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A Multi-Disciplinary Study based on Archaeometry and Artificial Intelligence: a New Approach for the Investigation of Hearths at the Riparo Mochi Paleolithic Site

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***Abstract*—This paper deals with analytical analyses of samples from one of the most important prehistoric sites, which is Riparo Mochi in Ventimiglia (Imperia - Italy), that aim at obtaining a reliable reconstruction of the combustion structures at the site. The adopted multi-analytical approach relies on Fourier Transform Infrared (FTIR) spectroscopy and X-ray powder diffraction (XRPD). The qualitative analysis allowed the main mineralogical phases present in the findings extracted at the prehistoric site to be identified. To improve the quantitative performance in the materials' analysis, the available data was post-processed taking advantage of Artificial Intelligence (AI) strategies. A data-set of FTIR spectra that refer to pellets of known concentration of three of the identified substances, which are calcite, silicate and quartz, was built. Such a data-set was used to train and validate a neural network, which showed good performance in predicting the concentration of two out of three of the investigated substances. The low performance of the neural network in predicting the concentration of the third compound was directly related to the poor reproducibility of the pellet-production process, which remains an important challenge for improving the network performance.**

***Index Terms*—FTIR, XRD, archaeometry, artifical intelligence, neural networks**

I. INTRODUCTION

The management of fire represents a watershed moment in history due to its connection to advancements in cultural and cognitive development of the genus "Homo". Its importance cannot be understated: cooking, heating, increased sociability as well as its use in technical endeavours are all ways it was affected and driven human society and development. In the later case, it has allowed altering or improving other items and substances to allow for the production of tools, food storage and so much more. During the course of prehistory, pyro-technology developed, as evidenced by traces of fire in various structures associated with combustion: fireplaces, oven, kilns, etc. Utilising archaeological and archaeometric research methods, the project seeks to study such combustion structures that are characteristic of one Italian prehistoric site that are famous both nationally and internationally, i.e. Riparo Mochi in Ventimiglia (Imperia - Italy).

This research's main interest is the chemical-physical characterisation of findings excavated in the aforementioned site, with the far-reaching objective of drawing up a realistic panning shot of the original combustion frameworks at the site. The outcomes of these analyses will enable researchers in improving the knowledge related to the use and management of fire by human groups in the Upper Palaeolithic period. The

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project suggests field studies, laboratory analyses and experimental archaeology to investigate the construction and operation of various structures of combustion, identification and chemical characteristics of materials involved, in addition to seeing how these compare and contrast with the archaeological and historical record. More traditional archaeology methods will be used in conjunction with archaeometry state-of-the-art techniques while concurrently developing and improvising new methods of investigation.

Archaeological finds from the site, such as heated lithic materials, ashes, bones, reddened earths, and pigments, have been investigated using different characterisation analytical techniques both in laboratory and in-situ. Such multi-analytical approach is mainly based on techniques that are traditionally used in the field of cultural heritage investigations, especially suitable for inorganic materials and minerals [1]- [4]. Therefore, the chosen multi-analytical approach mainly relies on optical microscopy, electron microscopy coupled to X-ray microprobe (SEM-EDXS), X-ray powder diffraction (XRPD), Fourier Transform Infrared (FTIR) and Raman spectroscopies. In addition, colourimetry and fibre optics visible reflectance (FORS) have been used, especially for the rapid and in-situ characterisation of the coloured rocks [5].

In this paper, some of the results obtained on selected samples from the site through FTIR spectroscopy and XRPD in terms of chemical and mineralogical composition will be presented. Furthermore, in order to improve the performance of FTIR spectroscopy in the quantitative analysis of the mineralogical phases, a new challenging approach has been implemented. Indeed, the outcomes of FTIR analyses were post-processed taking advantage of Artificial Intelligence (AI) strategies and the first results will be discussed as well.

II. MATERIAL AND METHODS

The qualitative and quantitative characterisation of the mineral composition of clays has always been a demanding task. Usually, techniques such as X-ray fluorescence and atomic spectroscopy are used for the determination of the elemental chemical composition. Besides, XRPD is the more utilised technique for minerals identification and quantification, whereas FTIR spectroscopy has been considered just a complementary tool and especially suitable for qualitative purposes [6]. However, the latter method can be used for quantitative analysis as well, since the measured absorbance for each mineral phase is directly related to its concentration c through the Lambert-Beer law:

$$A = \epsilon \cdot c \cdot d \tag{1}$$

where A is the absorbance, ϵ is the absorption coefficient of the measured species and d is the pathway of the incident light through the sample. Moreover, it is worth highlighting that, although XRPD is widely utilised for semiquantitative and quantitative analysis of clay and mineral mixtures, it has some drawbacks mainly deriving from the trouble in determining amorphous substances and disordered clay minerals.

Since particle size and degree of crystallinity affect the absorption of compounds [6], a great effort was made to try to standardise at the best the grounding and mixing of the whole set of samples (references and findings) to optimise this important factor. One of the main steps of a quantification challenge of unknown mixture of clay and minerals is the calibration of the method that translates in the preparation and measurement of a data set of reference materials, hereafter named ARchaeological Markers (ARMs), which have been used to quantify each substance in the mixture itself.

In this preliminary work, the reference data-set was made of the main components of the findings, i.e. calcite, quartz, and silicates, whose presence was previously determined through FTIR and XRPD techniques. Later on, the spectral set of results obtained on specimens with known concentrations of ARMs were pre-processed and used to train and validate neural network-based algorithms. Both training and validation sets were built starting from single-ARM and multiple-ARM specimens, thus including in the model-identification step the effects of possible cross-sensitivities.

Along the whole identification and validation path, the uncertainty of input data was taken into account, with particular attention to the contributions related to repeatability and reproducibility of the proposed method.

A. Fourier Transform Infrared Spectroscopy

FTIR analyses have been performed by a Jasco FT/IR-4x spectrometer [7]. The spectra here presented were acquired on potassium bromide (KBr) pellets prepared as described down below. The selected spectral range was $(4000 \div 400)\ \mathrm{cm}^{-1}$ and the resolution $4\ \mathrm{cm}^{-1}$. All spectra were obtained as the result of 16 scans to achieve a satisfactory signal to noise ratio. Five measurements were taken in a row for each pellet. The weighed KBr pellets were prepared by following an accurate procedure that was adopted in order to optimise the reproducibility of the reference materials concentration, hereafter named standards. The weight of 1 g was kept constant for the total mixture of KBr with each standard, thus varying the relative concentrations of KBr and standard. The mixture was then homogenised using a mortar. From the mixture, 150 mg were taken 5 times on 5 different watch slides. The same procedure was then used to prepare 5 portions of 200 mg weight. Each 150 mg or 200 mg mixture was then used for the preparation of the pellets. Also in this case, the same procedure was repeatedly followed: the KBr pellet press machine was first left at $5 \cdot 10^3$ kg for 30 s, then the pressure was increased to $10 \cdot 10^3$ kg for 1 min. After the tablet was made, it was placed in the FTIR spectrophotometer, being careful to keep the door open for 5 s, and another 5 s were waited for the first measurement to start. The same method was then used for the preparation of the pellets made of mixtures of KBr and some of the samples taken from the site. The identification of the constituents of real samples, i.e. the qualitative analysis, was performed by comparing their FTIR spectra to the ones available in our database or found in the literature [8]- [10].

B. X-ray powder diffraction

XRPD analyses were performed on powdered samples through a Rigaku - Miniflex 600 diffractometer provided with a Cu X-ray tube (parameters 40 kV, 15 mA). The scan range was between 5.00 ° (start) and 90.00 ° (stop), with a scan step of 0.02 ° and a scan speed of 3.00 °/min.

C. Discussion

The qualitative analyses of the samples were performed though FTIR and XRPD. It is worth mentioning that in this case the available samples were enough to carry on both measurements, which is not so usual in archaeometric analyses. Indeed, XRPD was used above all to check the reliability of the interpretation of FTIR spectra from a qualitative point of view. All the analysed samples almost exclusively contain calcite ($CaCO_3$) and quartz (SiO_2), whose main bands stand respectively at $1798\ \mathrm{cm}^{-1}$, $1418\ \mathrm{cm}^{-1}$, $873\ \mathrm{cm}^{-1}$, $848\ \mathrm{cm}^{-1}$, $712\ \mathrm{cm}^{-1}$ and $1168\ \mathrm{cm}^{-1}$, $1082\ \mathrm{cm}^{-1}$, $1035\ \mathrm{cm}^{-1}$, $1010\ \mathrm{cm}^{-1}$, $916\ \mathrm{(sh)}\ \mathrm{cm}^{-1}$, $796\ \mathrm{cm}^{-1}$, $778\ \mathrm{cm}^{-1}$, $693\ \mathrm{cm}^{-1}$, $518\ \mathrm{cm}^{-1}$, $460\ \mathrm{cm}^{-1}$. Some differences can be observed in sample 9 (Fig. 1), where the peaks at $603\ \mathrm{cm}^{-1}$ and $597\ \mathrm{cm}^{-1}$ suggests the presence of calcium phosphate as hydroxyapatite with poor crystallinity index (CI). The latter identification was particularly challenging, since the main bands of phosphate, which is in the range $(1100 \div 1000)\ \mathrm{cm}^{-1}$, are hidden by the bands of the main component, i.e. the ones of silicate. The identification of hydroxyapatite is really important, since one of the aims of this research is to highlight the potential presence of bones in the site, which are mainly composed of that material. Moreover, the crystallinity index can be used to determine the temperature that findings underwent [11], [12] and, as a consequence, can be related to the presence of burnt or unburnt bones. Speaking of silicates, it also can be noted that in sample 9 the main bands of quartz are almost missing, whereas they were clearly distinguishable in all the other samples. Fig. 1 shows the comparison of sample 9 and sample 8 with reference standard samples of calcite, quartz, and sodium silicate. The identification performed through FTIR was then confirmed by XRPD.

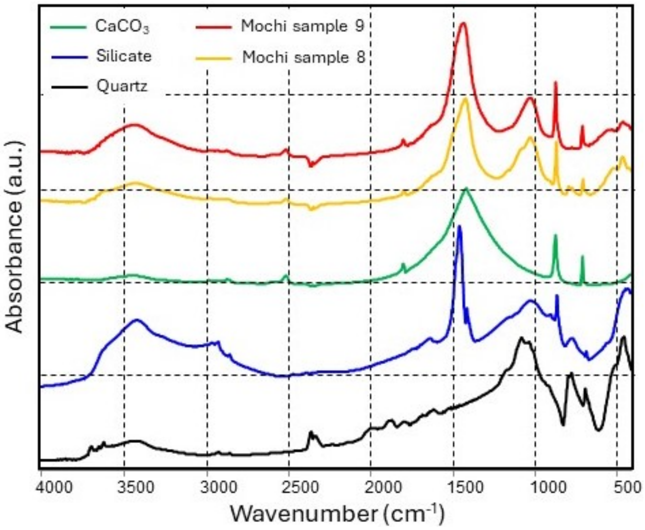

Fig. 1. FTIR spectra of sample 8 (yellow line), sample 9 (red line) and reference standards of calcite (green line), sodium silicate (blue line) and quartz (black line).

III. NEURAL NETWORK AND DATA-SET

All the acquired spectra were preliminary processed in order to assess the measurement quality and the repeatability of multiple measurements of the same sample as well as of different samples at the same nominal mineralogical phase concentrations. This initial analysis highlighted very good repeatability and good reproducibility of measurements carried out on calcite, silicate and their mixtures. However, samples prepared with quartz showed a poor reproducibility because of issues in the sample production. Therefore, at this stage, authors decided to remove from the dataset all the samples containing quartz with the aim of avoiding confusing the network. At the end of pre-processing, a total of about 550 spectra were available for training and validating the neural network. However, a significant part of them are repetitions of the same nominal concentrations that were acquired for the investigation of the repeatability, therefore the effective number of different data is actually quite low and this represents an important limitation in the training, since the network might potentially learn the data and not the model. In order to avoid this issue, authors adopted the following strategy: a proper number of different compound concentrations are randomly peeked and the original dataset is divided in training dataset and validation dataset, so that if one of the selected concentrations appears in a spectrum, the spectrum is added to the validation dataset. All the remaining spectra are instead used for training, trying to maintain in average a ratio 3:1 between training and validation datasets. This approach guarantees that the network will be properly validated using spectra with concentrations never seen during training.

Preliminary tests conducted with various neural networks have shown that the number of available data is anyway insufficient to ensure a proper generalisation of the network and an extension of the dataset is therefore strictly necessary. Unfortunately, the preparation of a large number of reference standards is a highly time-consuming activity, thus authors decided to add synthetic spectra created taking advantage from the linearity of the FTIR absorbance spectrum in terms of substance concentrations.

In particular, authors decided to train and validate the network using as input the difference between two experimental spectra (SA and SB) scaled by a coefficient α randomly selected in the range from 0 to 2. In the same way, nominal target concentrations (TCA and TCB) of the experimental spectra are subtracted and multiplied by the same coefficient to obtain the target concentration (SC) of the synthetic spectrum, as shown in Eq. 2:

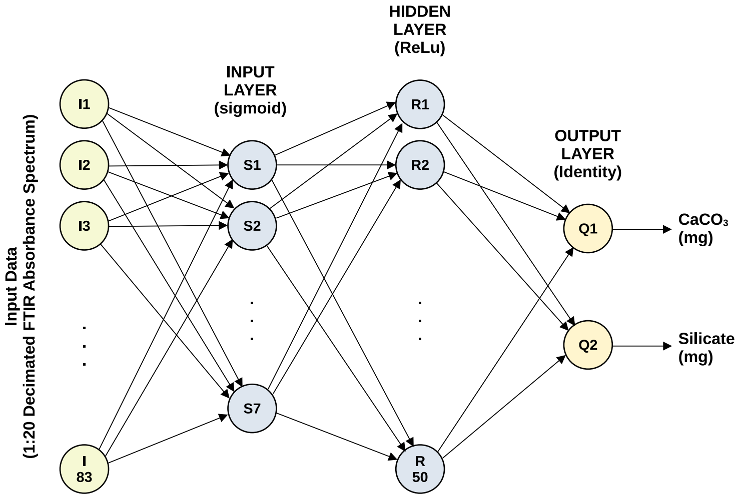

Fig. 2. Structure of the designed neural network. The network is composed by one input layer of 7 neurons (sigmoid activation), one hidden layer of 50 neurons (ReLu activation) and one output layer with 2 neurons (identity activation).

$$\begin{aligned} Synthetic\_Spectrum &= \alpha \cdot (SA - SB) \\ SC_{CaCO_3} &= \alpha \cdot (TCA_{CaCO_3} - TCB_{CaCO_3}) \\ SC_{Si} &= \alpha \cdot (TCA_{Si} - TCB_{Si}) \end{aligned} \quad (2)$$

The two experimental spectra are randomly selected in the original dataset, which is composed by about 380 experimental spectra, and the resulting spectrum added either to training or validation dataset according to the original mineral concentrations.

This approach has also the advantage of automatically taking rid of the spectra background, which is virtually equal for all spectra of the original dataset. Moreover, these preliminary tests showed how the performance of the network does not significantly degrade decreasing the spectra resolution, while significantly reducing the network processing time. Hence, spectra that are initially represented by means of 1661 data-point in the range $(2000 \div 400)\ \mathrm{cm}^{-1}$ were decimated taking one data-point out of 20, leading to 83 points. The decimation factor of 20 was experimentally determined progressively increasing it and monitoring the prediction error of the network till the performance started to degrade. Spectra employed both in training and validation were not normalized in any way, being the information about chemical composition contained in the spectra peak amplitudes.

The final topology of the neural network employs three fully connected layers (input, hidden and output layers) respectively composed by 7 neurons with a sigmoid activation function and taking as input the decimated absorbance spectra, 50 neurons with ReLU (Rectified Linear Unit) function and 2 output neurons using the identity function [13]. All neurons have a bias input. Fig. 2 shows the structure of the neural network, where the outputs are the predicted concentrations of carbonate and silicate, respectively. The network training is carried out by using back-propagation with the Adam optimizer with statistical learning over mini-batches of 500 elements and weight decay of 0.02. Network performance are assessed by calculating the mean square error. Relative error is evaluated as well, in the case the absolute sum of the two nominal concentration is higher than 0.25 mg, by calculating the difference between the predicted output concentrations and the nominal target concentrations, and further divided by the average of predicted and target concentrations with the aim of better assessing the performance when the compound nominal concentrations are close to 0 mg.

IV. EXPERIMENTAL RESULTS

As described in the section II-A, five pellets were obtained for each known concentration of a single ARM (carbonate, silicate, quartz) or of a combination of these ARMs. For each pellet with the same nominal concentration, five spectra were acquired, thus obtaining 25 spectra. The analysis of a group of five spectra related to the same pellet allows the instrumental repeatability to be evaluated, while the dispersion of a group of 25 spectra that refers to five different pellets with the same concentration provides information related to the reproducibility of the procedure implemented for obtaining the reference ARM standards.

An example of spectra that refer to two different concentrations of $\mathrm{CaCO_3}$ (0.6/150 and 0.9/150) is reported in Fig. 3, where groups of five lines with the same colour represents the repeated results obtained analysing the same pellet and the different colours refer to the five different pellets. One should note that the spectra obtained on the same pellet are almost not distinguishable, thus highlighting a very good instrumental repeatability. Maximum absorbance differences not greater than $3 \cdot 10^{-3}$ and $5 \cdot 10^{-3}$ were obtained for the pellets with concentration 0.6/150 and 0.9/150, respectively. Similar results were obtained for the other pellets with different concentrations. The reproducibility among the different pellets is instead not negligible: considering the main peak for the $\mathrm{CaCO_3}$ @ $1418\ \mathrm{cm}^{-1}$, the difference among spectra reaches about 0.1

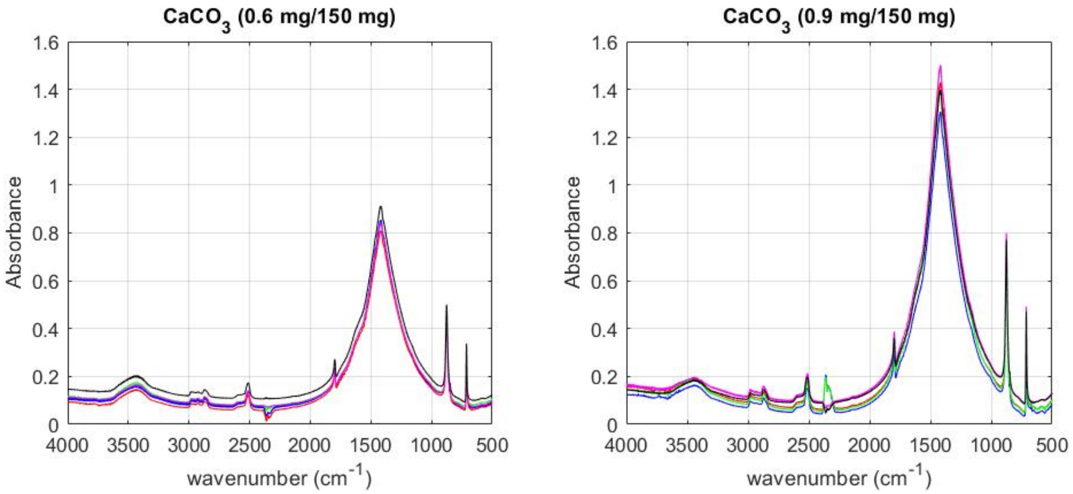

Fig. 3. Example of spectra obtained on two reference standards of calcite. The line colours refer to each group of 5 pellets with the same nominal concentration.

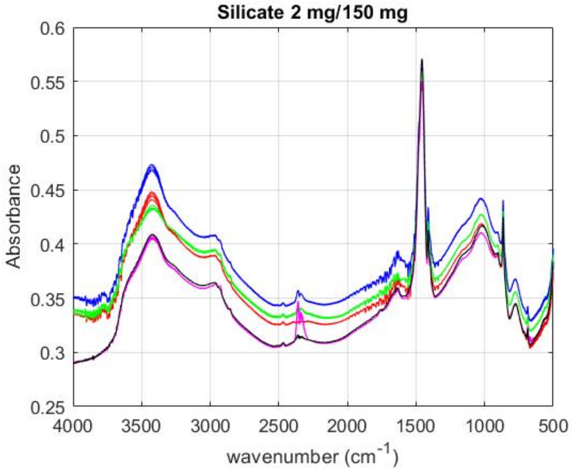

Fig. 4. Example of spectra obtained on a reference standard of sodium silicate. The line colours refer to each group of 5 pellets with the same nominal concentration.

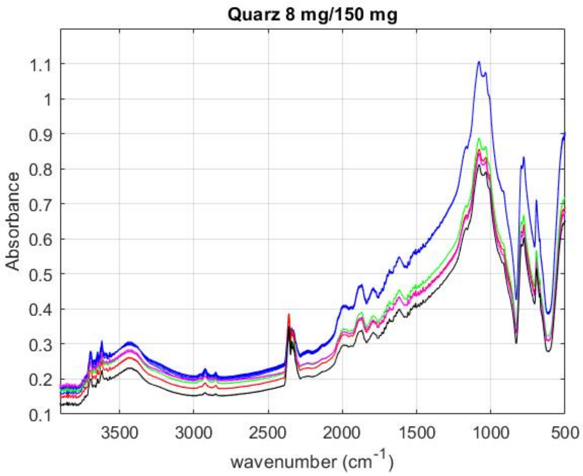

Fig. 5. Example of spectra obtained on a reference standard of quartz. The line colours refer to each group of 5 pellets with the same nominal concentration.

for the concentration 0.6/150 and 0.2 for the concentration 0.9/150.

Another example of spectra that refer to silicate pellets with nominal concentration 2/150 is shown in Fig. 4. Also for these single-ARM pellets, good repeatability results were obtained, while the dispersion among the spectra of the five pellets with the same concentration remains not negligible, showing a maximum absorbance difference that reaches the value of 0.04 in the wavenumber range from $500\ \mathrm{cm}^{-1}$ to $2000\ \mathrm{cm}^{-1}$.

An example of acquired spectra for the quartz pellets with nominal concentration 8/150 is eventually shown in Fig. 5. As anticipated in the section III, the obtained results highlight the poor reproducibility of these single-ARM spectra, which were not considered during the training of the neural network.

As far as the performance of the neural network is concerned, the obtained mean square errors after the training phase resulted in 0.51 mg for the training set and 0.53 mg for the test set, while the relative errors was of about 16% for the calcite and 14% for the silicate. Further investigations were also performed considering spectra containing only either carbonate or silicate. In this case, the relative error in the determination of carbonate decreased to 6% while it remained around 14% for the determination of silicate. Fig. 6 shows the relative error for predictions of $\mathrm{CaCO_3}$ on the validation dataset achieved by the network as a function of the training batches. It is possible to observe how the network quickly improves its performance in the first 2500 training batches reaching the steady error (about 16%) after more than 15000 batches. A similar trend is achieved in the prediction of silicate concentration.

V. CONCLUSIONS

A new approach is presented in this paper that has been conceived to implement a quantitative analysis on findings from

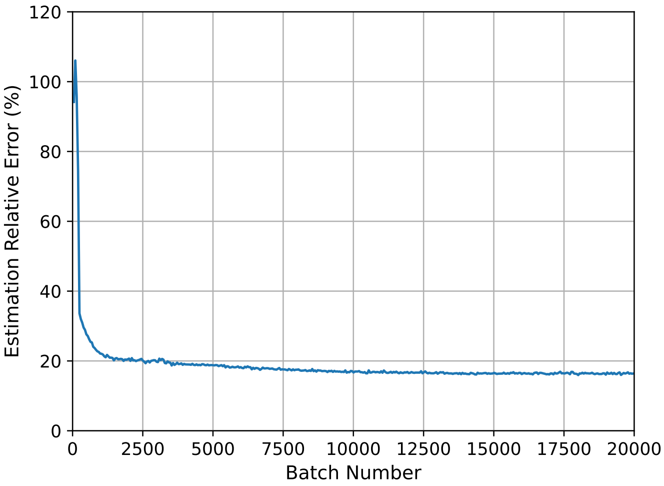

Fig. 6. Validation performance achieved by the neural network in the prediction of $CaCO_3$ concentration as a function of the training batches.

one of the most important prehistoric sites in Mediterranean Europe, i.e. Riparo Mochi in Ventimiglia (Italy).

Initially, thanks to a qualitative analysis that takes advantage of a combination of XRPD and FTIR techniques, the main components of the samples extracted at the prehistoric site have been identified, which have been named ARchaeological Markers (ARMs). Then, reference standards have been assembled with known concentration of three of the ARMs of interest, which are calcite, silicate and quartz, and the corresponding FTIR spectra allowed a large data-set for training and validating a neural network to be built. Eventually, the performance of the trained neural network in terms of capability in evaluating the concentration of the components of interest in single-ARM and double-ARM pellets have been evaluated.

The obtained results showed the effectiveness of the proposed method, also highlighting that the neural-network performance is strictly related to the reproducibility of the sample-production process. Indeed, a training of the neural network that included all the available reference standards provided very poor performance because of the large dispersion of quartz pellets. Excluding quartz spectra from the data-set, the neural network was able to provide relative errors of about 6% and 14% for calcite and silicate, respectively, for single ARM pellets.

Authors are now working on the realisation of pellets made up of other ARMs that are present in the samples extracted at the prehistoric site. Furthermore, particular attention will be paid towards the standardisation of the process implemented to obtain the reference standards, since the obtained results highlighted that the neural-network performance can benefit from an improvement of the overall reproducibility.

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