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Archaeomagnetism applied to medieval ceramics from Southern Europe: preliminary results

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According to the information provided by historical sources and archaeological interventions, the *carreaux de pavement* founded in Tiebas Castle (Navarre region) in Northern Spain were made around 1256, when the castle was built. According to the typology of the tiles, they were manufactured by a workshop from the Champagne region in Northeastern France. The tiles may have been made in Champagne and later transported to Tiebas; or they could also have been manufactured in the vicinity of the castle, where the artisans had to travel to from Champagne. In the summer of 1378, the castle was burned during a war between the Kingdom of Navarre and the Kingdom of Castile. The tiles were already placed on the floor of the Castle's North room and were affected by the heat of the fire.

During the heating of the raw clay at high temperatures, some of their minerals, such as hematite or magnetite, are usually oriented according to the Earth's magnetic field in a determined geographical location and time. In this way, archaeomagnetism is generally used to date a material, based on its relative position, orientation and geographical location of the archaeological material. In this communication, we try to investigate the reverse process: if we know the remaining magnetism and the approximate date of the heating processes, the original relative position and the orientation of the archaeological artefact could be determined.

Based on previous experimental data by XRD, FTIR-ATR and TG-DTA, the tiles were fired in the kiln at temperatures between 875 and 950 ºC, keeping the magnetic field of Navarre or Champagne regions of those years as remanent magnetism. The way the tiles were stacked in the oven (orientation) at ca. 1256 would determine the remaining magnetic vector of each tile. Otherwise, the position of the tiles on the pavement during the 1378 fire could have created a second magnetic imprint based on the magnetic field at that other time.

To obtain the remanent magnetism, several cubic samples of five original *carreaux de pavement* from Tiebas castle (2-2.5 cm side) were cut and analyzed by two different demagnetization processes, thermal demagnetization (TH) and alternating field demagnetization (AF).

The results showed the presence of some tiles with a single magnetic vector and tiles with both magnetic vectors. In the first case (a single magnetic vector), due to its orientation and demagnetization temperature, it is discarded that they could have been caused by the fire, so could match with that of the original firing. In the second case (two magnetic vectors), the vector with the lowest demagnetization temperature (100-450 ºC) could be related to the fire, and the vector with the highest demagnetization temperature (450-640 ºC) to that of the original firing. Several analyses are required to fully specify the stacking in the furnace, still the current results allow us to foresee part of the tiles' organization.

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Use of the medieval ceramic (13th century) magnetic properties to solve archaeological questions

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Introduction and hypothesis

During the heating of raw clay to high temperatures, some of its minerals, such as hematite or magnetite, can be oriented according to the Earth's magnetic field. The direction of the magnetic field depends on the specific geographic location and time. As the material cools, these vectors remain permanently fixed in that direction. It will only be modified if the material is heated again and this modification will depend on whether the applied temperature reaches the Curie temperature (Tc) of the magnetic minerals. In this way, archaeomagnetism is generally used to date archaeological or geological materials. Depending on the relative position, orientation and geographical location of the material to be studied, an estimate for the dating is obtained, according to the knowledge of the Earth's magnetic field at each time. In this communication we try to investigate the inverse process: knowing the remaining magnetism and the approximate date of the heating processes, an attempt is made to determine the original relative position and the orientation of the archaeological artifact in order to answer some questions that history raises.

In this study, samples of Gothic tiles (13th century), called *carreaux de pavement*, from Castillo de Tiebas (Spain) are analyzed. According to bibliographic sources referring to the production of contemporary *carreaux de pavement* in the 19th century [1], the tiles were manufactured in a single firing. This manufacturing tradition may have been similar in the 13th century. According to the previous experimental data by XRD, FTIR-ATR and TG-DTA, it has been estimated that the tiles were fired in the kiln at a maximum temperature of between 875 and 950 ºC [2]. However, archaeological evidence indicates that they could also undergo a second heating due to the fire of 1378, a century after they were manufactured. Depending on the temperature that the tiles could reach, this fire could create a new remaining magnetic vector, partially or completely erasing the magnetic footprint of the original heating (Figure 1).

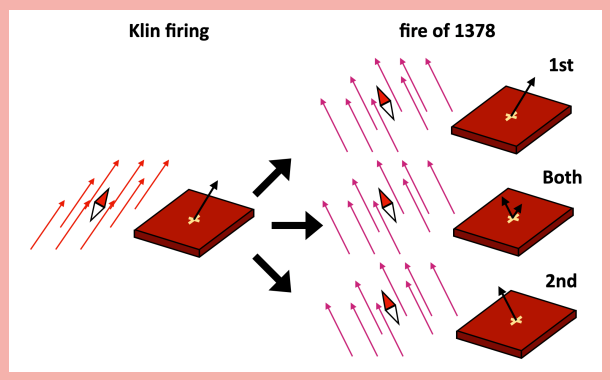

Figure 1. Representation of the vectors in each of the possibilities.

Objective 1: Geographic location

Currently, the place of manufacture the *carreaux de pavement* found in the Castle of Tiebas is under debate. The tiles may have been made in Champagne and later been transported to Tiebas. Or they could also have been manufactured in Navarra, so the artisans had to travel to from Champagne (Figure 2) [3].

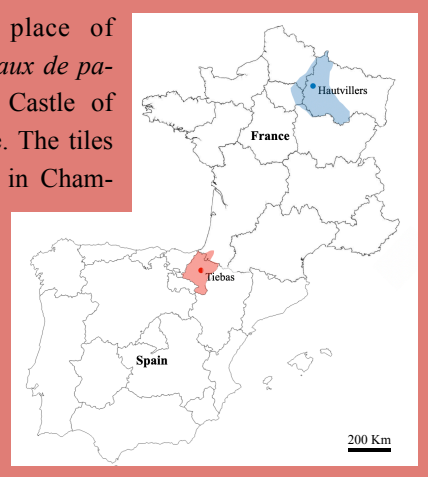

Figure 2. Locations in Navarra (red) and Champaña (blue) where the tiles could be made.

Objective 2: Stacking

How was the stacking of tiles used by medieval craftsmen is one of the less clear questions in historiography (Figure 3). Depending on the stacking of the tiles, they can receive more or less heat than necessary at all times or a greater or lesser oxygenation. Furthermore, the compactness of the kiln determines the volume of tiles that could be manufactured in one go. One of the biggest problems is in the glazing since it makes it impossible to place the tiles by supporting the upper surface with the rest of the tiles as they would stick together.

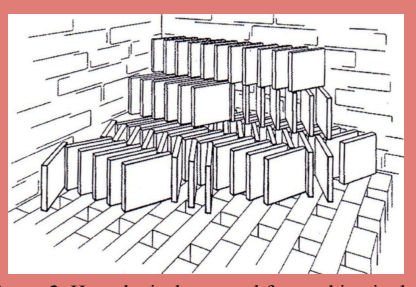

Figure 3. Hypothetical proposal for stacking in the kiln, according to *Carrette, M. et al. (1985)* [4].

Objective 3: Arrangement on the northern hall

In the case of having a magnetic vector caused by the fire of 1378, its orientation could be obtained on the pavement of the north room. This aspect is relevant since in the part of the pavement that has reached our days, two types of orientation of the tiles can be differentiated. Some orientating their grid orthogonal to the walls of the room; and others obliquely at 45º (Figure 4). Each of the two orientations is characterized by completely different decorative motifs from the others.

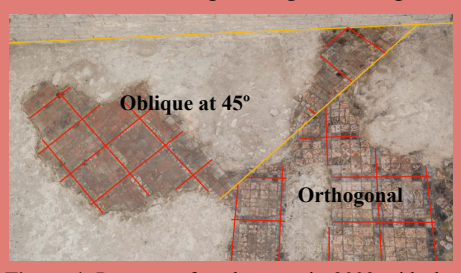

Figure 4. Pavement found *in situ* in 2009 with the arrangement of tiles orthogonal to the walls of the room and the oblique arrangement at 45º [3].

Materials and Methods

To obtain the remaining magnetism, samples of five original *carreaux de pavement* fragments from Tiebas Castle were analyzed. The tile fragments were cut by a non-magnetic sheet to obtain three cubic samples (25 mm side) of each tile (Figure 5). These samples were analyzed by two different demagnetization processes, thermal demagnetization (TH) and alternating field demagnetization (AF). TH demagnetization was applied to 10 samples, while alternating field demagnetization was applied to 5 of them, ensuring that at least one of the samples from each tile was analyzed by each. M1-22 and M11 have been selected taking into account its decorative motifs, since they correspond to the orthogonal and oblique (45º) decorative motifs with respect to the walls of the north hall.

Figure 5. Analyzed samples.

Results and Discussion

The results of the 15 samples analyzed show that at least in 4 of them two remaining magnetic vectors could be identified when the samples are processed by thermal demagnetization (Figure 6). Thermal demagnetization allows to better determine magnetic vectors than demagnetization by alternating fields, since it ensures separating both vectors when demagnetizing the samples (Figure 6).

Samples with a single magnetic vector must correspond to the original firing due to their high demagnetization temperature (500-580 ºC), but above all since their declination and orientation are totally incompatible with those of the fire and are compatible with those of the stacking in the furnace (Figure 7).

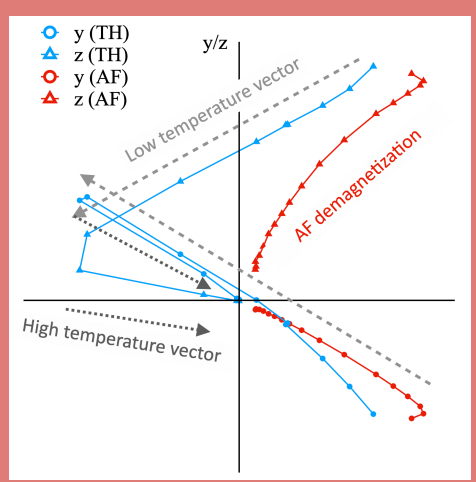

Figure 6. Zijderveld diagram of sample M1-22.

In samples with 2 remanent vectors, the high temperature magnetic vector (from 300-500 to 700 ºC) can be associated with the original firing and the low temperature (from 0 to 200-450 ºC) with the fire. There could be the possibility that this second vector was due to a second firing for the glaze but the temperature reached is not enough to melt the mixture of clay and lead oxides.

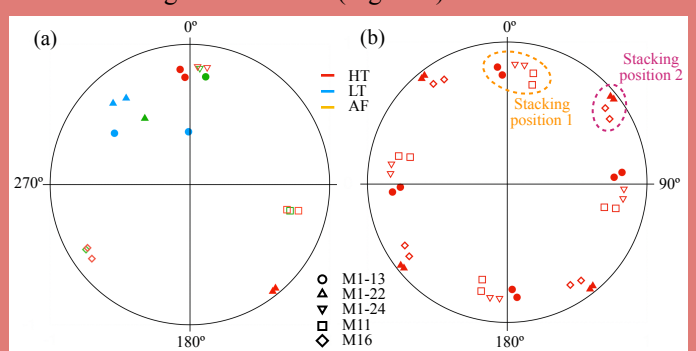

Figure 7. Stereographic representation of all the vectors obtained with respect to the coordinates of each tile (a) and high temperature vectors, taking into account the four possible orientations (b).

First conclusions

- Samples with one and two magnetic vectors have been detected fulfilling the initial hypothesis.
- The high temperature vector is compatible to the original firing and the low temperature vector is consistent with that of the fire in the castle. It is ruled out that this second vector could be from a second firing in the kiln. The castle fire temperature was different for each tile.
- Results are not significant enough to identify Navarra or Champagne as manufacturing sites.
- Results are compatible with two stacking positions in the kiln, although more samples are required to determine in detail the method of stacking.
- Results are consistent with the layout in the north room. In the future it will continue with other decorative motifs to try to virtually rebuild the pavement of the north room.

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