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#### ORIGINAL ARTICLE

# An integrated approach to reconstruct the role of the heat treatment within the reduction sequence of chert artefacts: The case of the early Neolithic site of Lugo di Grezzana (north-eastern Italy)

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#### Abstract

The lithic assemblage from the well-known site of Lugo di Grezzana (Italy) attributable to the Fiorano group (5,300-4,900 BC cal) is analysed to shed new insights on the early Neolithic lithic technology in North-Eastern Italy. Techno-typological data are discussed allowing detailed reconstruction of the reduction sequence, thanks also to several refittings. A complex laminar production with several technical objectives has been identified. Whereas clues for the heat treatment of lithic materials have been identified, we verified it by a combined experimental approach. In the first place, we used density measurements, which already proved to be useful for prescreening analysis, to detect potentially heated artefacts. Afterward, we performed Fourier Transform Infrared (FT-IR) spectroscopy tests to assess the actual (de)hydration condition of the siliceous materials. Reflectance spectra have been acquired, analysing any item and avoiding any constraint related to both morphology and thickness of the sample. The archaeometric results, combined with the reduction sequence reconstruction, provide new indications on the technical capabilities and knowledge of the human groups that inhabited the region during the early Neolithic.

#### **KEYWORDS**

density measurements, early Neolithic, Fiorano group, lithic technology, reduction sequence, reflectance FT-IR spectroscopy

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#### INTRODUCTION

The heat treatment of siliceous raw materials is one of the first evidence of pyrotechnology in human civilization (Brown et al., 2009; Tixier & Inizan, 2000). The controlled heating of rocks is a technological practice identified in several archaeological contexts since the Pleistocene all over the world (see details in Monik et al., 2021; Schmidt, 2019). The oldest clear evidence of heat treatment in Europe comes from Upper Palaeolithic sites (Schmidt & Morala, 2018), although it is during the Neolithic that this technique becomes more important (Binder, 1984; Milot et al., 2017). This seems to be related to the diffusion of laminar production, involving the pressure knapping technique, tentatively linked to the emergence of expert flint knappers and/or to the development of commercial routes (Binder, 1984; Léa, 2005; Wadley, 2013).

The presence of heat-treated tools in archaeological lithic assemblages can be preliminarily assessed by eye inspection by observing different macroscopic modifications, such as lustre and colour shades (Inizan et al., 1995). Several methods have been used to standardize and strengthen visual investigation and quantify the observed alterations (Fiers et al., 2021). However, rock variability and possible surface alterations render uncertain the result of this simple macroscopic approach. Moreover, autoptic data do not allow the ability to clearly quantify the heat treatment parameters, such as the precise exposure temperatures and duration, particularly if specific experimental data for each geological source are missing. Therefore, more reliable methods, based on archaeometric techniques, have been developed to characterize structural modifications of rocks due to the intentional heating process. X-ray diffraction (XRD) as well as X-ray fluorescence (XRF), although used in this research field (Domański et al., 2009; Domański & Webb, 1992), turned out to be poorly effective owing to their limited sensitivity to the actual structural changes induced by the heat treatments and to the lithotypes involved in the analysis (Cochrane et al., 2012). Mechanical tests proved to be potentially useful; however, because they can be partially destructive, can be applied with some difficulty to a large number of specimens from archaeological collections (Domański & Webb, 1992; Moník & Hadraba, 2016; Purdy & Brooks, 1971). Lately, roughness measurements were used to characterize successfully and detect the heat treatment on lithic artefacts. However, requiring relatively sophisticated equipment, they are not always available in archaeological laboratories (Murray et al., 2020; Schmidt, 2019). Dating techniques such as thermoluminescence and electron spin resonance might be useful to detect heat treatments. However, both methods exhibit some limitations in detecting low-temperature heat treatments (Brown et al., 2009; Richter et al., 2011; Valladas, 1983). Raman spectroscopy is relatively effective in detecting heat treatments (Agam et al., 2020), although fluorescence effects may render the relevant results challenging to interpret or to acquire, in the first place (Wadley et al., 2017). A major contribution to the evaluation of the heat treatment of siliceous lithic materials comes from Fourier Transform Infra-red (FT-IR) spectroscopy (Schmidt et al., 2011, 2012; Schmidt, Léa, et al., 2013; Schmidt, Nash, et al., 2017; Schmidt, Porraz, et al., 2013; Schmidt, Spinelli Sanchez, & Kind, 2017; Weiner et al., 2015). The approach can be profitably strengthened when combined with other measurements, like scanning electron microscopy (SEM) observations (Santaniello et al., 2016), density (Santaniello et al. 2021), and roughness tests (Bachellerie & Schmidt, 2022). The FT-IR analyses are quite fast and easy to be conducted nondestructively. Some limitations may derive from the size, morphology, and conservation state of the analysed samples. FT-IR analyses are generally performed in transmission on sample regions whose thickness ranges from about 0.5 up to 4 mm at most. Moreover, sample surfaces should not present any significant alteration, such as patination, which may otherwise affect the analysis results. Finally, a possible restriction to the applicability of this technique is associated with the sample size that should fit into the FT-IR instrument sample chamber, which may result to be relatively small, thus preventing to conduct tests on large samples.

Following archaeological data as well as ethnographic records (Akerman, 1979; Akerman et al., 2002), heat treatment is used to modify the original colour of siliceous rocks (Hermo, 2008) and to produce lithic tools with sharper cutting edges (Key et al., 2021). However, the role of the heat treat-

ment is generally related to the improvement of the rocks' knapping quality and to the amelioration of the lithics production process (see among others Crabtree & Butler, 1964; Schmidt et al., 2019). Despite of this strict connection between the heat treatment and the production process of the stone tools, the timing of the heat treatment within the reduction sequence has been rarely addressed, even in view of the intrinsic difficulty of assessing this matter (Binder, 1984). In order to highlight this aspect, we analysed the archaeological assemblage from Lugo di Grezzana (Verona, Italy), dated to the early Neolithic and attributed to the "Fiorano" group. Technological data and several refittings helped to reconstruct the reduction sequence, revealing the presence of possible heat-treated stone tools. We selected artefacts from different phases of the reduction sequence to verify the actual presence of thermal treatment, through a novel protocol allowing us to analyse any lithic item, with no restrictions as concerns sample shape and size. The protocol includes both density measurements and FT-IR analyses in reflection mode. The density tests are particularly valuable for prescreening analyses, whereas reflection FT-IR spectroscopy allows analysing each item obtaining results comparable to the transmission mode, used so far. The combination of technological and archaeometric results provides a complete picture on which basis it is possible to assess when the heat treatment has been performed within the reduction sequence, thus revealing new aspects of the operational chain attested in the site.

## MATERIALS AND METHODS

## The site of Lugo di Grezzana

Lugo di Grezzana archaeological site (45°33'58"N - 10°59'40"E) is located in the Valpantena valley near the Progno river (Verona province, Italy), at about 300 m asl (Figure 1.1). The archaeological deposit was discovered in 1990 by F. Zanini and G. Chelidonio, during the archaeological prospection related to some earthworks carried out for the construction of industrial premises. The archaeological excavation started in 1991 under the direction of the Superintendence of Veneto and supported since 1996 by the University of Trento (the former Laboratorio Bagolini, currently named LaBAAF). The site has been divided into 16 sectors, which were excavated during several campaigns until 2005 (Cavulli, 2008; Cavulli & Pedrotti, 2003; Salzani, 1993). Since then, the University of Trento is carrying out research activities to interpret the archaeological artefacts. The micromorphological analyses (Angelucci, 2003) show that the site formation is the result of the succession of anthropic and environmental events. Located on a late Pleistocene alluvial deposit, the site has been inhabited since 5,400-5,060 BCE cal.; this occupational phase is characterized by several structures with ceramic elements attributable to the Fiorano group (Cavulli, 2008; Costa et al., 2015). After a period of partial abandonment, the site was reoccupied at the beginning of the V millennium (4,900-4,720 BC cal.). In this phase, in addition to Fiorano and Vhò ceramic remains, some evidence attributable to the first stage of the Square Mouthed Pottery has been found (Pedrotti et al., 2015). Archaeobotanical (Rottoli et al., 2015) and archaeozoological (Maccarinelli et al., 2015) analyses demonstrate the presence of a complex agropastoral economy. Remains of three different species of cereal were found, and the breeding is clearly attested by the presence of cattle as well as sheep, goats, and pigs remains, whereas the hunting activity is marginally documented. Lugo di Grezzana represents something unique in the Northern Italy scenario because of the finding of thousands of lithic remains. This suggests that the site played an important role as concerns the chert 'circulation' in the early Neolithic framework (Pedrotti & Salzani, 2010; Santaniello et al., 2015), also considering the site location, standing close to the well-known chert outcrops of the Lessini mountains (maiolica or Biancone, Scaglia Variegata, Scaglia Rossa), widely exploited during the Neolithic in northern Italy and frequently associated with the use of pressure knapping technique (Pessina, 2000: 86).



**FIGURE 1** (1) Localization of Lugo di Grezzana site (Verona) modified by d-maps.com; (2) localization of sector XIII (in red position of the ES 116/03); (3) ES 116/03 and position of the stratigraphic units 109/03 and 257/03 were have been found the artefacts analysed in this study (red– chert; Blu– pottery).

## The analysed lithic assemblage

During the excavation of the central-eastern area of sector XIII (Figure 1.2), a pit, named ES 116/03 (Figure 1.3), was discovered. The pit is filled with alternate anthropic and natural layers, the latter of which has been directly dated at 5207–5093 BCE cal. (Pedrotti et al., 2015: 100). The pit was 13 m long and 5.5 m large with NW–SE orientation. To the South-East the pit was deep about 15 cm, and it deepened down to about 1.5 m to the North-West. According to the morphology and considering its position on the lower part of the slope, the pit was probably used as a water tank, at least during the early stages of the site inhabitation. Since the earliest occupation period of the site, the deepest part at the NW part of the pit was partly filled with several anthropic layers (see details Pedrotti et al., 2015); successively, the bottom of the pit was regularized thanks to an intentional deposition of clay, which formed a flat surface. Above this surface, four different concentrations of lithics were recognized

and collected at different stratigraphic units (hereafter SU): SU 109/03, 248/03, 257/03, and 258/03. Among them, the units containing the largest number of lithic artefacts (SU 109/03 and 257/03) have been selected for this research, also considering the field documentation that suggested already the possible presence of several refittings. Totally, 1,650 remains have been technologically analysed (Table S1), and then ten items made of maiolica chert have been sampled in order to check for their heat treatment macroscopic evidence (Table S2).

## **Technological data**

The present investigation involved the collection of the following data: (a) raw materials provenance by means of a Leica EZ4D stereo microscope (system magnification: 10x to 35x), Hirox RH-2000 digital microscope, and comparing our reference collection (Barbieri et al., 2013) and literature data (Longo et al., 2004; Wierer & Bertola, 2016); (b) blank type determination (core, blade, flake, fragment, *déchet*, other); (c) dimensions of the artefacts (length, width, thickness); (d) technological patterns, such as type of butt, lateral profiles, and core striking platform attributes (following Inizan et al., 1995); (e) presence and localization of technical features (such as cortex, *débordant* side, fractures, retouch, etc.); (f) direction and sequence of scars on blanks/cores (following Boëda, 1994). After the data collection, the refittings have been performed by means of pressure-sensitive adhesive and removable adhesive tape. We tried to refit pieces considering each stratigraphic unit separately and after attempting conjunctions between them.

#### **Density measurements**

For the density measurements, we used as reference the values for the maiolica chert, discussed already in Santaniello et al., 2021. Geological samples were collected from six outcrops of maiolica (MN1, MN4, Veja1, Veja4, CIM9, S.MIC2, see relevant geological description in Barbieri et al., 2013) and then prepared by knapping. After, the samples have been thoroughly cleaned by immersing them into acetone for 2 min, rinsed with ethanol, and then dried up for 1 h in a muffle furnace at 50°C. Subsequently, every single specimen has been weighted in the air (W<sub>a</sub>). Then the samples have been immersed in distilled water for 24h to allow a complete filling of the open pores and cracks, before being reweighted with the scale plate immersed in distilled water (W<sub>w</sub>). The following equation has been used to evaluate the relevant density ( $\rho$ ):

$$\rho = \frac{Wa}{Wa - Ww} \rho w$$

Then the geological samples have been heat treated for 2.5 h in static air, at 250°C and 400°C in isothermal conditions. In all cases, furnace cooling back to room temperature followed. The density values of the heated samples have been measured according to the same procedure described above. Density decreases with increasing heating temperature for all the geological samples (see Santaniello et al., 2021: 119). Consequently, mean values with standard deviation for each temperature have been measured (Table S3) for a comparison with the density values of archaeological samples here considered.

Ten archaeological items made of maiolica from different phases of the reduction sequence (compare Technological results) have been selected. They have been prepared and analysed following the same protocol described for the geological ones.

## **FT-IR** analyses

For a reliable estimation of the heating temperatures employed in the manufacturing process of the archaeological samples, we carried out on them FT-IR analyses, using a Bruker Alpha II instrument,



**FIGURE 2** (1) reflectance module with the collimator used for the test; (2) example of the analysed sample positioning during analyses; (3) examples of FT-IR spectra in reflectance mode at room temperature (RT) and heat treated at 400°C.

equipped with a module for reflectance tests. This instrument set up allows contactless and nondestructive analyses, indeed widely used in cultural heritage and mineralogical research fields (Rosi et al., 2019). In order to set temperature reference values, we choose chert from four geological outcrops of maiolica (CIM9, MN1, MN4, Veja1, see localization in Santaniello et al., 2021). We knapped the original blocks from each outcrop to obtain smaller samples suitable for being tested in the following conditions: one in the pristine state (room temperature, untreated condition) and two after heat treatments, at 250°C and 400°C for 2.5 h in static air. For the heat treatments, we used a furnace FM-74 with a temperature ramp of 10°C/min. The samples were heated to the set temperatures, and after 2.5 h the furnace was turned off and cooled down. Prior to FT-IR tests, the samples have been conditioned, as concern their hydration, following a well-established methodology, used also for FTIR transmission tests (Santaniello et al., 2016; Schmidt, Léa, et al., 2013) with a few adjustments related to the use of the spectroscope in reflection mode. In particular, all samples have been cleaned with ethanol and dried for 1 h in a muffle furnace at 50°C and then immersed in deionized water for at least 48 h. Later on, the samples have been extracted from the water and dried with a towel before proceeding with IR tests. To perform the analyses, we positioned the ventral side of the blanks in front of the collimator (3 mm diam.) of the FT-IR reflection module (Figure 2.1, 2.2) and collected three measurements for each sample to account for possible inhomogeneities of the natural material. FT-IR data have been acquired with a resolution of 8 cm<sup>-1</sup> over a wave number range from 5,500 to 4,000 cm<sup>-1</sup>, and 256 repetitions for each data set. This spectral range is interesting for the present study because it includes the wave numbers of the main Si-OH vibrational modes and as such is suitable to detect heat treatments of the lithic materials (Figure 2.3). In particular, the variation in the intensity ratio of the lines at 4545 cm<sup>-1</sup> and 4,469 cm<sup>-1</sup> has proved to be an effective proxy for heat treatments possibly carried out during stone tools manufacturing (Schmidt et al., 2011, 2012).

The spectra have been transformed in absorbance using the Bruker software OPUS 8.1. To calculate the ratio between the intensities of the absorptions lines at 4545 cm<sup>-1</sup> and 4,469 cm<sup>-1</sup>, a straight baseline was drawn between the two lowest points on either side of the absorption bands. Then, we calculate the mean value and the standard deviation of the three measurements, each one on a different spot of the same geological rock samples (Table S4), in order to have reliable reference standards for the archaeological artefacts.

The same protocol previously described for geological samples has been used to prepare the archaeological artefacts that were used also for density measurements.

## RESULTS

#### **Technological results**

Detailed technological data and the refitting analysis have been recently published (Santaniello et al., 2020), here we revise the results to contextualise the archaeometric data. Flakes and knapping waste, including fragments and *déchets*, are abundant, whereas blades and cores are rare (compare Table S1). The investigated lithics are made from raw materials coming from maiolica and Scaglia Variegata, two local formations providing good quality cherts. These formations are well attested in the geological succession of the region (see among others Barbieri et al., 2013; Castellarin et al., 1968): Maiolica (Tithonian inf. and Aptian inf.) has very thin micritic limestones with variable shades of colour (grey, white) with radiolarites and tinttinids. Scaglia Variegata (Aptian and Cenomanian) is represented by micritic limestones of different colours (grey, green, pink) with radiolarites and other bioturbated foraminifera.

Blade production is the objective of the reduction sequence. The length–width ratio of the unbroken blades (Figure 3) shows a continuous reduction trend. The presence of large and thick blades/ elongated flakes with cortex residues, flat or rarely prepared butts, and partially twisted profile is attested (Figure 3.2). These could be produced by indirect percussion (see details in Pelegrin, 2000, 2006). We also noticed the occurrence of smaller blades (Figure 3.1) with parallel negatives on the dorsal surface, a straight and narrow profile, parallel edges, a short and pronounced bulb, and generally a prepared isolated butt, which suggest the use of the pressure technique (see details Binder, 1984; David et al., 2019; Gómez Coutouly, 2018; Pelegrin, 2012). Overall, 62 refittings have been realized (32 in the SU 109/03 and 30 in the SU 257/03). Eighteen refittings contain five or more pieces, and the largest refitting includes up to 53 items, which allows us to fully reconstruct the original chert nodule. There are no refittings whose components are shared between the two stratigraphic units. The technological features of the whole lithic ensemble, as well as the refittings, indicate a continuous reduction sequence, with different production steps, to realize two kinds of laminar blanks. We propose the following reconstruction of the reduction sequence in four main phases:

A. Preparation: the abundant cortical blanks suggest that cherts were introduced to the site as nodules (Figure 4.1). The primary aim in this phase was to shape one side of the core, usually the narrower one, for the production of the blades. The products of this phase are fully cortical flakes used to prepare the striking platform and elongated cortical flakes (Figure 4.2) to initiate the debitage

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**FIGURE 3** Length/width ratio of blades from stratigraphic units 109/03 and 257/03, with 75% concentration ellipses (black dots–Scaglia Variegata; red squares–maiolica; gray cross–presence of isolated butt); details of (1) pressure blade from Phase D and (2) indirect percussion blade from Phase B (compare Figure 4).



**FIGURE 4** Reconstruction of the lithic reduction phases: (a) Preparation; (b) primary production; (c) Core repreparation; (d) secondary production. Refittings (n° 1–5) from stratigraphic units 109/03 and 257/03 and two final cores (n° 6) from coeval housing structures of Sector XIII of Lugo di Grezzana.

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surface. The flakes show cortical or flat butts as well as very prominent bulbs, suggesting the use of direct percussion. When natural flat surfaces are present on the nodules, these surfaces could similarly be used as striking platforms. The crested blades were occasionally detached to regularize the natural blanks and to shape the first scar for further laminar production.

- B. Primary production: Here, the first laminar blanks are produced (see Figure 3.2). They are long and thick blades or elongated flakes with frequent cortical residues. The blade scars sequence as well as the refittings highlight the common use of alternate unidirectional debitage (Figure 4.3) (see Binder, 1987). The butts of these blanks are flat or rarely prepared. The blades seem to be mainly produced by indirect percussion. The striking platform is prepared thanks to the detachment of centripetal flakes, which are often reflected (Figure 4.4). These flakes are used to regularize the angle between the striking platform and the debitage surface. Most of the cores are abandoned at the end of this phase.
- C. Core re-preparation: in this phase, the core (Figure 4.5) morphology is regularized through the detachment of semicortical flakes along its flanks. If the striking platform is irregular, this can be totally removed with a *tablette*. When the debitage surface does not allow to pursuit a laminar production, it is flattened by detaching a thick elongated flake, or it is regularized with new crested blades. When the original dimensions of the cores do not allow the production of long blades (see previous primary production), the debitage starts directly at this stage to shape the core for the following secondary production stage.
- D. Secondary production: This phase is dedicated to the production of regular and thin blades by pressure technique (compare Figure 3.1). As already attested in the primary production, the most common debitage sequence is the alternated one. The butts of the blades made by pressure are usually prepared by several detachments from the debitage surface to the striking platform, which can isolate an elevated surface. The products of this phase are rare, whereas the cores are totally absent. On the other hand, these blades and cores are commonly found in other coeval areas of the site (Figure 4.6) (Moser, 2000, 2002; Pedrotti & Salzani, 2010).

## Archaeometric results

Ten archaeological samples of maiolica chert from phases B and D of the reduction sequence (compare Figure 2), showing evidence of heat treatment, have been selected for the archaeometric investigations (compare Table S2).

The density values of the archaeological artefacts confirm the use of the heat treatment processes in their manufacturing. In Figure 5, the grey bands indicate the density ranges (mean values and relevant standard deviations) of the geological maiolica samples, measured at room temperature and after furnace treatments, as described in the former "Material and Methods" section, at 250° and 400°C. From



**FIGURE** 5 Comparison between density values of the maiolica geological samples (compare Table S3) - room (RT) and relevant temperatures (gray bands are centred on the mean values and include the standard deviations at each temperature) and archaeological artifacts (red dots–blanks from phase B; blue dots–blades from phase D).



**FIGURE 6** Comparison between 4545/4469 cm<sup>-1</sup> ratios obtained for maiolica geological samples (compare Table S4) - room (RT) and relevant temperatures (gray bands are centred on the mean values and include the standard deviations at each temperature) and archaeological artifacts (red dots–blanks from phase B; blue dots–blades from phase D).

the plot, it turns out that the density values of the archaeological artefacts all fall in the density ranges (grey bands) pertaining to the geological samples treated at high temperatures, that is, 250°C and 400°C (Figure 5). In particular, the blanks from phase B seem to be exposed at higher temperatures, all in excess of 250°C, whereas blades from phase D seem to be heat treated at relatively lower temperatures.

The same archaeological samples used for density measurements were FT-IR tested. In Figure 6, the ratios of the 4,545 cm<sup>-1</sup> and 4,469 cm<sup>-1</sup> IR lines obtained with the artefact from Phases B and D of the reduction chain are displayed. In the figure, the grey bands are centred on the 4,545 cm<sup>-1</sup> and 4,469 cm<sup>-1</sup> ratios for the geological maiolica specimens, heat treated in the lab, being the width of each band given by the relevant standard deviation. In agreement with the density measurements, blades from Phase B seem to have been heat treated at a higher temperature when compared with blades from phase Step D. Moreover, the B-blades show a higher variability, considering the standard deviation values (compare Table S2). On the contrary, blades from Phase D result to be heat treated at least at 250°C, in one case reaching 400°C. This temperature might also represent a partial overheating, considering that at 400°C the chert develops fracturing and crazing. Blades from Phase D were clearly heat treated a maximum of about 250°C, which is a temperature comparable with other archaeological evidence (see for instance Schmidt, Léa, et al., 2013).

## DISCUSSION

Our results contribute to the discussion about heat treatment from both a methodological and archaeological perspective. From a methodological point of view, the use of a combined characterization approach confirms the usefulness of the density measurements and demonstrates the potential of FT-IR analyses, conducted in reflection mode, to assess the presence of effects deriving from the heat treatment associated with the artefact manufacturing. The reflectance FT-IR analyses avoid any bias due to the sample's selection. For example, the archaeological artefacts from Lugo di Grezzana show variable dimensions. which could prevent FT-IR analysis in transmission mode. In the present study, FT-IR tests were carried out on samples with variable morphology (see Figure 2) and with thickness ranging from 2 and 7 mm (compare Table S2). These thickness values would be definitely out of the reach of FT-IR transmission analyses (see for instance Santaniello et al., 2016; Schmidt, Léa, et al., 2013). In reflection mode also large samples, like cores, which would not fit into the small cells for transmission analysis, can be tested instead. Moreover, reflectance tests can be easily conducted on different spots of the same specimen to obtain a sort of mapping of the stone tool.

Differences in temperature between blanks from Phases B and D of the reduction sequence give new insights into the condition of the heat treatment. Particularly, the higher heating temper-

ature detected on the blades from Phase B suggests that the natural blocks of unworked geological chert were heat treated without preliminary preparation, such as decortication or core shaping. The difference in temperature between blanks from Phases B and D can be ascribed to the difference in the propagation depth of the heat wave inside the rocks. However, other interpretations are possible, like for instance the incomplete water release due to an early closure of the pores in the peripheral outer regions of the blocks (see Milot et al., 2017). In this regard, further investigations are needed. Anyway, it turns out that knapping activity started only after the heat treatment was completed, and no evidence of any other significant heating is detectable. This behaviour fits with the findings of several ethnographic studies (Brandt & Weedman, 2002; see also the movie by Belkin et al., 2006, minute 9:30) where entire natural blocks are firstly heat treated and knapped at a later stage. Based on this experimental evidence, it can be concluded that the final aim of the Neolithic craftsmen was to give the best heat treatment to the inner volume of blocks, the chosen part to produce blades by pressure knapping. This is particularly evident if we consider FT-IR results (Figure 6); they show a notable consistency between the values from blades from Phase D and a certain variability among the blanks from Phase B. Blanks from Phase B could be also overheated (see for instance Sample 1 in Figure 6) suggesting that the blocks were heat treated until the achievement of a complete internal alteration.

Considering the scenario of early Neolithic in North-Eastern Italy, the identification of heat treatment at Lugo di Grezzana allows new chrono-cultural considerations. The heat treatment was previously verified in the site of La Vela (Trento province), which is attributed to the Gaban group, roughly dated between 5,000–4,700 cal. BC (Pedrotti, 2001); the evidence from Lugo di Grezzana backdates the introduction of this technique in North-Eastern Italy because the analysed assemblage is stratigraphically older than 5,207–5,093 BCE cal. (compare Material and Methods section; more radiocarbon dates are in progress). Moreover, the presence of the heat treatment in both Fiorano and Gaban contexts underlines the connection among the early Neolithic groups, confirming the similarities already suggested by typological (Bagolini & Biagi, 1977), technological, and functional (Mazzucco et al., 2018; Santaniello et al., 2021) studies of lithic assemblages. Finally, complex human relationships may be seen in this region during the early Neolithic, which is possibly characterized by the presence either e of specialized artisans moving between the Alps and the Po plain or the presence of a shared technical knowledge and/or exchanges routes among different groups.

#### **CONCLUSIONS AND FUTURE PERSPECTIVES**

This study proposes a methodological advancement for the investigation of intentional thermal treatment of archaeological chert artefacts. The use of a multi-analytical approach proved to be particularly valuable for confirming what was recently stressed by other authors (Murray et al., 2022). Both density measurements and FT-IR reflectance analyses allow to test any kind of archaeological sample avoiding any selection due to size and morphology. However further developments can be envisaged for a wider use of these techniques: (a) in order to propose more detailed interpretations, a larger set of samples will be analysed considering other geological formations, significant for the investigated area, such as Scaglia Variegata and Scaglia Rossa; (b) the protocol here proposed will be repeated on geological samples heated at other relevant temperatures to refine the reference values here presented.

Our data allow for the first time to clearly relate the heat treatment of chert with the reduction sequence used in the site. This demonstrates the skills of the Neolithic artisans, who were most likely able to use different techniques, such as indirect percussion or pressure and obviously heat treatment, all along the stone tool manufacturing process. This raises new research questions related to the specific procedure employed to realize the chert heat treatment, for instance, through the use of the sand bath (Schmidt & Morala, 2018). In this respect, it is interesting to remind that several firing structures have been found at the site of Lugo di Grezzana (Costa et al., 2019; Pedrotti et al., 2015), demonstrating a specialized and expert use of pyrotechnology. These aspects need to be investigated further through a specific experimental program devoted to the chert heat treatment.

When compared with the available literature, the results herewith presented enable us to confirm that the heat treatment is a technique attested in two early Neolithic sites referred to different groups of North-Eastern Italy (Santaniello et al., 2016): Gaban and Fiorano ones, as defined by typological studies (Bagolini & Biagi, 1977; Pedrotti, 2001). The analysis of other coeval archaeological contexts in northern Italy attributed to these groups but also to other ones such as the Vhò group, could be essential to consolidate the role of the siliceous rock heat treatment as one of the technical innovations related to the appearance of the Neolithic in Northern Italy.

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#### DATA AVAILABILITY STATEMENT

The data that supports the findings of this study are available in the supplementary material of this article

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