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Early Neolithic pottery of Ifri n'Etsedda, NE-Morocco – Raw materials and fabrication techniques

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ABSTRACT

Ifri n'Etsedda is a rock shelter in northeastern Morocco, at the southern flank of the Kebdana Mountains. The site was discovered in 2008 and excavated during three field campaigns between 2012 and 2014 and provides Epipalaeolithic and Neolithic deposits. From the Neolithic deposits we analyzed 30 pottery sherds by macroscopic determination, Infrared (IR) Spectroscopy, polarizing microscope and thermodilatometric analysis (TDA) in order to determine information about the clay and natural inclusions in the clay and temper materials used as well as the manufacturing process. In addition, we identified and sampled 12 potential raw materials sources during one survey campaign in 2015, which were analyzed by IR spectroscopy as well. We identified 8 groups by means of inclusions in the clay: extrusive igneous rocks (EIR), EIR with amphiboles, EIR with fragments of micro quartz, granite, dolerite or gabbro with fibrous quartz and unidentified spherulites, schist, organic limestone and grog. Trends in the succession of clay traditions could be identified. Most of the pottery was fired in an oxidizing atmosphere and were placed upside down in a pit. Burning temperatures range between 800 and 1000 °C. No local clay source seems to have been used for the pottery production at Ifri n'Etsedda. With regard to the small artifact samples size as well as the incomplete raw material survey, further studies are needed to shed more light on Ifri n'Etsedda pottery.

1. Introduction

The paper presents a pilot study, which deals with mineralogical and geochemical analyses on early pottery from Ifri n'Etsedda, a rock shelter in NE Morocco (Fig. 1A). The site was discovered in 2008 and excavated during three field campaigns between 2012 and 2014 (Linstädter et al., 2016). Ifri n'Etsedda provides Epipalaeolithic and Neolithic deposits dated between 10.0 and 6.0 ka calBP and thus embody the Neolithic transition. Neolithic deposits are preserved in situ only in the excavation of 2012. Epipalaeolithic sediment couldn't be determined in this part of the shelter. In contrast, Epipalaeolithic deposits are preserved in situ in the excavations of 2013 and 2014 (Linstädter et al., 2016: Fig. 2). Neolithic deposits with the two latter excavations are disturbed during later occupation. In total 11 layers could be determined for Ifri n'Etsedda and labeled as INES-1 to INES-11 (Fig. 3):

The first Neolithic occupation is comprehensible in INES-5 due to the appearance of pottery, perforated snails and domesticated animals and plants. INES-5 can be dated around 7.2 ka calBP, but is a very thin layer. The following layer INES-6 is dated around 6.8 ka calBP. In both layers ceramic sherds with *Cardium* decoration are present. INES-7 represents the largest occupation phase and can be dated between 6.7 and 6 ka calBP. The pottery of this layer show herringbone motives which were formed by marine shells. INES-8 is not absolutely dated, but pottery with comb impressions and undecorated, polished vessels suggest a late Neolithic occupation. Altogether the ceramic assemblage consists of 120 pottery units with focus on the late Early Neolithic phase ENC.

Only a few archaeometric studies on pottery are known from the area (Linstädter and Müller-Sigmund, 2012), so that the here-presented analysis allows to significantly further our knowledge about the minerology of the early Neolithic pottery of Morocco. The focus of our study lies on characterizing the used clay with natural inclusions and temper materials present, finding out their provenance, reconstructing the pottery production techniques and understanding the changes of these features through time. We aim to answer two questions: which

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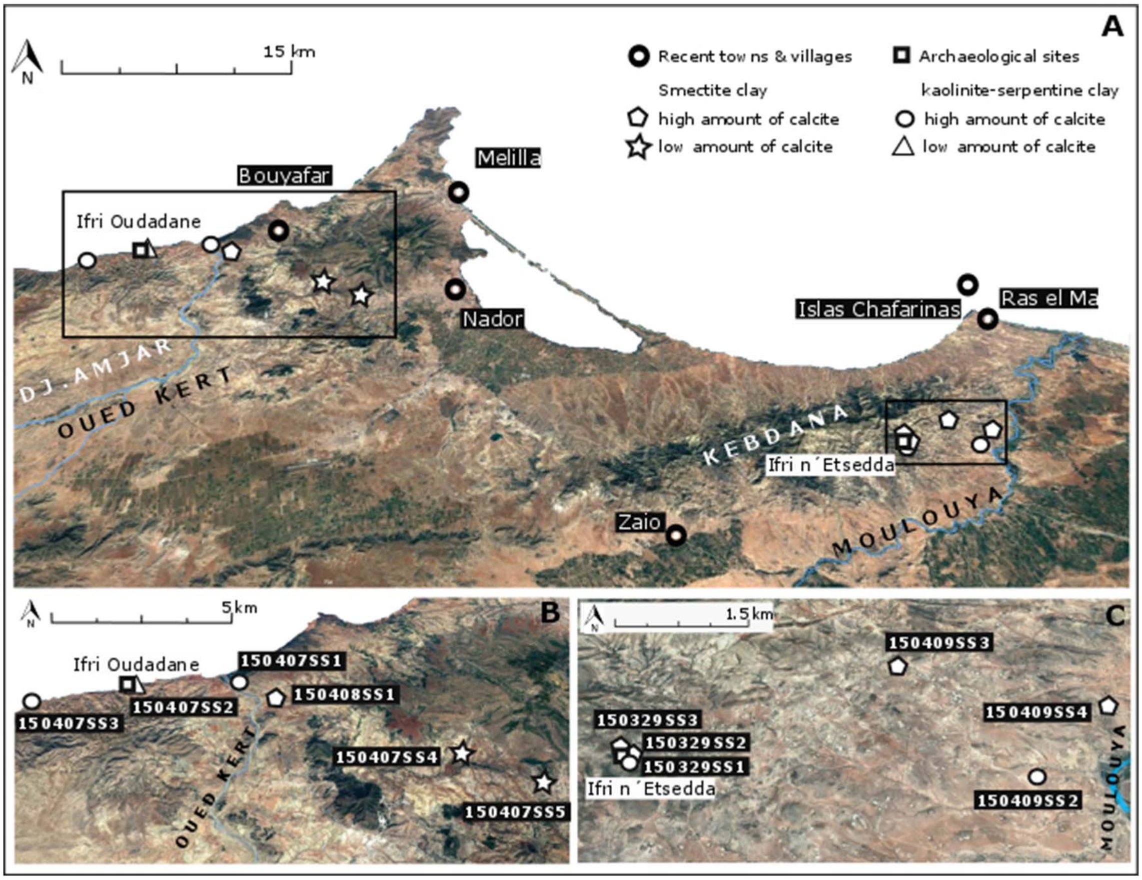

Fig. 1. Map of the research area with the raw material sources sampled. Close-up on the two regions around Ifri Oudadane and Ifri n'Etsedda.

kind of raw materials were used and where did they come from? How was the pottery produced? To answer these questions we undertook systematic survey and sampling and applied a set of archaeometric analyses. A systematic stylistical study of the Ifri n'Etsedda pottery assemblage is still ongoing.

The Neolithic transition, understood here as a concept describing the transition to food production, is one of the most interesting phenomena in archaeology, providing far-reaching consequences for human societies. The Western Mediterranean, or so-called Alboran Territory, provides a fascination laboratory to study this process since it includes rather diverse ecological zones spanning from temperate Western Europe to semi-arid Northwest Africa.

Based on the studies of the lithic industries of Ifri Oudadane one can assume that no population exchange occurred (Linstädter et al., 2015). Raw material supply, technology and tool composition of Epipalaeolithic and Neolithic assemblages are rooted in the same tradition. The transitional process however is marked by the appearance of domesticated plan and animal species. In northwestern Maghreb the cultivation of domesticated crops and animal husbandry is estimated to appear at around 7.5 ka calBP (Morales et al., 2013; Zapata et al., 2013). However, the composition of the various new food components is not homogeneous. A concomitant analysis of multiple Neolithic sites shows the combination of species (e.g. crop species) differs from assemblage to assemblage (Morales et al., 2016). Furthermore, the percentage of domesticated species varies and amounts often < 50%. Hunting and gathering (Hutterer et al., 2014) as well as the use of marine resources still play an important role. Thus, in large part of northwestern Maghreb food production can be considered a risk minimizing facet, rather than a dominant economy.

Beside its impact on diet, the transition to food production has indirect influence on the environment (Linstädter et al., 2016), fuel supply (Lehndorff et al., 2014) and material culture. Pottery, one of the first synthetic materials known, is one of the most eye-catching features of this transitional process. In general, pottery is one of the most frequent find categories at archaeological excavations dating from the Neolithic onwards. The study of shapes and decorations of ceramic objects plays an important role for the development of chronologies and mobility hypotheses. Recent studies on early pottery from the Mediterranean Maghreb attest of an affiliation of this region to the Western Mediterranean circle (Linstädter, 2004; Linstädter and Wagner, 2013).

While typological studies allow the identification of transregional relations, the use of chemical and physical analyzes provides new possibilities for the study pottery, its technical development and its provenance (Maggetti, 2008; Wagner, 2007). The analysis of raw material provenances is at the basis of several other studies, such as land use and mobility pattern reconstruction (Basso et al., 2006; Kibaroğlu, 2008; Papmehl-Dufay et al., 2013). However, within northwestern Maghreb such analyses have rarely been applied until now. A few examples are the study of Neolithic material discovered in the Oukaïmeden Valley, Moroccan High Atlas (Maicas et al., 2014), and the analysis of pottery from divers sites of the eastern Rif (Linstädter and Müller-Sigmund, 2012).

2. Materials and methods

2.1. Samples and sample preparation

Since no systematic stylistical study of the pottery of Ifri n'Etsedda could be used for sample selection, we selected 30 sherds of different stratigraphic origin based on macroscopic criteria like clay color, wall thickness and macroscopic inclusions (Fig. 3). It was not possible to analyze a larger samples size, but to be representative of the site's

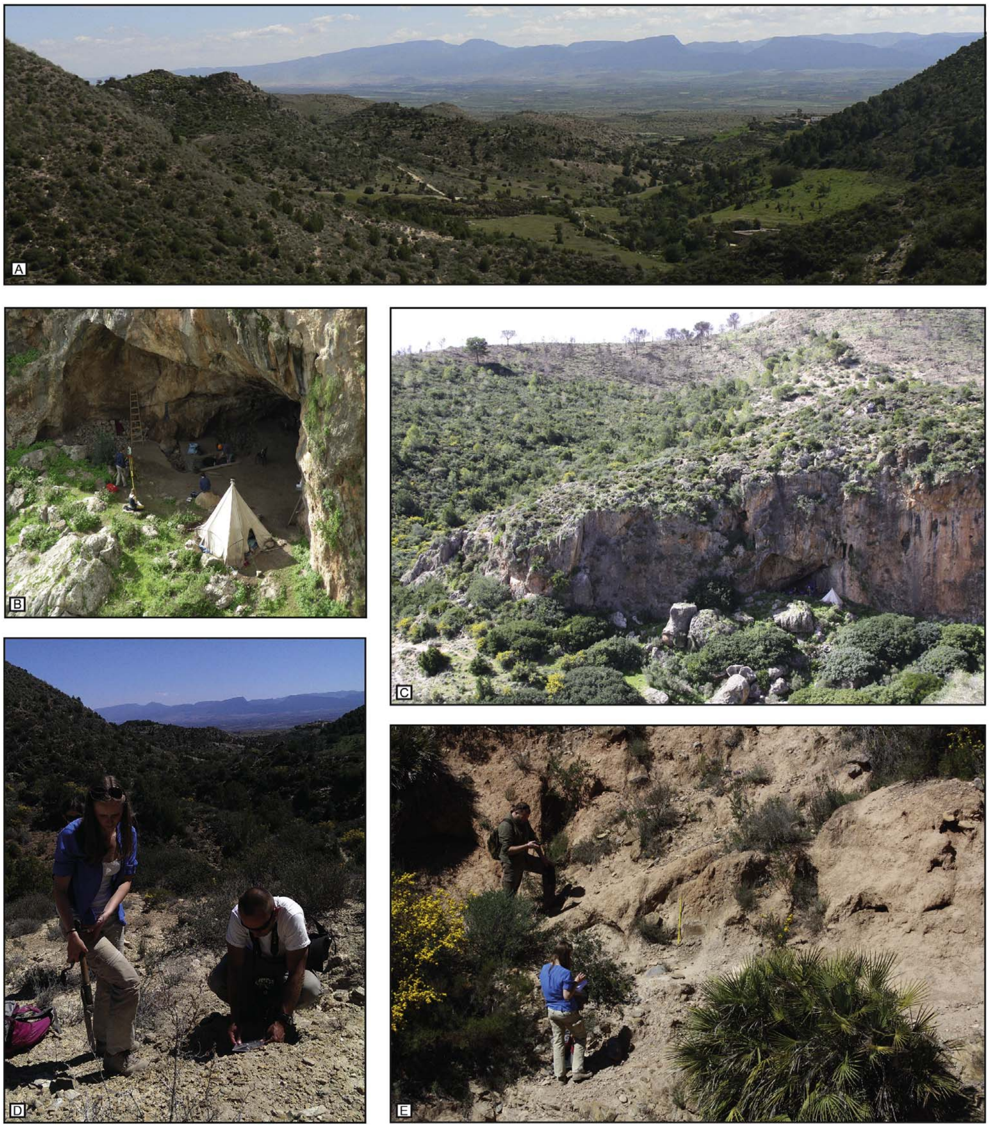

Fig. 2. Selection of the pottery sherds sorted by layer.

pottery assemblage, we chose the samples based on color, surface, amount of oxidation and reduction zones and macroscopic aspect of the temper (Table 1). Due to a very low amount of decorated sherds as well as determinable sherds such as rim sherds, we were only allowed to use undecorated sherds (except one) as well as small, undeterminable pieces for analysis, which can only be identified as part of the body. The analyzed sherds were excavated from four Neolithic layers INES-5 to INES-8 as well as from two disturbed layers on top of these called INES-9 and INES-10 (Linstädter et al., 2016). Stratigraphy and sample provenance is illustrated in Fig. 3. On the basis of radiocarbon dates and artifact material (Linstädter and Wagner, 2013), the stratigraphic units of Ifri n'Etsedda (INES) are paralleled with the occupation phases of Ifri Oudadane (Early Neolithic A to Late Neolithic): INES-5 (~7.2 ka calBP) was assigned to the Early Neolithic A (ENA), INES-6 (6.5–6.8 ka calBP) to the Early Neolithic B (ENB) and INES-7 (6.6–6.1 ka calBP) to the Early Neolithic C (ENC; Fig. 3). INES-8 is undated; however the vessels providing comb impressions and undecorated pottery suggest a Late Neolithic (LN) occupation. Sample numbers, ages and descriptions are summarized in Table 1.

We cut 30 μm standard thin sections from 25 of the sherds for petrographic analysis and prepared a powder of a few milligrams from each sherd for Infrared (IR) spectroscopy. Approximately 2 × 3 × 0.5 cm measuring thick sections were cut from 7 sherds for thermodilatometry.

In order to identify potential clay and temper sources, we recorded 12 geological reference samples of clay during one survey campaign in 2015 (Fig. 1). Sample numbers of geological samples are summarized in Table 2. During this fieldwork we systematically sampled the area between the southern flank of the Moulouya and the Kebdana Mountains up to the Mediterranean coast near Ras el Ma (1C). All raw material sources found are located in an area around ~7 km in the east of Ifri n'Etsedda. To obtain a larger sample of clay sources that can potentially be found in the greater region, we surveyed a supplementary area from the lower Oued Kert around the site Ifri Oudadane (Fig. 1B), a Neolithic site situated at approximately 70 km away from Ifri n'Etsedda. According to Linstädter and Müller-Sigmund (2012) a sherd from Mtlili 6 (Moulouya), provides inclusions with andesite composition (Linstädter et al., 2012). Following Linstädter and Müller-Sigmund (2012) this andesite comes from a clay deposit at the Djebel Amjar west of the lower Qued Kert (Fig. 1). In order to verify the suggested connection between both regions, we took another six samples from the area east of Djebel Amjar area (Table 2).

2.2. Analyses performed

All archaeological samples underwent macroscopic analysis to

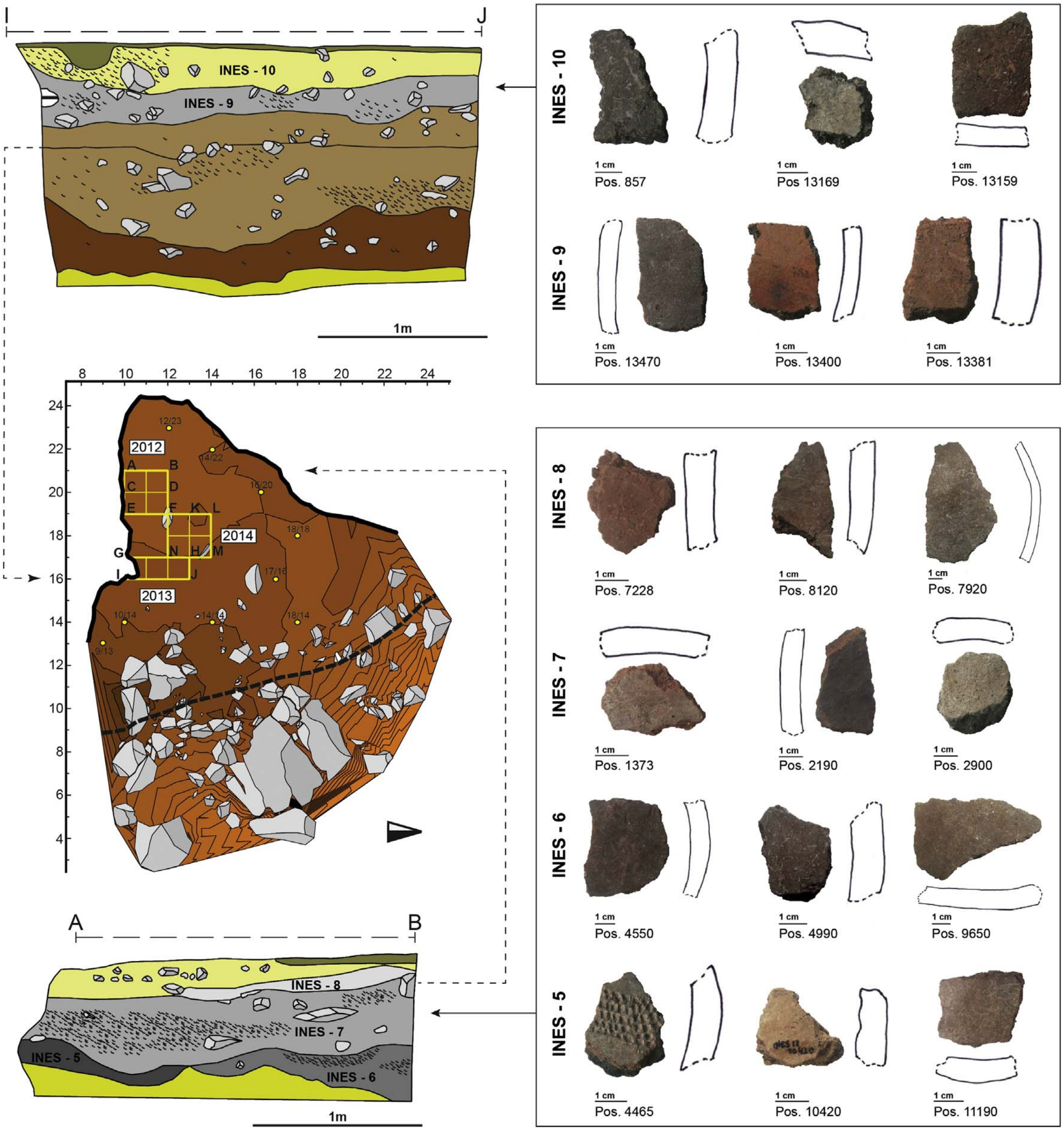

Fig. 3. Stratigraphy of Ifri n'Etsedda (Linstädter et al., 2016).

determine the parameters: wall thickness, burning atmosphere, position in the kiln, colors of the sherd, amount of inclusions and maximum grain size (Noll, 1991; Riederer, 1987; Velde and Druc, 1999). Due to the availability of our research equipment and the sometimes varying suitability of the samples for specific techniques, different numbers of samples were used within the different analytical techniques: IR spectroscopic analysis was performed on all 30 sherds samples. 25 of these samples were analyzed by a polarizing microscope as well (except sample 4465 (ENA), 5080 (ENB), 1373 (ENC), 7920 (LN?) and 13,380 (Neol. mixed). Thermodilatometry analysis was performed on seven samples in total: 5080 and 9650 (ENB), 1230, 4210 and 8870 (ENC), 13,470 (Neol. mixed) as well as 1330 (subrecent mixed).

We used IR spectroscopic analysis to determine clay minerals and the presence of crystalline and non-crystalline impurities (Komadel and Madejová, 2001; Madejová, 2003). The analysis was performed on powdered samples of all artifacts and geological samples in Attenuated Total Reflectance (ATR) mode using an Agilent Cary 660 FTIR spectrometer. The obtained IR spectra were compared with absorption band values from the literature (Hiltmann, 1998; van der van der Marel and Beutelspacher, 1976; Wilson, 1995) as well as with the spectra of our (SS, PS) institute's database of known samples. The Spectra were recorded between 4000 and 400 cm^{-1} with a 64 scans repetition and a resolution of 2 cm^{-1} on all archaeological sample powders. Spectra of geological samples were recorded in the same spectral range but with a 100 scans repetition and a resolution of 4 cm^{-1}.

We performed petrographic analysis of the 25 thin sections with a Zeiss Axio Imager A.2 polarizing microscope to observe the petrographic features of inclusions (shape, color and pleochroism, cleavage, refractive index and optical symmetry). Furthermore, quantitate analysis was performed on 20 samples only, because the thickness of 5 thin sections was insufficient. The average grain size of each type of inclusions was registered and the number of grains was counted at five different locations in each thin section.

We used Thermodilatometric analysis (TDA) to determine the firing temperatures of seven of the sherds. The analyses were performed by with Prof. Dr. Quirmbach of the *Deutsches Institut für Feuerfest und Keramik GmbH (DIFK)* according to the protocol specified in the German industrial standard DIN51045. Samples were heated to 1070 °C

Table 1
Sample numbers, origins, ages, macroscopic description (wall thickness, atmosphere, orientation during firing, amount of inclusions, max. grain size) and archaeometric results of the pottery sherds from Ifri n'Etsedda. EIR = extrusive igneous rock, Ox. = exterior surface oxidized, Ox.* = interior surface also oxidized, (Ox) and (Red) = sample surfaces show a poor state of preservation, temperatures under "firing temperature estimation" were obtained by thermodilatometric analysis if marked "TDA" and through inference of the mineral assemblages identified by infrared spectroscopy if marked "IR".

Sample	Layer INES-	Dating	Cultural phase	Clay color	Surface color	Wall thickness (mm)	Surface Oxidized/ reduced	Orientation during firing	Size of inclusions	Amount of inclusions (macrosc.)	max. Grain size	Infrared Spectroscopy	Petrography (group identified)	Firing temperature estimation
4465	5	7.2 ka calBP	ENA	Reddish	Yellow/ beige	9	Ox.	Mouth	Coarse and fine	20%	2,6 mm	Smectite	–	IR: < 850 °C (smectite, OH present)
6050	5	7.2 ka calBP	ENA	Brown/ reddish	Grey	9	Ox.	Mouth	Coarse	40%	1,2 mm	No result	Granite	–
10,420	5	7.2 ka calBP	ENA	Beige/ grey	Beige/ grey	9	Ox.	Mouth	Coarse (grog)	5%	2,3 mm	Smectite	Grog	IR: < 850 °C (smectite, OH present)
11,190	5	7.2 ka calBP	ENA	Brown/ reddish	Grey	8	Ox.	Mouth	Coarse and fine	10%	2,3 mm	No result	Dolerite/Gabbro	–
12,070	5	7.2 ka calBP	ENA	Brown/ reddish	Brown/ reddish	6	Red.	–	fine	5%	1,1 mm	No result	EIR-microquartz	
4550	6	6.5–6.8 ka calBP	ENB	Brown/ reddish	Grey	10	Ox.	Mouth	Coarse and fine	10%	3,0 mm	Smectite	Dolerite/Gabbro	IR: < 850 °C (smectite, OH present)
4990	6	6.5–6.8 ka calBP	ENB	Brown/ reddish	Grey	11	Ox.	Mouth	Coarse and fine	30%	2,2 mm	No result	EIR	–
5080	6	6.5–6.8 ka calBP	ENB	Brown/ reddish	Grey	9	Ox.	–	Fine	40%	1,9 mm	No result	–	TDA: 850 °C–900 °C
9650	6	6.5–6.8 ka calBP	ENB	Brown/ reddish	Grey	11	Ox.*	Foot	Coarse	20%	1,5 mm	No result	EIR-microquartz	TDA: 950 °C–1000 °C
1230	7	6.1–6.6 ka calBP	ENC	Brown/ reddish	Brown/ reddish	11	(Ox.)	Mouth	Coarse	30%	3,8 mm	Smectite	Schist	TDA: 800 °C– 850 °C IR: < 850 °C (smectite, OH present)
1373	7	6.1–6.6 ka calBP	ENC	Reddish	Yellow/ beige	8	Ox.	Mouth	Coarse and fine	20%	6,1 mm	No result	–	–
2190	7	6.1–6.6 ka calBP	ENC	Reddish	Dark grey	9	Ox.	Mouth	Fine	10%	2,3 mm	Smectite	EIR	IR: < 850 °C (smectite, OH present)
2900	7	6.1–6.6 ka calBP	ENC	Beige/ grey	beige/grey	10	Ox.	Mouth	Coarse and fine (shells)	10%	3,2 mm	Smectite	Beachsand/shell limestone	IR: < 850 °C (smectite, OH present)
4210	7	6.1–6.6 ka calBP	ENC	Reddish	Reddish	8	Ox.	–	Coarse and fine	10%	1,5 mm	Smectite	EIR-microquartz	TDA: 800 °C–850 °C IR: < 850 °C (smectite, OH present)
8870	7	6.1–6.6 ka calBP	ENC	Reddish	Reddish	9	Ox.	–	Coarse and fine	10%	2,9 mm	Smectite	Schist	TDA: 800 °C–850 °C IR: < 850 °C (smectite, OH present)
7228	8	Undated	LN?	Reddish	Reddish	11	Ox.	–	Coarse	10%	5,2 mm	Smectite	Schist	IR: < 850 °C (smectite, OH present)
7920	8	Undated	LN?	Brown/ reddish	Grey	7	Ox.	Mouth	Coarse and fine	20%	2,3 mm	Smectite	–	IR: < 850 °C (smectite, OH present)
8120	8	Undated	LN?	Reddish	Reddish	10	Ox.	Mouth	Coarse	10%	4,6 mm	Smectite	Schist	IR: < 850 °C (smectite, OH present)

(*continued on next page*)

Table 1 (*continued*)

Sample	Layer INES-	Dating	Cultural phase	Clay color	Surface color	Wall thickness (mm)	Surface Oxidized/ reduced	Orientation during firing	Size of inclusions	Amount of inclusions (macrosc.)	max. Grain size	Infrared Spectroscopy	Petrography (group identified)	Firing temperature estimation
13,381	9	Mixed	Neol. (mix.)	Reddish	Reddish	12	Ox.	–	Coarse and fine	20%	3,8 mm	Smectite	–	IR: < 850 °C (smectite, OH present)
13,400	9	Mixed	Neol. (mix.)	Brown/ reddish	Brown/ reddish	6	Ox.*	(Foot)	Coarse	30%	2,1 mm	Smectite	Granite	IR: < 850 °C (smectite, OH present)
13,470	9	Mixed	Neol. (mix.)	Brown/ reddish	Grey	9	Ox.	–	Coarse	40%	2,6 mm	No result	Granite	TDA: 850 °C–900 °C
857	10	Mixed	Sub-recent (mix.)	Brown/ reddish	Grey	13	Ox.	Mouth	Coarse	20%	7,4 mm	No result	EIR	–
1330	10	Mixed	Sub-recent (mix.)	Brown/ reddish	Beige/ brown	11	Ox.	Mouth	Coarse and fine	20%	3,2 mm	No result	EIR-amphibole	TDA: 950 °C–1000 °C
6770	10	Mixed	Sub-recent (mix.)	beige/ grey	Beige/ grey	10	(Ox.)*	Foot	Coarse and fine	10%	2,6 mm	Smectite	Beachsand/shell limestone	IR: < 850 °C (smectite, OH present)
8250	10	Mixed	Sub-recent (mix.)	Brown/ reddish	Brown/ reddish	9	Ox.	Mouth	Coarse and fine	20%	2,0 mm	Smectite	EIR-microquartz	IR: < 850 °C (smectite, OH present)
11,170	10	Mixed	Sub-recent (mix.)	Brown/ reddish	grey	10	Ox.	Mouth	Coarse and fine	20%	2,3 mm	Smectite	EIR-amphibole	IR: < 850 °C (smectite, OH present)
13,159	10	Mixed	Sub-recent (mix.)	brown/ reddish	brown/ reddish	9	Ox.	Mouth	Coarse and fine	20%	5,1 mm	No result	EIR-microquartz	–
13,169	10	Mixed	Sub-recent (mix.)	beige/ grey	beige/grey	10	Red.	–	Coarse and fine	20%	2,1 mm	No result	EIR-amphibole	–
13,310	10	Mixed	Sub-recent (mix.)	beige/ grey	dark grey	10	Ox.	Mouth	Coarse and fine	20%	2,1 mm	No result	EIR-amphibole	–
293	–		Sub-recent (mix.)	beige/ grey	–	11	(Red.)	–	Fine (mica)	20%	2,8 mm	Smectite	EIR-amphibole	IR: < 850 °C (Smectite, OH present)

Table 2
Sample numbers, origins and the results of the IR clay mineral identification.

Region	Source nr.	Number of samples	Coordinates	Clay mineral	Amount of calcite
Ifri n'Etsedda	150329SS1	4	30 S 0542813 UTM 3873828	Kaolonite/ serpentine	High
	150329SS2	5	30 S 0542791 UTM 3873881	Smectite	High
	150329SS3	3	30 S 0542597 UTM 3873932	Smectite	High
	150409SS2	6	30 S 0548467 UTM 3873720	Kaolonite/ serpentine	High
	150409SS3	2	30 S 0546545 UTM 3875899	Smectite	High
	150409SS4	2	30 S 0549833 UTM 3875141	Smectite	High
Ifri Oudadane	150407SS1	3	30 S 0483047 UTM 3896896	Kaolonite/ serpentine	High
	150407SS2	2	30 S 0476875 UTM 3896865	kaolonite/ serpentine	Low
	150407SS3	1	30 S 0471953 UTM 3895300	kaolonite/ serpentine	High
	150407SS4	4	30 S 0494653 UTM 3891677	smectite	Low
	150407SS5	3	30 S 0498163 UTM 3889798	smectite	Low
	150408SS1	1	30 S 485355 UTM 3895588	smectite	High

with a speed of 300 K/h in air by a connecting rod dilatometer.

Based on the combination of IR and TDA, we tried to estimate approximate firing temperatures for some of the samples. Criteria therefore were: If smectite was identified in the IR spectra, we suggested a firing temperature < 850 °C, because the amorphisation process of smectite clays is expected to be complete above these temperatures (Grim, 1962). Although different smectite clays amorphise at lower temperatures and the process is in all cases progressive, differential thermal analyses of most smectite endmembers show a high temperature peak between 800 °C and 900 °C (Odom, 1984), suggesting that if no smectite clay is detected any more, the ceramic's firing temperature was most likely above 850 °C.

3. Results

3.1. Macroscopic description of archaeological samples

Wall thicknesses of the pottery samples range between 6 and 13 mm and show a normal distribution. The outer surface of 25 samples is clearly oxidized. For two more badly preserved samples oxidation of the outer surface can be assumed. Three of these 27 samples preserve an oxidized surface at the inside of the sherd as well. A reduced surface was recognized for three samples although the surface of one of them shows a poor state of preservation making the attribution less certain. Macroscopically, the amount of inclusions appears to vary between ~5 and ~40%, with most sherds containing ~10 or ~20% (22 samples). Maximum grain sizes vary between 1.1 and 7.4 mm. These results are summarized in Table 1.

3.2. Petrographic analysis of temper elements

Petrographic analysis reveals a high variability of temper components and types of inclusions. The samples contain quartz, plagioclase, alkali-feldspar, mica, pyroxene, calcite, amphiboles, but also rock-fragments of schist, plutonic and extrusive igneous rocks (EIR). A few samples contain grog (or chamotte, i.e. previously fired ceramic that was ground and included in the pottery paste), organic materials (e.g. cereal grains) and shell limestone. The results of the qualitative and quantitative analysis are summarized in Table 3.

Based on the nature of rock-fragment inclusions the 30 samples can be subdivided into 8 groups (Fig. 4). Three of these groups show EIR as main rock fragments. They can be differentiated by considering the other inclusions. [1] EIR along with pyroxenes: 3 samples; [2] EIR along with amphiboles: 5 samples; [3] EIR along with fragments of micro quartz containing rocks: 5 samples; [4] granite fragments, partially weathered and sericitized: 3 samples [5] fragments of dolerite or gabbro along with fragments of schist, fibrous quartz aggregates and unidentified spherulites: 2 samples; [6] schist as dominant type of inclusion: 4 samples; [7] Organic limestone: 2 samples (while one of these samples contains mainly shells and shellfish debris the other one mainly contains foraminifera debris); [8] different types of grog and a few shell inclusions: 1 sample.

Organic inclusions are present in sample 293 (group [2]) and in sample 1230 (group [6]). The samples of group [3], with EIR and fragments of micro quartz, also show micritic calcite inclusions (with the exception of sample 9650). A very low amount of amphiboles was found in samples 2900, 8250 and 10,429.

3.3. IR-spectroscopy of archaeological samples

We identified the group of clay minerals used for 17 of the sherds on the basis of the presence of specific OH absorption bands in their IR spectra (Fig. 5). All these 17 sherds are made from smectite clay, as documented by a single OH band near 3620 cm^{-1}. No clay could be determined in the 13 other samples because all hydroxyl was lost during firing.

13 samples yielded a more or less intense broad CO_3 band near 1430 cm^{-1} associated with sharp bands at 877 cm^{-1} and 713 cm^{-1}, documenting the presence of calcite in the samples. The calcite of group [7] is explained by the presence of shells and shellfish debris. A few shell fragments were recognized in sample 1420 of group 8 as well. The calcite in 4 samples of group [3] (except sample 9650) can be explained by inclusions of micrite. In contrast, calcite could not be identified in the last five samples. While secondary calcite was found in pores of samples 857 and 8870, the origin of the calcite in samples 293, 2190 and 4990 remain unclear.

3.4. IR spectroscopy of geological samples

Based on their IR spectra, geological samples can be subdivided into two groups: kaolinite/serpentine clays can be identified on the basis of two OH bands near 3700 cm^{-1} and 3620 cm^{-1} (Fig. 6). Five of our samples contain kaolinite/serpentine clay. A single OH stretching band near 3620 cm^{-1} in combination with a H_2O bending band near 1639 indicates smectite clay. We identified smectite in 7 samples (Fig. 7). Calcite was identified in all samples. Both groups can be further differentiated into groups of high and low amounts of calcite (Fig. 6). Table 2 summarizes the results obtained from the geological samples.

3.5. Thermodilatometric analysis (TDA)

Three of the 7 samples (1230, 4210 and 8870) analyzed by TDA shrank from 800 to 850 °C upwards (Fig. 8A). Their lost volume ranges from 2 to 3.5%. In samples 5080 and 13,470 a drop of 0.5 vol% is measured between 850 and 900 °C (Fig. 8B). The last two samples 1330 and 9650 started to shrink between 950 and 1000 °C, losing 0.5 and 1% of their volume (Fig. 8C). Fig. 8 shows the dilatometer curves and Table 1 summarizes the temperatures at which the samples started to shrink.

Table 3
Results of the qualitative and quantitative analysis by polarizing microscope (in %). For 5 samples was no quantitative analysis possible, so the inclusion types present are marked with "X".

Sample	Layer INES-	EIR	Granite	Dolerite/ gabbro	Schist	Organic limestone	Grog	Organic inclusion	Pyroxene	Mica	Amphibole	Micro quartz	Fibrous quartz	Spherulites	Micritic calcite	Quartz	Plagio-clase	Alkali-feldspar
4465	5	Not performed																
6050	5			17.2						1,0						14.6	3.5	63.6
10,420	5					4.0	90.6				< 0.1					5.3		
11,190	5	2.6		10.3	5.2				< 0.1			1.9	7.1	1.9		1.3	5.2	64.5
12,070	5	7.1							< 0.1	1.2			1.2		7.1	73.8	9.5	
4550	6	5.6		12.1	4.5				0.5				10.6	1.0		1.5	5.6	58.6
4990	6	19.4							1.7	0.4						2.9	4.6	70.9
5080	6	Not performed																
9650	6	6.3							2.5	0.6		8.2					12.0	70.3
1230	7				49.0			3.4				3.4				44.2		
1373	7	Not performed																
2190	7	43.5							4.8	< 0.1						6.5	21.0	24.2
2900	7					4.6			0.5	0.5	0.5	2.0			21.9	2.6	11.2	56.1
4210	7	5.3					37.3		0.7			1.3			0.7	45.3	8.0	1.3
8870	7				X								X		X	X		
7228	8				32.6		4.3					< 0.1				63.1		
7920	8	Not performed																
8120	8				66.3							3.6				30.1		
13,381	9	Not performed																
13,400	9		16.5													5.7	6.3	71.5
13,470	9		21.4							1.1						8.0	7.5	62.0
857	10	24.4							3.6							1.4	10.1	60.4
1330	10	X								X	X					X	X	X
6770	10					X									X			
8250	**10**	7.2			7.2				2.4	1.6	0.8	12.8			4.0	53.6	6.4	4.0
11,170	10	X								X	X					X	X	X
13,159	10	13.2							2.6			2.6			1.3	14.5	15.8	50.0
13,169	10	X								X	X					X	X	X
13,310	10	29.3							0.7	2.1	13.3					4.9	7.7	42.0
293	–	17.1						5.3	5.3	2.6	9.8					2.0	19.7	38.2

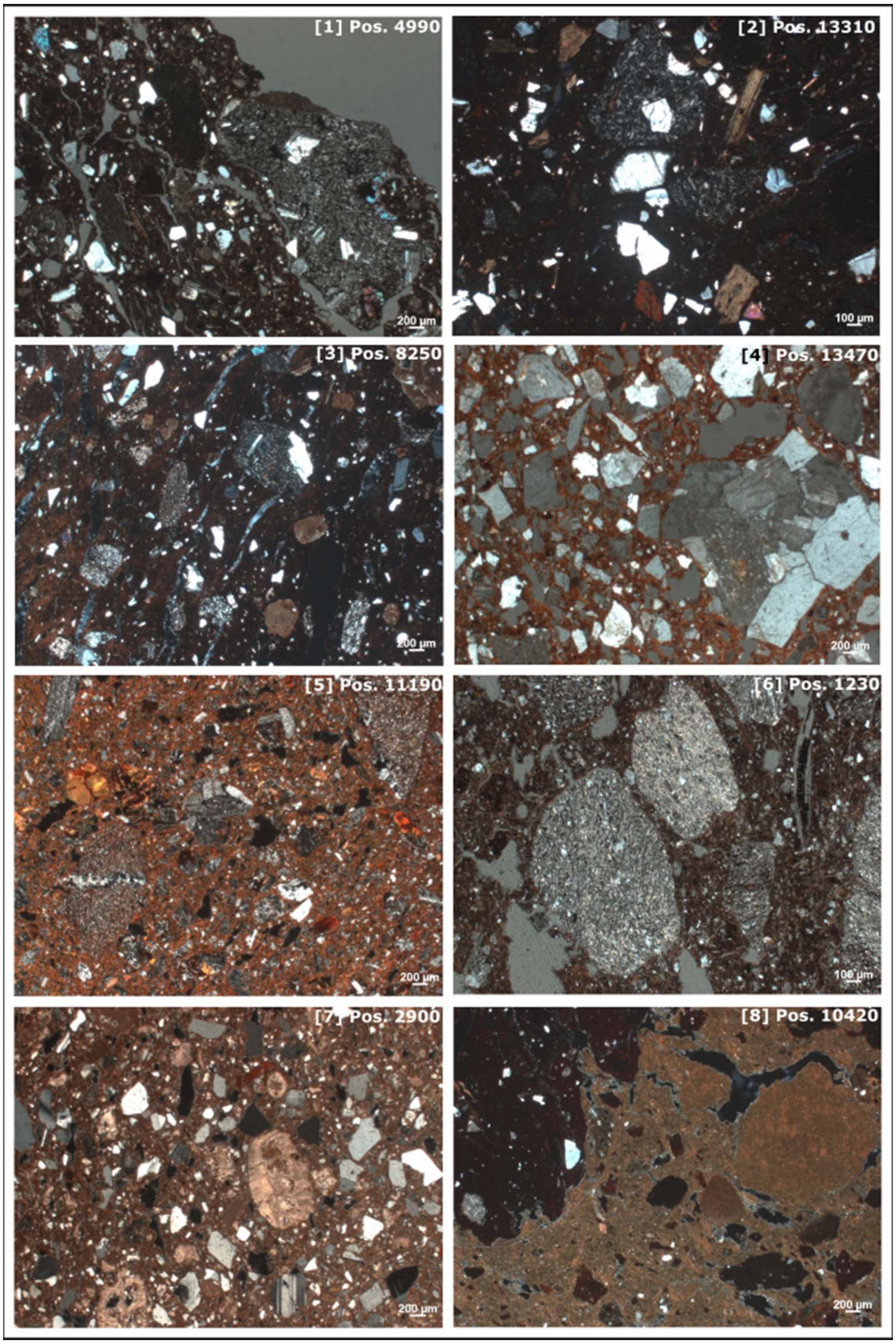

Fig. 4. Examples of the groups identified by polarizing microscope: [1] EIR; [2] EIR with amphiboles; [3] EIR with micro quartz containing rocks; [4] granite; [5] dolerite or gabbro with schist, fibrous quartz aggregates and unidentified spherulites; [6] schist [7] Organic limestone [8] grog.

4. Discussion

4.1. Raw materials used for the Ifri n'Etsedda pottery

The results suggest that only smectite clay was used for pottery production in Ifri n'Etsedda. However, there are different types of inclusions in the clay. Two explanations are possible: (i) different smectite clay sources, containing different mineral impurities were used for pottery production. (ii) Temper may have been intentionally added to the clay. Because the shape of the inclusions differ in various stages of rounded and sharp, it is more likely that the inclusions occur naturally in the clay. The intentional adding of temper can only be determined for grog, shell limestone and organic materials, which all usually do not occur naturally in clay. Although small amounts can be added accidental during the sampling or manufacturing process. Nevertheless, following traditions in the use of different clay deposits and temper can be supposed (Fig. 9): clay with granite as well as dolerite/gabbro was determined in samples from layers INES-5, -6 and -9 and can therefore

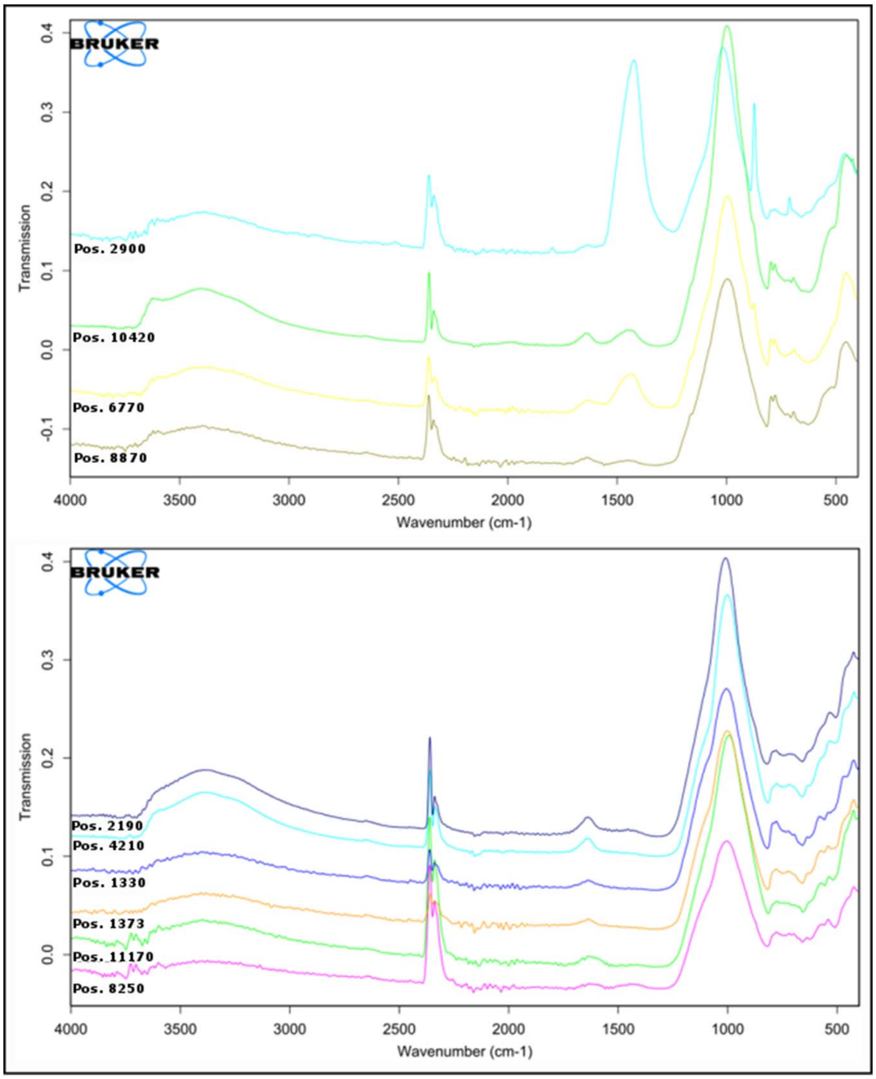

Fig. 5. Examples of the IR spectra of the artifact samples.

be considered as a clay tradition of the Early Neolithic A. INES-9 is most probably a disturbed layer, excavated in 2013 and 2015 from above the site's Epipaleolithic layers. It has yielded Neolithic artifacts. However, ^{14}C dates obtained from cereal grains in the layer show ages of only several hundred years B.P., revealing the sediments to be reworked (Linstädter et al., 2016). Hence, the layer could possibly be Early Neolithic A or at least contain ENA material.

Sherds with EIR inclusions were found in layers INES-6, -7 and -10, possibly revealing a separate Early Neolithic B and C clay tradition. Like INES-9, layer INES-10 may be reworked, containing sediments from the ENB and C. The results suggest that the use of clay deposits with schist inclusions was introduced during the Early Neolithic C and Late Neolithic. Although small amounts of schist were identified in samples from INES-5 and -6, schistous rock fragments are the main inclusions in samples coming from INES-7 and -8. If one considers a sort of mixing of archaeological material due to the intensive occupation of the site, clay with schist inclusions could also be considered as a Late Neolithic peculiarity. Shell-, organic materials- and grog-temper occur throughout different time periods and have therefore no importance for chronological interpretation. Regarding the small sample sized, these result should be seen as tendencies that have to be proven by further studies.

Comparing geological samples and artifacts it is obvious that only smectite clay deposits are potential raw material sources (c.f. Table 2). Because of the absence of large amounts of calcite in all artifacts, geological samples with a large amount of calcite can also be excluded as possible raw material sources. This hypothesis only works if no preparation of the clay was undertaken prior to making the pottery paste (this would be the case if sources 150329SS2, 150329SS3, 150408SS1, 150409SS3 or 150409SS4 had been used). The normally large effort to remove inclusions from clay however, and the high amounts of temper and inclusions in the samples, makes such a preparation unlikely. In this light, our results suggest that only two of the clay sampled deposits are potential raw material sources of the Ifri n'Etsedda pottery: clay source numbers 150407SS4 and 150407SS5, both being sources in the area of the lower Oued Kert approximately 50 km far away. This does not mean that the Ifri n'Etsedda pottery's clay raw material originated from these two sources as we did not exhaustively survey the whole area for clay deposits. The only secure assertion stemming from our sampling strategy is that no local source around Ifri n'Etsedda could have been used for the production of the site's pottery. Thus, at least the clay raw material was not strictly local. The variability of different temper-materials and inclusions in the 30 sherds may indicate that the raw materials, or the finished pottery items themselves, do not originate from a single place but rather from different sites.

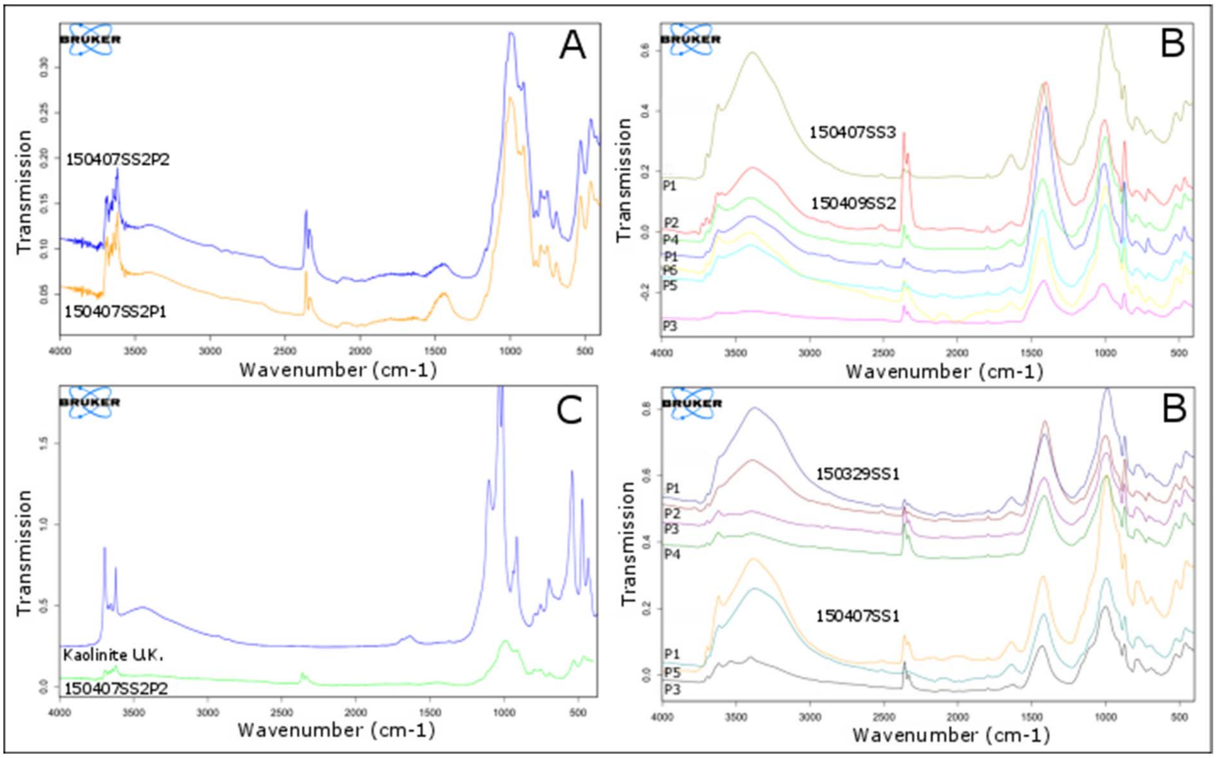

Fig. 6. IR Spectra of the geological samples containing kaolinite-serpentine clay. A: low amount of calcite; B: high amount of calcite; C: comparison with a kaolinite spectra of our database.

4.2. Fabrication of the Ifri n'Etsedda pottery

We assume an oxidizing atmosphere for most of the sherds. Only three samples (293, 12,070 and 13,169) may be an exception to the generalized use of an oxidizing atmosphere. In case of sample 293 a reducing atmosphere may have been used but no clear interpretation can be made due to the poor preservation of the sherd's surfaces. An upside-down orientation during the firing process for 18 samples seems to be likely because the oxidation zones are only present on the outside of these sherds. During such firing, the pottery mouth is placed upside-down during firing so that the inside of the pot is not exposed to air (Velde and Druc, 1999). While two other samples were placed on their foot (c.f. Table 1), determining the orientation of the remaining 10 samples is impossible because the progression of the oxidation/reduction-reaction was complete throughout the entire sherds, leaving behind a uniform aspect (for detailed description of the process see (Velde and Druc, 1999)).

The results also suggest open firing in a pit. Surface firing is possible

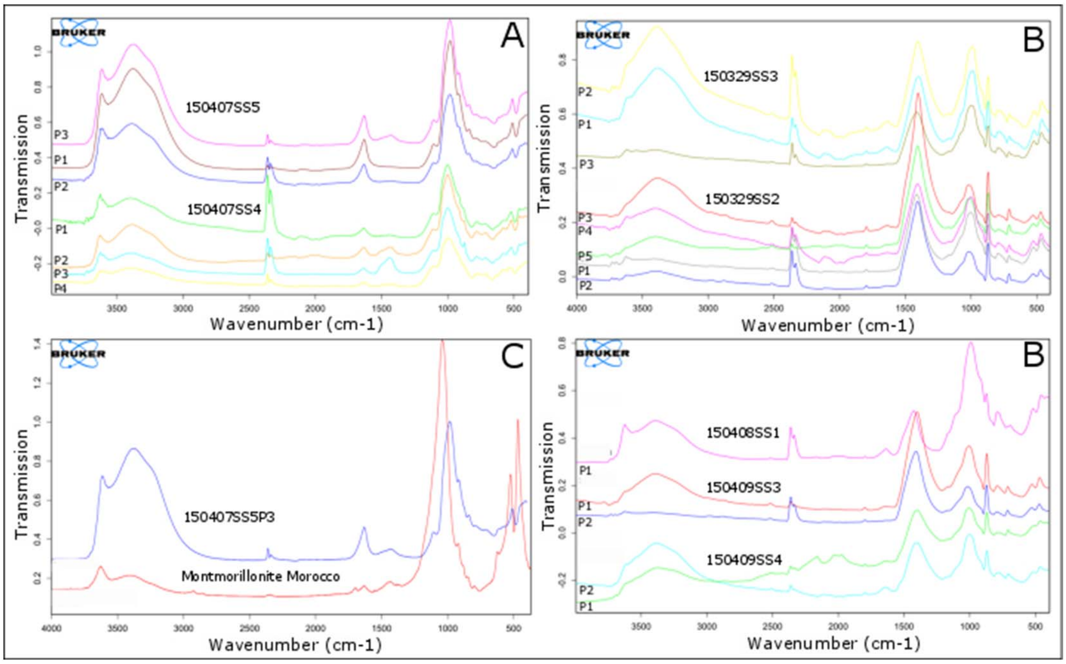

Fig. 7. IR Spectra of the geological samples containing smectite clay. A: low amount of calcite; B: high amount of calcite; C: comparison with a kaolinite spectra of our database.

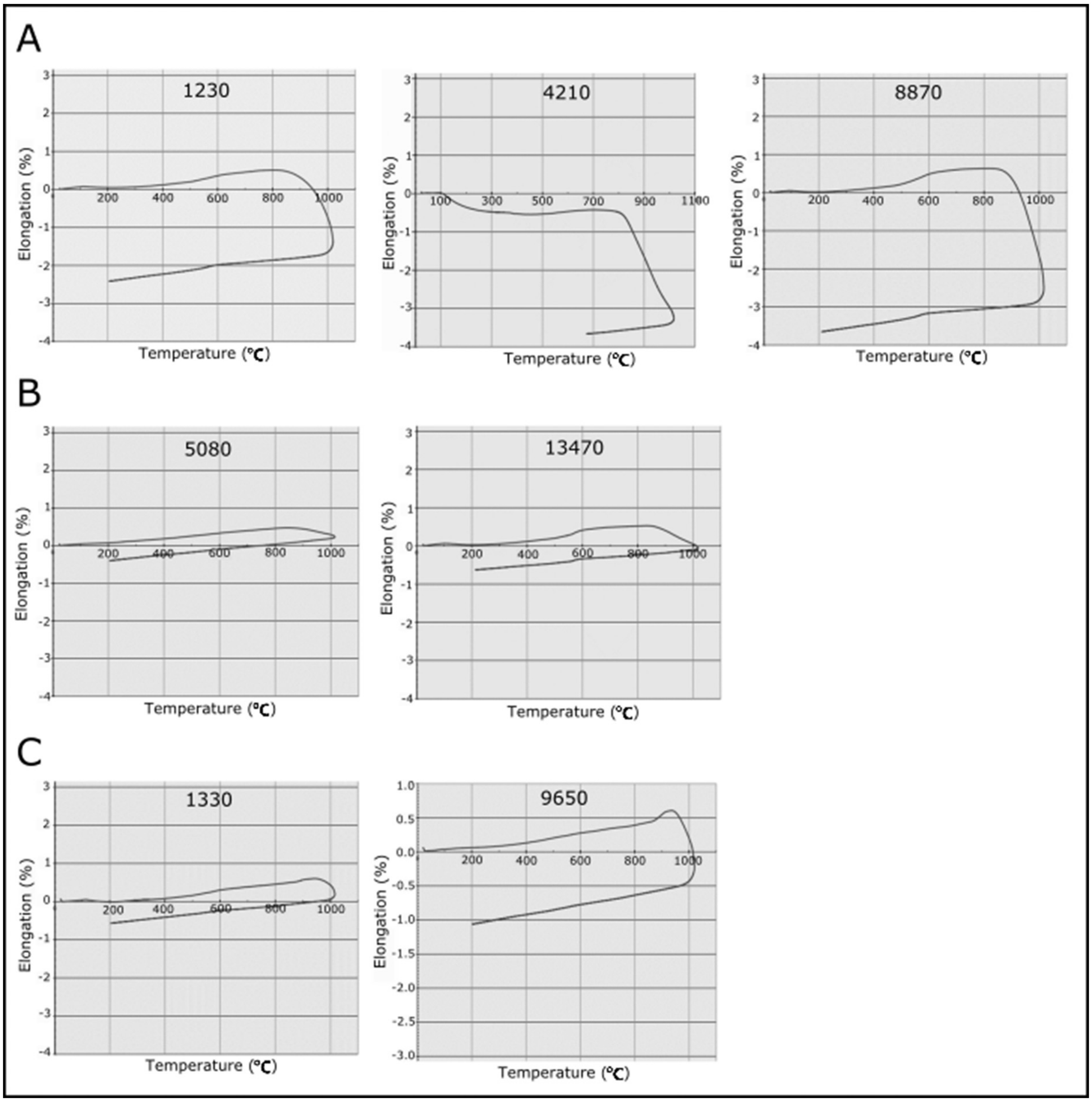

Fig. 8. Dilatometer curves of the artifact samples (Deutsches Institut für Feuerfest und Keramik GmbH). A: Samples shrank from 800 to 850 °C upwards with a drop between 2 and 3.5 vol%. B: Samples shrank from 850 to 900 °C with a drop of 0.5 vol%. C: Samples shrank from 950 to 1000 °C upwards with a drop of 0.5 to 1 vol%.

as well, but normally reaches only temperatures between 600 and 800 °C (occasionally up to 900 °C). In contrast, temperatures up to 900 °C can be reached in such open pits (Rice, 2007; Velde and Druc, 1999). Burning temperatures determined by thermodilatometry range from 800 to 1000 °C. Combining the results obtained by TDA and IR spectroscopy, we tentatively estimate the firing temperatures of the Ifri n'Etsedda pottery as: samples in which no clay minerals could be determined by IR may were most likely fired to above 850 °C because the amorphization process of the trioctahedral smectite clays (or any other potentially used natural clay mineral) is complete above these temperatures (Grim, 1962; Odom, 1984). However, it is also possible that another type of clay with lower amorphisation temperature (e.g. Kaolinite, around 550 °C) was used (Noll, 1991). If such was the case, the firing temperature may have been lower than 850 °C. Thus, firing temperatures of the Ifri n'Etsedda sherds that could be analyzed in this way are likely to range from ~750 °C to ~1000 °C (compare Table 1). An analysis of the modelling techniques was not assessed yet, but will be suggested for future studies.

5. Conclusion

Because mineralogical clay and temper analyses of Neolithic pottery of Northwest Africa are not a standard procedure yet, our study may be of importance for further works in the region. Even though its sample size is limited, the study provides prospects and some interesting results that we interpreted cautiously.

We identified in total 8 different groups of temper and types of inclusions in this study. The combined petrographic and macroscopic results indicate that macroscopic characteristics may be different from the petrographic groups. This shows how important archaeometric analysis can be to complement stylistic pottery analysis. This is further highlighted by the fact that it was possible to identify trends in the use of different types of clay and clay deposits. It seems that plutonic rock is more typical for Early Neolithic A pottery, while EIR becomes more frequent towards the end of the Early Neolithic. Clay with schist inclusions potentially represents the dominant part of Late Neolithic raw materials. The fact that samples tempered with plutonic rock fragments

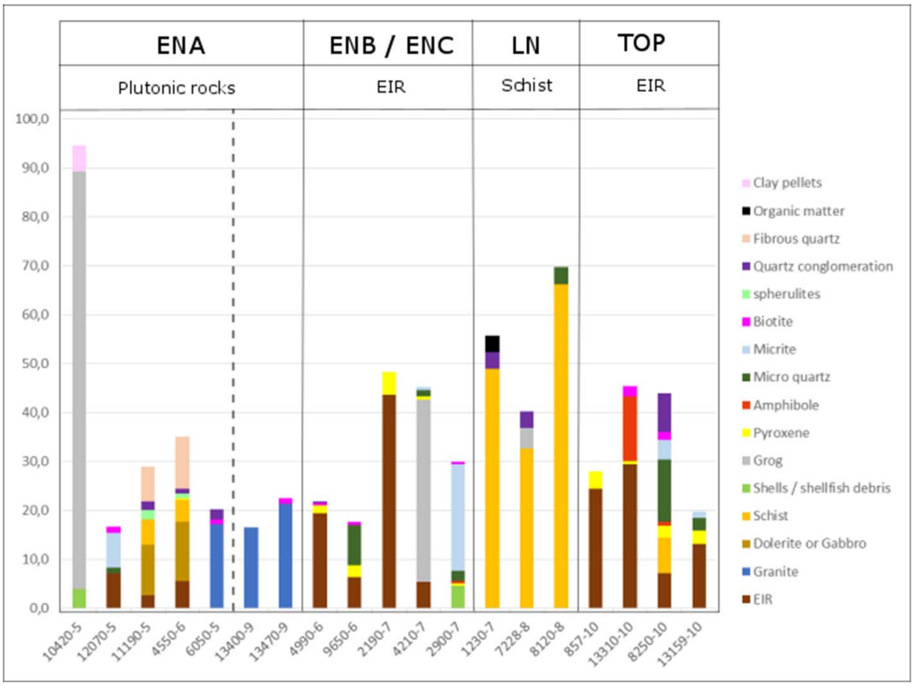

Fig. 9. Image of the clay and temper trends. Only components are displayed, which were used for differentiation or which are of significance (e.g. organic matter). Monocrystalline quartz, plagioclase and feldspar are excluded for a better illustration. The samples are sorted by stratigraphic units from INES 3 to INES 10. The INES 9 deposit could not be dated by radiocarbon method. However all INES 9 samples provide a very specific granite temper. This temper otherwise appears only in ENA context (INES 5). Therefore the INES 9 samples are arranged here close to the other ENA samples (separated by a dashed line) in order to call attention to this fact.

which come from undated and not yet understood layers may be used as a criterion that these layers belong to the Early Neolithic occupation (ENA) of the site. Mineralogical studies may therefore also help to better understand parts of the disturbed stratigraphy of Ifri n'Etsedda.

Most of the Ifri n'Etsedda pottery was fired in an oxidizing atmosphere and placed upside down in a pit. The firing temperature, as determined by thermodilatrometric analysis and petrographic proxies were ranging from ~800 °C to ~1000 °C. Only smectite clay was identified in our samples. However, the clay minerals used to produce 13 of the samples could not be determined because the samples were fired above the temperature at which clay amorphisation is completed.

At that time, no local clay source seems to have been used for the pottery of Ifri n'Etsedda. With regard to the small artifact samples size as well as the incomplete raw material survey, further studies are needed to shed light on the Ifri n'Etsedda pottery.

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