried out the clay paste. Two sieves of different sizes were used in the process to separate powder, grog inclusions up to the size of 1 mm, and the inclusions up to 2.5 mm, the latter being recorded in Bronze Age ceramics. Chaff was added as an organic temper, along with straw cut with scissors. The amount of grog added to each briquette was measured by weight in relation

Clayey sample label	Region (NW Croatia)	Coordinates	Distance from the archaeological site	Macroscopic description	Mineralogical determination
M	Turopolje	45.657977 16.084164	4 km	brown-yellow	clayey silt
G	Turopolje	45.700522 16.063316	0.6 km	grey-brown	silty clay
K	Turopolje	45.690340 16.071262	0.5 km	grey-brown	silty clay
P.Pg	Podravina	46.181544 16.892333	30-70 m	grey with yellow spots	silty clay
P.Pv	Podravina	46.221772 16.848861	30-70 m	grey-brown	silty clay loam

Table 1. List of clayey materials used for the experiments discussed in the article.

Figure 5. Temper used in the briquettes showing chaff with straw and grog grains in two sizes (photographs by A. Kudelić).

to the weight of dry clay to which it was added (5-10%), while the organic material was measured by volume (Figure 6). The amount of grog added to the paste was an estimation of the percentage area visible in the sample fresh fracture (Quinn 2013, 82).

In the drying process, the resistance of clay material to relatively fast drying was documented. In order to simulate quick drying conditions, the briquettes were dried with the help of a heating device. Heating response was dependent upon clay types and temper used. It was noticed that sample G shrank noticeably in the course of drying, and that it dried out more slowly than other briquettes.

Two copies of experimental briquettes were made from clay samples M and G (Table 2). The aim was to establish characteristics and differences visible on fresh fractures of experimental ceramic samples made using different recipes and fired under different conditions (Table 3), and to compare them with the Bronze Age pottery. One set of briquettes was fired in an open fire in incomplete oxidising conditions at a maximum temperature of 730°C, the other set in an oxidising atmosphere in an electric kiln at the same temperature (Table 3; Kudelić 2013). The firing in both cases lasted 5-10 minutes, after which the briquettes were removed. The briquettes fired in the fire the soaking time was short, approximately 5-10

Figure 6. Cup with volume markings of clay and temper used in the experiments with cow dung, grog and clay pellets (photographs by A. Kudelić).

	Clayey sample	Recipe		Fired briquettes	Firing conditions			Unfired briquettes
					Bonfire		Electric kiln	
First set of experiments	M; G	0	without temper	3			+	
	M; G	1	5% grog	6	+		+	
	M; G	2	10% grog	6	+		+	
	M; G	3	chaf and straw	4	+		+	
	M; G	4	chaf and straw and 5% grog	4	+		+	
	M; G	5	sand 10%	6	+		+	
	M; G	6	5% sand and 5% grog	6	+		+	
					Pit firing (A)	Bonfire (B)	Electric kiln (C)	
Second set of experiments	K; P.Pg; P.Pv	1	without temper	9	3	3	3	3
	K; P.Pg; P.Pv	2	dung 50%	9	3	3	3	3
	K; P.Pg; P.Pv	3	dung 30% and grog 20%	9				3
	K; P.Pg; P.Pv	4	clay pellets 20%	3	3 0	3 0	3 3	6
	K		clay pellets	2	2			
Total number of briquettes	**82**			**67**				**15**

Table 2. Summary of experiments discussed in the text.

minutes after which the briquettes were taken out of the fire. The briquettes fired in the electric kiln, after 10 minutes of soaking time, were left in the kiln to cool for approximately 2 hours. The study showed that the local clay has properties of sufficient plasticity for vessel shaping, and withstands rapid drying. However, the briquettes made of clay sample G, regardless of the recipe, were damaged during firing in an open fire. Defects in the form of fire spalls were recorded on two briquettes, where such defects are normally associated with evaporation of water from the clay during a rapid rise of temperature. Considering that sample G demonstrated a slow discharge of moisture during the drying process, it was assumed that the clay contained minerals that were subject to swelling. However, subsequent archaeometric analyses showed that the clay, just like clay sample M, does not contain such minerals (smectites and vermiculite), but that clay is of good quality for pottery production as it contains a mixture of illite, kaolinite, chlorite and abundant quartz. It was also confirmed that the presence of chaff temper in the clay paste significantly affects the colour of the core of the ceramic sample, as well as the firing conditions (Table 3).

Second set of experiments

The second set of experiments focused on aspects of organic temper, and on the raw material from which grog was made. Forty-seven experimental briquettes were made (Table 2) using three different clays (samples K, P.Pv and P.Pg), and following four distinctive recipes (marked with numbers: 1, 2, 3, 4). The first group did not contain temper, and it was used as a reference (Table 4). The second group was tempered with cow dung (50%); the third group with cow dung (30%) and grog (20%); and the fourth group with clay pellets (20%). Briquettes were made in three series (marked with letters: A, B, C,) using different firing conditions (Table 5).

Cow dung and grog

The first set of experiments suggests that straw needed to be chopped up with a sharp tool in order to properly prepare it as temper. However, although chopped straw was added to the clay samples, the temper appeared differently in the briquette microstructure compared to that of the Bronze Age pottery (Table 3). The Bronze Age pottery fabrics had abundant tiny pores and voids suggesting that the organic temper was very finely processed.

Recipe	Macrostructures of the fired briquettes		Firing conditions
	M	**G**	
No temper			Electric kiln max. 730°C
5-10% grog			incomplete oxidation max. 730°C 
5% grog, chaff and straw			
chaff and straw			
chaff and straw			Electric kiln max. 730°C
5-10% grog			Electric kiln max. 730°C

Table 3. Fresh fractures of briquettes from the first set of experiments.

Recipe	**Firing conditions**		
	A (Pit firing - incomplete reduction, max. 640°C; soaking time 20 min.)	**B** (Bonfire - incomplete oxidation, max 730°C; soaking time 10 min.)	**C** (Electric kiln - incomplete oxidation max 650°C; soaking time 10 min.)
K1		*damaged during firing*	
P.Pv 1	*damaged during firing*		
P.Pg 1		*damaged during firing*	

Table 4. Fresh fractures of untempered briquettes fired in different conditions (empty boxes: briquettes were severely damaged during firing).

Therefore, it was concluded that chopped-up straw was not used for manufacturing the early Urnfield pottery. Because of the high number of tiny pores and voids found within the Bronze Age pottery fabric, organic temper seemed to have been very finely processed (Figure 4). As mentioned above, the amount of organic temper in some ceramic samples was up to 40% (Figure 4). As temper of plant origin chopped so finely can be found in cow dung, and it requires no tools for its preparation.

Therefore, dry cow dung was added as experimental temper. Pieces of dung were easily crumbled with fingers to the required size, although, as with grog preparation, it was necessary to sieve the dust and separate larger dung pieces from the rest. Such temper can be easily mixed with clay, even though water must be added to facilitate mixing by maintaining sufficient plasticity of the clay paste when more than 30% by volume has been added. 50% of cow dung temper was the maximum amount that could be added to a clay without losing all the plasticity (Table 5).

In the third group of briquettes, grog (20%) was added with cow dung (30%). Grog was made of waste produced in previous experiments and, unlike grog made from ceramic building material, these inclusions were more rounded and porous.

As well as to recreate the appearance of temper in the briquettes and determine the amount of cow dung required to replicate the Bronze Age ceramics, another objective was to determine to what extent such temper affects the colour of the ceramic core in cross-section during firing under different conditions. Since the research is still in progress, these results are based upon the experiments of three groups of briquettes fired under different conditions. The first group of un-tempered briquettes and two tempered groups totalled 30 briquettes (Tables 2 and 5). One group of briquettes was fired in a pit with a partially reducing atmosphere (series A) using dry branches, dry grass and cow dung as fuel. The firing lasted almost 5 hours and the highest temperature achieved was between 600°C and 640°C, and was maintained for about 20 minutes. Unfortunately, the samples were not completely fired because the experiment was interrupted by rain. It is likely that two more hours of firing would have been sufficient for completion and to reach at least 700°C. The second group was fired in an open fire (series B) in incomplete oxidising conditions at a maximum temperature of 730°C. The fuel used was wood and

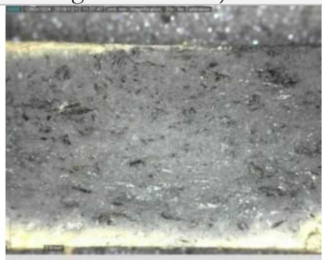
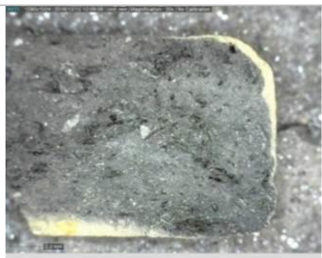
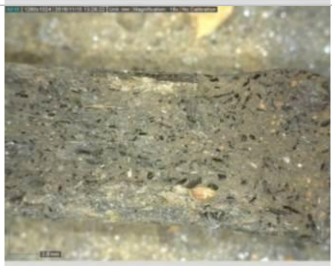
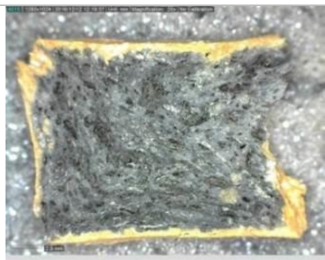
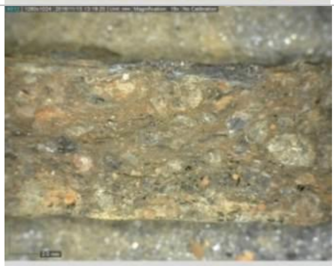
Recipe	Firing conditions		
	A (incomplete reduction max 630°C; soaking time 20 min.)	**B** (incomplete oxidation max 730°C; soaking time 10 min.)	**C** (incomplete oxidation max 650°C; soaking time 10 min.)
K2 50% cow dung			
K3 20% grog 30% cow dung	*damaged during firing*		
P.Pv 2 50% cow dung			
P.Pv 3 20% grog 30% cow dung			
P.Pg 2 50% cow dung			
P.Pg 3 20% grog 30% cow dung			

Table 5. Fresh fractures of briquettes from the second set of experiments.

cow dung. The firing lasted between 35 and 40 minutes with a short soaking time, approximately 5-10 minutes, after which the briquettes were removed from the fire. The third series (C) of briquettes were fired in an electric kiln at 650°C for 25 minutes with a short soaking time (10 minutes) after which the samples were removed from the kiln.

Clay pellets as tempering material

The first experiments showed that a relatively large amount of raw material for making grog turned to powder, which is not effective for use as temper. In this experiment an alternative variant of temper with similar characteristics was tested: dry lumps of clay were prepared as pellets, by grinding and sieving. One of the research goals was to determine the properties of this temper, and whether, after firing, there are visible differences between the grains of temper added as grog and those added as unfired clay pellets. One series of nine briquettes tempered with clay pellets was fired in the electric kiln and two samples were fired in the pit (Table 6).

Preliminary results

The fresh fracture of experimental briquettes, regardless of firing conditions, demonstrated that cow dung significantly influenced the colour of the ceramic samples' core. The cores are dark grey with sporadically distributed pronounced black smudges. Apart from the colour, the organic temper also appears in the form of well-distributed pores of oval or elongated shapes, ranging in size between 0.3 and 1.0 mm. One sample (Table 5, sample P.Pg 2A) shows a predominant number of black oval or circular voids which occurred when the fine plant seeds combusted and burned out during the firing process. Fractured briquettes tempered with cow dung and grog, share common physical characteristics with a high number of Bronze Age pottery sherds (Table 7). In contrast to the grog produced from contemporary ceramics during the first experiments, the clay pellets added to the briquettes (Table 6), display more rounded (softer) shapes and porous structures and are also very similar to the grog of the Bronze Age ceramics.

The results of the archaeometric analyses of the Bronze Age ceramics indicate that the maximum firing temperature was around 700°C (Kudelić *et al.* 2017); the experimental firing conditions reached a similar temperature and the colours indicated both oxidising and reducing atmospheres with relatively short soaking times (Table 7).

With the exception of two briquettes tempered with dried clay pellets that were fired in a pit at a maximum temperature of 650°C, three additional briquettes made of the same recipe were fired in an electric kiln (series C) at the same temperature with a short soaking time (10 minutes). Visible on a fresh fracture of a fired briquette, pieces of the original dried clay pellets show diffuse or rounded edges, i.e. the grains are partially fused with the matrix (Table 6). However, in most cases the grain edges are partially angular and clearly separated from the matrix (Table 6), therefore fully corresponding to the appearance of grog. According to the literature and the already established morphological characteristics, the fired grains from clay pellets have rounded edges while grog has partially angular grains (Whitbread 1986; Quinn 2013, 84; Albero Santacreu 2014, 62). The results of these experiments need to be checked in a larger number of samples.

Discussion

This experimental research confirmed hypotheses about finely fragmented organic temper of vegetal origin which appears to have been used in the preparation of the early Urnfield culture clay pastes. Moreover, the experiments suggest the possibility that the Bronze Age potters used cow dung as organic temper, in addition to grog (and/or clay pellets?).

In many cases temper has a functional purpose, although there are notable reasons of cultural or ideological nature for the use of grog (De Boer 1974, 336; Smith 1989, 61; Chapman 2000; Brück 2006; Kreiter 2007). The tradition of using cow dung in pottery production (as fuel as well as a temper) has been recorded in ethnographic sources (Sillar 2000; Gosselain 2008, 34; Albero Santacreu 2014, 70), although there are indications that it was also used in the past (Albink 1999, 134). Non-plastic temper material (grog, sand, etc.) as well as organic tempers (straw, chaff, bones, shells, etc.) are added to the clay paste in order to reduce its plasticity, facilitate the forming and drying of the vessel, and ensure a good level of heat resistance during the firing process and during the vessel's use (Velde and Druc 1999, 83; Gibson and Woods 1990, 27; Skibo *et al.* 1989). The main function of a highly-tempered clay paste in open-fired pottery is to open the body, allowing the water to evaporate during the early stages of firing (Gibson and Woods 1990, 207). It is therefore important to determine the type and characteristics of temper, as its properties directly affect aspects of vessel production and use. For example, a relatively low firing temperature (550°–700°C) and selection of firing atmosphere (reducing or incomplete oxidation), appear to be closely linked to the specific temper used in clay pastes. Paste rich in organic matter has a much higher rate of sustaining fractures when slowly fired. Rapid firing is a more appropriate method for a paste tempered with an organic material and such paste might serve as an indicator of the use of low firing temperatures (550–650°C) (Albero Santacreu 2014, 99).

Recipe	Firing conditiones	
	C (incomplete oxidation max 650°C; soaking time 10 min.)	
	Fresh fracture	Surface
K4		
P.Pv 4		
P.Pg 4		
	(incomplete reduction max 650°C; soaking time 20 min.)	
K4		

Table 6. Fresh fracture of experimental briquettes tempered with clay pellets (black arrow: grains are partially fused with the matrix; blue arrow: angular grains).

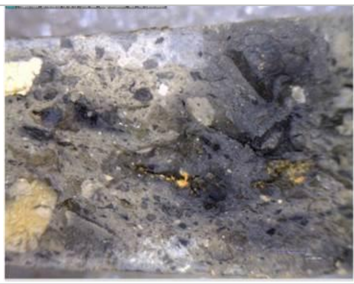
Recipe and firing conditions	**Experimental briquettes**	**The Bronze Age pottery**	
P.Pg 3A			
P.Pg 3B			
K2A			
P.Pg 2C			
M chaff and straw			

Table 7. Fresh fracture of experimental briquettes and Bronze Age ceramics (made by A. Kudelić).

In addition, another advantage of this type of paste is that its porous wall can absorb an additional wet layer of clay added to the vessel in the form of a slip (London 1981, 194). Due to the fact that some of the examined Bronze Age pottery fragments have traces of slip, it is possible to consider the function of fine organic temper and its relationship with final surface treatment techniques.

Conclusion

Bronze Age early Urnfield pottery in north-western Croatia was hand-made and of low quality, being highly porous and low-fired.

The temper added to the Bronze Age clay pastes was primarily grog and fine organic material. Grog is recorded in almost all studied vessel types sometimes in large quantities (30-40%), which suggests that a large amount of ceramic waste was produced from the settlement. Experiments showed that during grog preparation, grinding and sieving produced an extremely small amount of grog in relation to the initial quantity of raw material indicating a need for a relatively large amount of source material. Furthermore, this may be an indication of special treatment of the ceramic waste in a settlement or household by collecting and storing it, either by potters or the community. The extent of this type of recycling material is not easily measured. This will hopefully be investigated by future research. On the other hand, there are instances in which dry clay pellets were probably deliberately added as well as, or instead of, recycled pottery.

Experiments were conducted on briquettes made from locally collected and relatively good quality clay, which was an easily available raw material in the Bronze Age. The experiments were aimed towards finding a recipe with added temper, similar to that found in the Bronze Age pottery. Fractured briquettes tempered with cow dung and grog, share common physical characteristics with a high number of Bronze Age pottery sherds. However, the experiments point to caution in interpreting temper selection because deliberately added dry clay pellets in the unfired clay, appear to look similar to grog in the freshly fractured ceramics.

The frequent presence of fine porosity in the archaeological samples seems to suggest the possibility that ruminants' dung or a highly crushed temper of plant origin was added. It was shown that producing temper from cow dung requires grinding and sieving. This might be also associated with surface finishing techniques, such as the application of slip. However, other archaeometric research shows that slips were applied to well-burnished vessel surfaces, e.g. in the Neolithic of the central Balkans (Spataro 2016).

Research is still ongoing. The results of these studies should certainly be supplemented by analysis in thin section of selected experimental briquettes in order to gain a better insight into porosity, as well as other characteristics of the clay pastes.

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