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Archaeomagnetic and rock magnetic study of six kilns from North Africa (Tunisia and Morocco)

Miriam Gómez-Paccard,1 Gregg McIntosh,2,3 Annick Chauvin,4 Elisabet Beamud,5 Francisco J. Pavón-Carrasco3 and Jacques Thiriot6

1 Instituto de Ciencias de la Tierra Jaume Almera, ICTJA-CSIC, Lluís Solé i Sabarís s/n, 08028 Barcelona, Spain. E-mail: mgomezpaccard@ictja.csic.es
2 Instituto de Geociencias (UCM-CSIC), Facultad de Ciencias Físicas, Universidad Complutense de Madrid, Avda Complutense s/n, 28040 Madrid, Spain
3 Departamento de Física de la Tierra, Astronomía y Astrofísica I (Geofísica y Meteorología), Facultad de Ciencias Físicas, Universidad Complutense de Madrid, Avda Complutense s/n, 28040 Madrid, Spain
4 Géosciences-Rennes, CNRS, UMR 6118, Université de Rennes 1, Campus de Beaulieu, 35042 Rennes, Cedex, France
5 Grup de Geodinàmica i Anàlisi de Conques (GGAC), GEOMODELS Research Institute, Laboratori de Paleomagnetisme CCIUB-CSIC, Institut de Ciencies de la Terra Jaume Almera, CSIC, Solé i Sabarís s/n, 08028 Barcelona, Spain
6 Laboratoire d’archéologie médiévale méditerranéenne, UMR 6572, Maison méditerranéenne de Sciences de l’Homme, CNRS, 13094 Aix-en-Provence, France

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SUMMARY
New full-vector archaeomagnetic data for North Africa recovered from the study of six kilns, five from Tunisia and one from Morocco, are presented. Archaeological and historical considerations, along with three radiocarbon dates, indicate that the age of the kilns ranges between the 9th and 15th centuries AD. Rock magnetic analyses showed that the principal magnetic carriers are magnetite and low Ti titanomagnetite, along with variable contributions of thermally stable maghemite and a high coercivity phase with low unblocking temperatures. The magnetic mineralogy of the studied material is thermally stable and behaves ideally during archaeointensity experiments. Stepwise alternating field demagnetization isolated a single, stable, characteristic remanence component with very well defined directions at both specimen and structure levels. Mean archaeointensities have been obtained from successful classical Thellier experiments conducted on between five and eight independent samples per kiln. Thermoremanent magnetization (TRM) anisotropy and cooling rate effects upon TRM intensity have been investigated. The results showed that these effects are low for four of the six studied kilns, with differences between the uncorrected and corrected means of less than 3 per cent. For the other two structures differences between the uncorrected and corrected mean site intensities are 4.4 per cent and 5.8 per cent. These results highlight the necessity for TRM anisotropy and cooling rate corrections in archaeomagnetic studies if accurate archaeointensities are to be obtained. The new results suggest that high intensities occurred in Northwest Africa during the 9th century. Although more data are clearly needed to define this period of high intensity, the results are in agreement with the available European archaeointensity data. A comparison between the new data, other available archaeomagnetic determinations in nearby locations, and palaeosecular variation (PSV) curves derived from the regional SCHADIF3k and global ARCH3K.1 geomagnetic field models shows good agreement between the new data and directional results derived from the models. However, some differences are observed between geomagnetic field models intensity results and available archaeointensity data for the studied regions. This highlights the need for new data for unexplored regions such as North Africa. The new data presented here better constrains the evolution of the geomagnetic field during historical times in this region. They represent a new step towards the construction of a reference PSV curve for Northwest Africa. Once established, this curve will represent a new dating method for this region.

Key words: Archaeomagnetism; Palaeointensity; Palaeomagnetic secular variation; Rock and mineral magnetism; Africa.
1 INTRODUCTION

Archaeomagnetic data are used to compute regional palaeosecular variation (PSV) curves (e.g. Schnepf & Lanois 2005; Gómez-Paccard et al. 2006b, 2008; Tema et al. 2006; Márton 2010) or regional/global geomagnetic field models (e.g. Korte et al. 2009; Pavón-Carrasco et al. 2009) that can help to describe the temporal and the spatial behaviour of the geomagnetic field on a centen-nial/millennial scale. Obviously, the accuracy and the validity of such regional curves and models rely on the quality, the quantity and the spatial and time distribution of the data used for their construction. Future progress in geomagnetic research is therefore dependent on the availability of new, reliable and well-dated data from unexplored regions.

Up to now, very few data are available for Northwest Africa (Fig. 1). For Morocco there are seven directional data (Kovacheva 1984; Najid 1986) and four archaeointensity data (Kovacheva 1984; Casas et al. 2008) for the last two millennia (Supporting Information). For Tunisia one directional and two intensity data have been published for the first millennium BC (Thellier & Thellier 1959), but no archaeomagnetic data are available for the last 2000 years. The study of Odah et al. (1995) includes one intensity data from a site in Libya that is close to the Tunisian border.

This research addresses this shortfall by presenting new archaeomagnetic results from three archaeological sites in Northwest Africa (Fig. 1): Sabra al-Mansuriya, Raqqada (Tunisia) and Rirha (Morocco). The new full-vector information is a start in filling the gap for this region and may be extremely useful for future constructions of PSV curves and geomagnetic field models. Once established, these curves and models will represent a new dating tool for burned archaeological remains from this region.

2 ARCHAEOLOGICAL CONTEXT AND DATING

Six very well preserved kilns have been studied. Five of them come from two archaeological sites in Tunisia, North Africa: a brick-firing kiln (RQD-105, Fig. 2a) from Raqqada, 9 km south of Kairouan and three pottery-firing kilns (SBR-6038, SBR-6043 and SBR-6081, Fig. 2b–d) and a glass-firing kiln (SBR-FV, Fig. 2e) from Sabra al-Mansuriya, now part of urban Kairouan (Fig. 1). Raqqada was the capital of the Aghlabid dynasty, which controlled an area encompassing eastern Algeria, Tunisia and parts of modern day Libya during the 9th century AD. The city may have been occupied by the Aghlabids from the beginning of the 8th century AD and fell to the Fatimid dynasty in 909 AD. It was abandoned in the first quarter of the 10th century AD when the Fatimids moved to the new capital Mahdiya. Sabra al-Mansuriya was founded a little later, in 947–948 AD, marking the return of caliphate power in the region around Kairouan. It remained the capital of the Zirid governors up to 975 AD when the caliphate leader was installed in Cairo. Bedouin tribes eventually overthrew the Zirids, and Kairouan was abandoned around the middle of the 11th century AD.

In Raqqada the last period of use of kiln RQD-105 was before the construction of the Dar al-Bahr Palace in 905 AD. Two radiocarbon dates obtained from charcoal samples studied at the Centre de datation pour le Radiocarbone (CNRS, Université Claude Bernard Lyon 1) are also available. The first radiocarbon sample was taken from RQD-105 and places its use between 673 and 857 calibrated years AD, at a 95 per cent confidence level. The second radiocarbon sample corresponds to a different structure that was in use after RQD-105 and gives an age of 782–976 calibrated years AD at a 95 per cent confidence level. This latter age is in agreement with archaeological and historical evidence that place the abandonment of Raqqada at 905 AD. Together, the historical/archaeological constraints and radiocarbon dates place the last use of the kiln RQD-105 between 800 and 905 years AD.

For the kilns sampled at Sabra al-Mansuriya, archaeological and stratigraphic constraints indicate that kilns SBR-6038 and SBR-6043 were abandoned before kilns SBR-6081 and SBR-FV. The earliest use of the kilns could coincide with the founding of the city in 947–948 AD, although it is more likely that the workshop was established some time after the caliphate moved to Cairo in 975 AD (Cressier, personal communication, 2011). The abandonment of the ceramic and glass workshop appears to be contemporaneous with the capture and destruction of the city in 1057 AD (Cressier & Rammah 2004, 2006; Thiriot 2009). On this basis the last use of the kilns has been placed between 1000 and 1057 AD.

One kiln (RHA) from Rirha (Garb region), Morocco, has been studied (Figs 1 and 2f). Rirha is located 35 km northwest of the archaeological site of Volubilis and 8 km north of Sidi Slimane (Kénitra province). Rirha is a city dating back to the Mauritanian and Roman periods. Archaeological remains found in this site include mosaics, burial inscriptions, pottery and coins. Three phases of occupation can be distinguished: the Mauritanian phase dating back to the 5th century BC, the Roman occupation of the site between the 1st and the 3rd centuries AD and the Islamic phase between the 9th and the 14–15th centuries AD. Kiln RHA corresponds to the Islamic occupation and has been dated by archaeological constraints and one radiocarbon date performed at the Poznan Radiocarbon laboratory (Callegarin et al. 2006, 2011; Coll-Conesa et al. 2011). The radiocarbon (charcoal) sample gave an age between 1290 and 1410 calibrated years AD at a 95 per cent confidence level. Therefore the last use of the kiln has been placed inside this interval. The ages ascribed to each one of the kilns studied for archaeomagnetism are shown in Table 1.

3 METHODOLOGY

3.1 Sampling

At least 18 independently oriented block samples were taken from each kiln. The samples were composed of well-consolidated burned clay or bricks and were oriented by magnetic and solar compass on a horizontal surface prepared using plaster of Paris (Figs 2a–f). The block samples were then subsampled at the Palaeomagnetism Laboratory of the Universidad Complutense (Madrid, Spain) by cutting cubic specimens (2 cm side) close to the interior (most heated) faces of each block. To retrieve the archaeomagnetic directions one specimen per sample was used. Between six and 10 unoriented cubes, one per sample, were used for the archaeointensity experiments.

3.2 Laboratory treatment

The archaeodirectional study was carried out in the Palaeomagnetism Laboratory of the Universidad Complutense (Madrid, Spain). Alternating field (AF) demagnetization of the natural remanent magnetization (NRM) was carried out using a 755–4K SRM (2G Enterprises) superconducting rock magnetometer with an in-line AF demagnetiser or a JR-5 (AGICO) spinner magnetometer and a GSD-5 (Schonstedt) AF demagnetizer. AF demagnetization
was carried out with at least seven steps up to a maximum peak AF of 100–120 mT.

Archaeointensity experiments were carried out at the Palaeomagnetism Laboratories of the Institute of Earth Sciences Jaume Almera (Barcelona, Spain) and Géosciences-Rennes (Rennes, France), following the classical Thellier palaeointensity method (Thellier & Thellier 1959). This method is based on the comparison of the NRM lost and the partial thermoremanent magnetization ($p$TRM)

Figure 1. Map of Europe and North Africa showing the location of Rirha, Sabra al-Mansuriya and Raqqada (black dots). Grey circles indicate location of other directional available directional (a) or archaeointensity (b) data.
Figure 2. Photographs of the sampled kilns at (a) Raqqada, (b)–(e) Sabra al-Mansuriya and (f) Rirha.

Gained in a known laboratory field at progressively higher temperatures. Remanent magnetization was measured on a 755R SRM (2G Enterprises) superconducting rock magnetometer. Specimens were heated in a MMTD-80 (Magnetic Measurements) furnace from 100°C up to temperatures at which more than 85 per cent of the initial magnetization was lost. A laboratory field of 500 mT was applied.

At each temperature step, samples were first heated and cooled with the laboratory field applied along their $Z$-axis and second, were heated and cooled with the laboratory field applied in the opposite sense. Between seven and 19 temperature steps were needed.

The TRM anisotropy tensor was calculated for each specimen from TRM acquisition in six different directions. The cooling rate dependence of TRM intensity was also analysed by measuring four additional TRM acquisition steps at the end of the Thellier experiments. Comparing the rapid (the typical laboratory cooling time of about 1.5 hours) and slow cooling results (about 24 hours) allowed the quantification of the cooling rate effect upon archaeointensity estimates for each specimen. For a detailed description of the experimental procedures followed in the archaeointensity protocol see Gómez-Paccard et al. (2006a).

Rock magnetic studies were carried out on AF demagnetized specimens or on subsamples prepared from block off-cuts at the Palaeomagnetism Laboratory of the Complutense University (Madrid). Low field magnetic susceptibility ($\kappa$) of each archaeodirectional specimen was measured using a KLY-3 kappabridge (AGICO). Magnetic hysteresis and backfield isothermal remanence (IRM) curves were measured on crushed samples using a J-Meter coercivity meter (Iassanov et al. 1998) with a maximum applied field of 500 mT. Backfield IRM curves were measured on additional AF demagnetized specimens using a JR-5 (AGICO) spinner magnetometer and an IM-10–30 (ASC Scientific) impulse magnetizer with a maximum applied field of 2 T. The same specimens were used to study the thermal (TH) demagnetization of orthogonal IRMs (Lowrie 1990), separating the total IRM into soft (0–0.1 T), medium (0.1–0.3 T) and hard (0.3–2 T) components. The specimens were demagnetized using a TSD-1 (Schonsted) thermal demagnetizer. Strong field thermomagnetic curves (in air, heating rate 40°C min$^{-1}$, applied field 300 mT) of representative crushed subspecimens were measured using a MMVFTB (Petersen Instruments) variable field translation balance.

## 4 MAGNETIC MINERALOGY

Initial NRM intensities and $\kappa$ of the archaeodirectional specimens ranged over several orders of magnitude, with the highest values observed for the RHA (Morocco) specimens (Fig. 3). Koenigsberger ratios (defined as $Q_{\text{K}} = \text{NRM}/\kappa H$, with $H = 39 \text{ A m}^{-1}$) between 5 and 30 are consistent with a thermomagnetic origin for the NRM.

In total, 45 magnetic hysteresis experiments have been performed. All but four yielded magnetic hysteresis curves that showed reversible behaviour by 300 mT and backfield IRM curves that reached saturation by 300 mT (e.g. sample SBR-6081–08, Fig. 4a). Therefore the increase in magnetization between 300 and 500 mT is considered to be due to paramagnetic behaviour, whose contribution has been estimated from the slope of the descending branch of hysteresis curve between 500 and 300 mT. Four samples exhibited curves that were slightly non-reversible beyond 300 mT and backfield IRM curves that did not approach saturation (e.g. RQD-105–06, Fig. 4b). This high field behaviour has been considered as

<table>
<thead>
<tr>
<th>Site</th>
<th>Latitude (°)</th>
<th>Longitude (°)</th>
<th>Kiln</th>
<th>Dating method</th>
<th>Dating (yr AD)</th>
</tr>
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<td>35.60 N</td>
<td>10.06 E</td>
<td>RQD-105</td>
<td>Historical/archaeological constraints + radiocarbon dates</td>
<td>800–905</td>
</tr>
<tr>
<td>Sabra al-Mansuriya</td>
<td>35.66 N</td>
<td>10.10 E</td>
<td>SBR-FV</td>
<td>Historical/archaeological constraints</td>
<td>1000–1057</td>
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<tr>
<td></td>
<td></td>
<td></td>
<td>SBR-6038</td>
<td>Stratigraphic constraints indicate that 6038</td>
<td>1000–1057</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>SBR-6043</td>
<td>and 6043 were abandoned before 6081 and FV</td>
<td>1000–1057</td>
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<td></td>
<td></td>
<td>SBR-6081</td>
<td></td>
<td>1000–1057</td>
</tr>
<tr>
<td>Rirha</td>
<td>34.31 N</td>
<td>5.93 W</td>
<td>RHA</td>
<td>Archaeological constraints + radiocarbon date</td>
<td>1290–1410</td>
</tr>
</tbody>
</table>
a mixture of paramagnetic and unsaturated ferromagnetic (s.l.) contributions. For these samples the high-field contribution has been calculated from the descending branch of the curve between 500 and 450 mT.

After high-field correction, saturation magnetization \( (M_s) \) varied between 0.51 and 371 mAm\(^{-2}\) kg\(^{-1}\) and coercive force \( (B_c) \) between 3 and 25 mT. The highest values of \( M_s \) and saturation remanence \( (M_r) \) were observed for samples from kiln RHA (Morocco).

The vast majority of backfield IRM curves (37 of 45) were square-shaped, showing a sharp change in IRM below 200 mT and minor amounts of high-coercivity IRM beyond 300 mT (SBR-6038–19 and SBR-6043–02, Fig. 4c), with coercivity of remanence \( (B_c) \) values ranging between 12 and 59 mT. A further eight samples exhibited a greater proportion of high coercivity IRM, with the shape of the curve clearly suggesting the presence of distinct coercivity phases (SBR-FV-04, Fig. 4c) of varying contributions. This included six samples from structure RQD-105 and two from structure SBR-FV. \( B_c \) values ranged between 29 and 233 mT, \( H_c \) increasing as the proportion of IRM acquired above 300 mT increases.

When plotted together on a Day plot (Day et al. 1977; Fig. 4d), the results tend to group together within the pseudo-single domain region. Six outlying values plot to the right of the main group, showing relatively high \( B_c/\mu_0 \) ratios. This includes four samples from structures RQD-105 and SBR-FV, which acquired a large proportion of IRM beyond 300 mT and thus exhibit relatively high \( H_c \) values. The other two samples are from kiln SBR-6043. They differ from the RQD-105 and SBR-FV samples in that they show a relatively small amount of IRM acquisition beyond 300 mT. They do have small \( B_c \) values (4 and 6 mT), which may contribute to the high \( B_c/\mu_0 \) values.

Thermal demagnetization of orthogonal IRMs indicated that the soft (0–0.1 T) IRM component dominated the total IRM in nine of the 10 studied samples, in all cases exhibiting maximum unblocking temperatures of 540–620 °C (e.g. RHA-17, Fig. 4e). In the case of sample RQD-105–07 (Fig. 4f) the soft (0–0.1 T) and hard (0.3–2 T) components contributed almost equally to the total IRM. The hard component was dominated by demagnetization between 100 and 200 °C (Fig. 4f). This was a thermally stable feature, with the same IRM and unblocking temperatures observed when repeating the acquisition and demagnetization after heating to 700 °C (green symbol, Fig. 4f). Similar unblocking temperatures and thermal stabilities were observed for the hard (0.3–2 T) component of the other two RQD-105 samples studied, although it contributed less to the total IRM. The hard component demagnetization curves of the remaining samples were noisy, although maximum unblocking temperatures of around 620–700 °C could be tentatively identified in four cases.

Twelve thermomagnetic curves have also been measured. The results showed a high degree of reversibility, indicating that no important magnetomineralogical changes occurred during heating. Five of the curves showed a single change in slope on the heating and cooling curves (e.g. RHA-22, Fig. 4g), which suggests a single dominant ferromagnetic phase. Curie temperatures \( (T_C) \) were calculated using the second derivative of the heating curve (Tauxe 1998), yielding values of 575–628 °C. Five samples showed a second, poorly expressed slope change (e.g. SBR-6081–20, Fig. 4h), indicating the presence of two ferromagnetic phases with \( T_C \) of 214–460 °C and 560–589 °C. The remaining two curves did not show a discernible slope change and no reliable \( T_C \) could be calculated.

Together, the results suggest that the magnetic mineralogy of the studied material is thermally stable and thus well suited to archaeomagnetic and archaeointensity studies. The kilns SBR-6038, SBR-6043 and SBR-6081 (Sabra al-Mansuriya) and RHA (Rirha) are dominated by low coercivity magnetic phases \( (H_c \leq 25 \text{ mT and } H_c \leq 59 \text{ mT}) \). \( T_C \) of 560–602 °C support the presence of magnetite or low titanium titanomagnetite, along with some maghemite that may be giving rise to the slightly elevated \( T_C \). Samples from kilns SBR-6043 and SBR-6081 also show \( T_C \) of 330–460 °C, which can be explained by the presence of titanomagnetite with higher titanium content. Similar unblocking temperatures of both IRM and NRM confirm that they contribute to the stable remanence of the material.

Kilns SBR-FV (Sabra al-Mansuriya) and RQD-105 (Raqqada) also show a low coercivity phase, with \( T_C \) of 575–628 °C and unblocking temperatures (IRM and NRM) of 570–630 °C supporting the presence of magnetite and/or maghemite.

Samples from Sabra (SBR-FV) and Raqqada (RQD-105), both near Kairouan, show the variable presence of a high coercivity phase with remanence coercivities greater than 0.3 T, IRM unblocking
Figure 4. Representative rock magnetic results. (a)–(b) Hysteresis curves, (c) backfield isothermal remanence (IRM) curves, (d) Day plot of magnetization and coercivity ratios, (e)–(f) thermal demagnetization of IRM and (g)–(h) strong field thermomagnetic curves.
Figure 5. Directional results. The left-hand side shows two representative orthogonal vector plots of AF demagnetization of NRM, with solid/open symbols denoting projection on the horizontal/vertical plane. The right hand side shows stereoplots of specimen characteristic directions in grey and the mean direction and $\alpha_{95}$ in black.

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temperatures lower than 300°C and Tc's of around 230°C. This phase is particularly common in samples from kiln RQD-105 where it may contribute more than half of the total IRM. NRM unblocking temperatures around 150–300°C and AF coercivities in excess of 100 mT suggest that the same phase contributes to the stable remanence of the material.

This phase shares the same characteristics as those exhibited by a thermally stable, high coercivity and low unblocking temperature phase described by McIntosh et al. (2007, 2011). They ascribe the phase to either hemoilmenite, inherited by the baked clays from adequate source rocks, or to substituted hematite or iron cristobalite, formed through the transformation of iron-rich clays during heating. The geology of the catchment area surrounding Kairouan consists of Tertiary and Quaternary sediments overlying Cretaceous limestones and marls (Dassi et al. 2005). Although Neogene volcanism is predominantly restricted to more northerly areas of Tunisia (e.g. Maury et al. 2000), the presence of titanomagnetite in kilns SBR-6043 and SBR-6081 would suggest that some volcanic-derived sediments are available around Kairouan that may also provide a viable hemoilmenite source. Differentiating between inherited hemoilmenite and neoformed hematites or iron cristobalite is not possible without more detailed knowledge of sediment sources, further magnetic tests (such as those summarized by McIntosh et al. (2011)), and/or microscopy studies.

There was some evidence for the presence of hematite in the IRM results that showed poorly defined unblocking temperatures of up to 650–700°C, although its contribution to the magnetic properties was minor in all cases. Where samples showed a large high coercivity contribution, this was always associated with unblocking temperatures lower than 300°C.

5 NEW ARCHAEO MAGNETIC RESULTS FOR NORTHWEST AFRICA

5.1 Characteristic directions

Progressive AF demagnetization revealed a single, stable NRM component between 5–10 mT and 100 or 120 mT (Figs 5a–f) in all but three of the specimens analysed. In most cases at least 85 per cent of NRM was demagnetized by 100–120 mT, although in isolated cases up to 58 per cent of NRM remained. This high coercivity NRM always preserved the same direction as the low coercivity NRM, suggesting that it represents the same stable NRM component, which is likely the TRM acquired during the last use of the kilns. Specimens from kilns SBR-FV (Fig. 5d) and RQD-105 (Fig. 5e) tended to have higher proportions of AF-resistant NRM.

Principal component analysis (Kirschvink 1980) was used to calculate the best-fit direction of the characteristic magnetization, yielding well-defined directions with maximum angular deviations (MAD) of less than 2°. The mean directions obtained for each kiln were calculated using Fisher (1953) statistics. The resulting means are very well defined, with their associated confidence angles (α95) lower than 1.5° for all the structures except for SBR-6043 and RHA, which have α95 of 2.1° and 1.9°, respectively (Table 2 and Fig. 5). Fig. 6 shows equal area projections of site mean directions. Similar directions were obtained for kilns SBR-FV, SBR-6038, SBR-6043 and SBR-6081 which, taking into account the errors, is in agreement with the available archaeological/historical information that proposed the same age interval for these structures (Table 1). The results do not distinguish the relative age difference between the kilns that was provided by their stratigraphic relationship. The mean site directions obtained indicate that important changes in inclination (of ∼30°) occurred in North Africa between the 9th and the 15th centuries (Fig. 6). This illustrates the potential of PSV geomagnetic field changes as a dating tool in Northwest Africa.

5.2 Archaeointensity determination

Archaeointensity estimations were attempted on 48 specimens, between 6 and 10 per structure. As rock magnetic experiments suggested, the magnetic carriers were stable during Thellier experiments. Very high quality linear NRM-TRM diagrams with successful pTRM checks were obtained corresponding to well-defined single component magnetizations trending toward the origin (Figs 7a–f). Maximum unblocking temperatures between 430–460°C (Fig. 7c) and 630°C (Fig. 7e) were obtained, in broad

<table>
<thead>
<tr>
<th>Site</th>
<th>Kiln</th>
<th>Age (yr AD)</th>
<th>n</th>
<th>Dk (°)</th>
<th>Ik (°)</th>
<th>α95 (°)</th>
<th>k</th>
</tr>
</thead>
<tbody>
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<td>Raqqada</td>
<td>RQD-105</td>
<td>800–905</td>
<td>19</td>
<td>9.8</td>
<td>59.7</td>
<td>1.5</td>
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<td>1000–1057</td>
<td>20</td>
<td>15.0</td>
<td>50.7</td>
<td>1.5</td>
<td>503.0</td>
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<td>SBR-6038</td>
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<td>49.0</td>
<td>1.2</td>
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<td>1000–1057</td>
<td>18</td>
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<td>52.3</td>
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<td>18</td>
<td>6.8</td>
<td>33.6</td>
<td>1.9</td>
<td>328.2</td>
</tr>
</tbody>
</table>

Figure 6. Stereographic projection of the mean direction of magnetization with α95 error circles of the new African data. All data relocated to Sabra al-Mansuriya.

Table 2. Summary of archaeomagnetic directions obtained from AF demagnetization. Site, archaeological site where the kiln was sampled; Kiln, name of the kiln; Age, age of the structure in yr AD; n, number of samples taken into account for the calculation of mean directions; Dk, Ik, mean declination and inclination in situ; α95, 95 per cent confidence cone of mean directions and k, precision parameter (Fisher 1953).
Figure 7. Archaeointensity results. Typical Arai plots of NRM lost against TRM gained. Solid symbols indicate data used for archaeointensity determinations. The initial NRM values, the intensity ($F$) and quality factor ($q$) obtained for each specimens are indicated. Temperatures are in °C. Also shown are the orthogonal vector projections of the NRM in sample coordinates, with solid/open symbols denoting projection on the horizontal/vertical plane.
Table 3. Summary of archaeointensity results. Kiln, name of the kiln; Sp., sample number; NRM, intensity of the natural remanent magnetization in A m$^{-1}$; $\kappa$, initial susceptibility in 10$^{-6}$ SI units; $T_{\text{min}} - T_{\text{max}}$, temperature interval used for the slope calculation in °C; $n$, number of data points within this temperature interval; $f$, fraction of the NRM component used in the slope calculation; $g$, gap factor; $q$, quality factor; MAD, maximum angle of deviation; DANG, deviation angle; CRM, potential error on the estimation of the palaeointensity due to the acquisition of CRM as a percentage of the applied field; $F \pm \sigma F$, intensity estimate and its standard error per specimen without TRM anisotropy correction; $F_\alpha$, intensity estimate per specimen after correction of TRM anisotropy; $F_\text{CRM}$, potential error on the estimation of the palaeointensity due to the acquisition of CRM as a percentage of the applied field; $F_\text{po}$, weighted mean intensity per site; $\Delta F_\text{TRM}$ (24 hr), correction factor per sample for a cooling time of about 24 hr; alt. (24 hr), alteration factor per sample for a cooling time of about 24 hr; $F_\text{pcr}$, weighted mean intensity per site after TRM anisotropy and cooling rate corrections; VDM and VADM, value of the virtual dipole moment and virtual axial dipole moment, respectively.

<table>
<thead>
<tr>
<th>Kiln</th>
<th>Sp. (A m$^{-1}$)</th>
<th>$\kappa$ (10$^{-6}$ SI units)</th>
<th>$T_{\text{min}} - T_{\text{max}}$ (°C)</th>
<th>$n$</th>
<th>$f$</th>
<th>$g$</th>
<th>$q$</th>
<th>CRM (per cent)</th>
<th>$F \pm \sigma F$ (µT)</th>
<th>$F_\alpha$ (µT)</th>
<th>$F_\text{CRM}$ (µT)</th>
<th>$F_\text{po}$ (µT)</th>
<th>$\Delta F_\text{TRM}$ (24 hr) (per cent)</th>
<th>alt. (24 hr) (per cent)</th>
<th>$F_\text{pcr}$ (µT)</th>
<th>VDM 10$^{22}$ A m$^3$</th>
<th>VADM 10$^{22}$ A m$^3$</th>
</tr>
</thead>
<tbody>
<tr>
<td>RQD-105 (800–905 AD)</td>
<td>02 1.89 2286 100–630</td>
<td>17 0.95 0.86 24.3</td>
<td>1.7 1.3 6.6</td>
<td>77.0 ± 2.6 55.5 66.6 ± 6.8 67.4</td>
<td>4.0</td>
<td>1.7</td>
<td>65.6 ± 6.2</td>
<td>11.3</td>
<td>11.9</td>
<td></td>
<td></td>
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<tr>
<td>SBR-FV (1000–1057 AD)</td>
<td>03 0.50 611 100–460</td>
<td>9 0.48 0.86 22.9</td>
<td>3.6 3.6 5.7</td>
<td>76.1 ± 1.4 74.1 64.9 ± 5.7 62.8</td>
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<td>SBR-603 (1000–1057 AD)</td>
<td>03 0.74 1123 100–555</td>
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<td>SBR-6043 (1000–1057 AD)</td>
<td>01 0.36 1302 100–460</td>
<td>9 0.85 131.2 2.3</td>
<td>1.3 1.9</td>
<td>56.5 ± 0.3 58.7 60.2 ± 4.4 58.3</td>
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</table>

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Table 3. Continued.

<table>
<thead>
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<td>9.01</td>
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<td>43.3</td>
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</table>

The quality of the obtained archaeointensity data has been assessed using the typical parameters of archaeointensity studies (e.g. Gómez-Paccard et al. 2006a) and are described in Table 3. The majority of the archaeointensity determinations have f factors bigger than 0.6 (Fig. 8a). One specimen with a f factor equal to 0.48 has also been accepted. All the specimens had MAD (Kirschvink 1980) values lower than 6° (Fig. 8b), and DANG (Pick & Tauxe 1993) values lower than 5° (Fig. 8c). The potential error of the archaeointensity caused by chemical remanent magnetization acquisition during Thellier experiments (the CRM parameter, Coe et al. 1993) was lower than 0.6 (Fig. 8d). The potential error of the archaeointensity caused by chemical remanent magnetization acquisition during Thellier experiments (the CRM parameter, Coe et al. 1993) was lower than 0.6 (Fig. 8d). It is worth mentioning that a maximum acceptable CRM of 15 per cent has been retained by Gómez-Paccard et al. (2006a).

The effect of TRM anisotropy and cooling rate upon TRM intensity acquisition should be determined and field strength estimations for each specimen must be corrected appropriately (e.g. Chauvin et al. 2000; Genevey & Gallet 2002; Gómez-Paccard et al. 2006a, 2008). As expected for the kind of material analysed (burned clay or bricks from kilns, e.g. Kovacheva et al. 2009b) the effects of TRM anisotropy are generally low, and very similar values of the intensity before and after TRM anisotropy correction were obtained (Table 3 and Fig. 9a). In only two cases (R105–02 and RHA-12) did the difference exceed 10 per cent.

The cooling rate dependence of TRM intensity was investigated for a cooling time of about 24 hr, assumed to be of the same order as the ancient cooling time of the structures, although there is no strong evidence to support this. In some cases, specimens broke during experiments and cooling rate correction factors could not be determined. As in previous studies, the cooling rate effect was quantified by carrying out supplementary TRM acquisition steps after the end of Thellier experiments. The archaeointensity values were corrected for specimens whose cooling rate correction factor was bigger than the magnetochemical alteration factor. This alteration factor quantifies the amount of magnetochemical changes that took place during the supplementary cycle of measurements (Gómez-Paccard et al. 2006a for further details). Fig. 9(b) shows the applied correction factors per specimen. A total of 22 archaeointensity values were corrected. In general, cooling rate correction factors were low (less than 4 per cent) and only two specimens showed correction factors higher than 10 per cent (12.6 per cent for SBR-FV-03 and 11.5 per cent for SBR-6081–11). Therefore, when cooling rate correction factors could not be determined the uncorrected archaeointensities were retained to calculate mean site intensities. The archaeointensity estimations per specimen presented in Table 3 fulfill the most demanding of quality criteria and are corrected for TRM anisotropy and cooling rate effects, where appropriate, so that the results can be considered of high quality.

Mean archaeointensities per kiln were calculated using between five and eight archaeointensity values per structure and were derived considering the weighting factor of Prévot et al. (1985) for each specimen. The dispersion of the means was expressed as the
standard deviation (Table 3). Very small TRM anisotropy corrections were performed at a site level, with differences between the uncorrected and the TRM anisotropy corrected means less than 2% per cent for the five Tunisian kilns and 4.7% per cent for the Moroccan kiln (RHA). Differences between TRM anisotropy corrected and cooling rate and TRM anisotropy corrected intensity means are lower than 3% per cent for all of the studied kilns. For kilns SBR-FV, SBR-6038, SBR-6043 and SBR-6081 the differences between uncorrected and TRM anisotropy and cooling rate corrected mean intensities (Fpocr in Table 3) are less than 3% per cent. For kilns RQD-105 and RHA these differences are higher (4.4 and 5.8% per cent, respectively). These results highlight the need for TRM anisotropy and cooling rate corrections in archaeomagnetic studies if reliable mean archaeointensity estimates are to be obtained. Values of the standard deviation of 7–10% of the estimated archaeointensities were obtained for all the kilns except SBR-FV, for which a value of 12.6 $\mu$T was obtained (20% per cent of the estimated archaeointensity, Table 3).

All four kilns from Sabra al-Mansuriya (SBR-FV, SBR-6038, SBR-6041 and SBR-6081) exhibited similar intensity values (see Table 3). This is consistent with the archaeological and stratigraphical constraints that suggest a similar age for the kilns. The lowest intensity value was observed for kiln RHA (Morocco, Table 3). The new results obtained for Morocco and Tunisia are plotted in Figs 10(a) and (b). The Tunisian results indicate that high intensities of about 66 $\mu$T occurred in this region during the 9th century AD (Fig. 10b). This result is in agreement with available European data that indicate that a rapid intensity fluctuation around 800 AD occurred in Europe (Genevey & Gallet 2002; Gallet et al. 2009; Kovacheva et al. 2009a). Although more data are clearly needed to confirm the occurrence of this fluctuation in North Africa it seems that it is also observed in this region.

6 DISCUSSION

6.1 Maghemite and archaeointensity results

Rock magnetic results suggest that the magnetic properties of the studied baked clays are in most cases dominated by magnetite, titanomagnetite (with variable but generally low Ti content) and maghemite. Each one of these magnetic phases contributed to the stable NRM retained by the burned clays. As expected for this kind of material the archaeomagnetic experiments carried out were successful for the majority of the samples analysed. Therefore the maghemite identified during rock-magnetic experiments (Section 4) shows a high degree of thermal stability, something that has been observed in synthetic (Özdemir & Banerjee 1984) and natural material (e.g. Gehring et al. 2009), including archaeomagnetic material (Jordanova et al. 1997).

It is the origin of stable maghemite that is of importance in archaeointensity studies and whether it carries a natural remanence that is chemical or thermal in nature. Stable maghemite formed through low-temperature oxidation (of magnetite, for instance) may acquire a chemical remanence (CRM). If it subsequently acquires a TRM during the intensity experiments then this would lead to a bias on the past field intensity determination.

Alternatively, maghemite may form through high-temperature processes, for example, the transformation of lepidocrocite during heating (Gendler et al. 2005). This study showed that the newly formed stable maghemite could acquire a TRM after multiple heating, giving rise to linear Arai plots until it starts to transform to hematite at temperatures above 550 C. Therefore thermally stable maghemite may also carry a thermoremanent NRM component and may behave ideally in archaeointensity experiments—with the limitations and caveats that normally apply to such experiments. Indeed,
this was found to be the case by Schnepp et al. (2009) in magnetite and maghemite bearing oven floors.

In this scenario the limiting factor may be the temperature reached by the burned clays. In the Gandler et al. (2005) study, the maghemites began to transform beyond 550°C, whereas the maghemites of Jordanova et al. (1997) and Gehring et al. (2009) were stable beyond their Curie temperatures of around 650°C. The temperatures reached by burned clays (and subsequently their magnetic properties) depend on their position with respect to the heat source and their thermal properties. Some parts of the kiln may then reach temperatures that do not exceed 600°C (Spassov & Hus 2006) and so the clays might then be expected to retain any stable maghemite that may have formed.

In the context of this study, the distribution of maghemite-bearing samples with respect to their location within each block sample is poorly constrained, so that determining their relation with respect to the combustion chamber is not possible. Furthermore their position with respect to the external part of the kiln structure is not constrained, which might have some bearing on the degree to which they may have suffered post-abandonment low temperature oxidation. This information should be recorded in future studies that may then address the question of the origin of stable maghemite in archaeomagnetic material.

6.2 Comparison with other available archaeomagnetic data

The new archaeomagnetic data obtained have been compared with other available data from neighbouring regions by compiling the archaeomagnetic data available for the last two millennia from Europe and North Africa. Historical volcanic Italian data have not been included due to the often present dating uncertainties and/or difficulties identifying with certainty the eruptive products from specific volcanic events (e.g. Tanguy et al. 2003; Tema et al. 2006). The archaeomagnetic compilations of Donadini et al. (2009) and Genevey et al. (2008) have been supplied with recent data (Ben-Yosef et al. 2008; Casas et al. 2008; Catanzariti et al. 2008; De Marco et al. 2008; Donadini et al. 2008, 2010; Gómez-Paccard et al. 2008; Herries et al. 2008; Peters et al. 2008; Ruiz-Martínez et al. 2008; Sapin et al. 2008; Suteu et al. 2008; Tema & Lanza 2008; Gallet et al. 2009; Genevey et al. 2009; Hartmann et al. 2009; Kovacheva et al. 2009a; Márton 2009, 2010; Nachasova & Burakov 2009; Schnepp et al. 2009; Tema et al. 2010). The location of the archaeomagnetic sites is plotted in Fig. 1. Only seven direction data (Kovacheva 1984; Najid 1986) and four archaeointensity values (Kovacheva 1984; Casas et al. 2008) have been published for Morocco. For Tunisia no archaeomagnetic data are available for the last 2000 years but one intensity data from Libya close to the Tunisian border is included in the study of Odah et al. (1995).

Two reference locations (Rilha in Morocco and Sabra al-Mansuriya in Tunisia) and two restricted regions of 800 km of radius centred at both locations have been defined (Fig. 1). The western selected area contains archaeomagnetic data from the Iberian Peninsula (described in Gómez-Paccard et al. 2006c, 2008) and the available Moroccan data obtained close to the Rilha archaeological site (Supporting Information). For the region centred at Sabra al-Mansuriya, all data come from Italy (see the compilations of Tema et al. 2006, 2010) except the one located at Libya (Odah et al. 1995). Directional and intensity data have been relocated to the corresponding reference location via the virtual geomagnetic pole and the virtual dipole moment (VDM), respectively (Fig. 10). The new data (directions and intensity) obtained for Morocco are in good agreement with the compiled data selected for the Rilha region (Fig. 10a, and Supporting Information). For the Sabra al-Mansuriya region no directional or intensity data are available covering the intervals of interest of this study (Fig. 10b).

6.3 Comparison with regional and global geomagnetic field models

The new data obtained can be compared with global and regional models of the Earth’s magnetic field. For comparison the global model ARCH3k.1 has been used. This model belongs to the last series of the Continuous Archaeomagnetic Lake Sediment family of models (Korte et al. 2009) developed using the spherical harmonic analysis. The ARCH3k.1 model is based on a global compilation of archaeomagnetic and volcanic data distributed all around the world (Donadini et al. 2009). These models represent the palaeofield at a global scale, but they are usually too smooth to represent rapid changes of the Earth’s magnetic field (Pavón-Carrasco et al. 2009).

An intermediate approach between local PSV curves (not available for Morocco and Tunisia) and global geomagnetic models is the
determination of regional models. Recently a new regional spherical cap harmonic model has been developed (Pavón-Carrasco et al. 2009). This SCHA.DIF.3k model is based on archaeomagnetic data (volcanic data are not included) from the European continent and neighbouring areas and is valid for Europe, North Africa and western Asia. These approaches (SCHA.DIF.3k and ARCH3k.1 models) may be the best possible solution to describe the long-term geomagnetic variations in Northwest Africa.

The results derived from the global (ARCH3k.1) and regional (SCHA.DIF.3k) geomagnetic field models are represented in Fig. 11 at Rirha and Sabra al-Mansuriya coordinates. Both the regional and global models predict similar variation of the Earth’s magnetic field at both locations. The models fit reasonably well the directional data available for the Rirha (Fig. 11a) and Sabra al-Mansuriya (Fig. 11b) regions, taking into account the dispersion observed.

It is worth mentioning the large inclination difference (more than 20°) between the data obtained by Márton et al. (1992) for the beginning of the fifth century AD and other data and model results available for this period (Fig. 10b and Supporting Information). The mean archaeomagnetic direction obtained by Márton et al. (1992) is very similar to the one obtained from the same archaeological structure and reported by Evans & Hoye (2005), which is in fact a revised version of a previous study (Evans & Mareschal 1987). This supports the reliability of the palaeomagnetic direction published by Márton et al. (1992). However, the anomalous high inclination and the large age uncertainty reported for this structure indicate that the mean age ascribed might be not well constrained. Archaeomagnetic results suggest that this structure is probably younger than the mean age proposed.

It should also be highlighted that these two (or even three) archaeomagnetic results corresponding to the same archaeological structure are reported separately in the available databases (Tema et al. 2006; Donadini et al. 2009). As it is really difficult to control all the available archaeomagnetic data, future publications including a revision of older studies must state clearly the archaeomagnetic data that have been updated. Data repetition and dating inaccuracies must be avoided when constructing regional SV curves or geomagnetic field models if accurate estimations of geomagnetic field changes are to be obtained.

Considering the complete archaeointensity data set it seems that geomagnetic field models underestimate intensity values at Rirha coordinates between the 11th and 17th centuries AD (Figs 10a and 11a). All the archaeointensity data selected for this region have been obtained from Thellier or Thellier derived methods, except the one obtained by Casas et al. (2008) which was obtained by microwave experiments, and generally are based on more than six samples per site (Supporting Information). Therefore they can be considered highly reliable. The majority of the archaeointensity compiled for this region are not included in the construction of the regional SCHA.DIF.3k or global ARCH3k.1 models. Therefore the recently obtained Spanish archaeointensities (Gómez-Paccard et al. 2008) as well as the new African data obtained here will improve the
accuracy of future geomagnetic field models in Northwest Africa and Southwest Europe.

For the Sabra al-Mansuriya region the models results fit reasonably well the new archaeointensity data obtained (Fig. 11b). The low number of previous intensity data for this region prevents a reliable comparison for the overall period. However, it can be seen that some of the archaeointensity data obtained by the Shaw technique (Evans 1986) gives ‘anomalous’ high intensities for the 1st century AD (Figs 10b and 11b). This high intensity period is not observed in the more numerous south European data. Some of these data are also based on a very low number of independent samples (one or two) (Supporting Information). Chauvin et al. (2000) mention that the validity of some of the published intensity data is often disputable. Therefore, these high intensities values for the 1st century AD might not be reliable. More data are needed to infer a robust description of geomagnetic field intensity changes in Tunisia.

Finally, it should be noted that predictions of geomagnetic field models in Northwest Africa could be affected by the low number of data available for this region—especially archaeointensity data. However, some discrepancies may also be due to the fact that geomagnetic models use all of the archaeointensity data available without a pre-selection process based on their quality. Overall the results highlight the need to obtain more reliable data for North Africa, such as the new data presented here.

7 CONCLUSIONS

An archaeomagnetic study of archaeological kilns from Tunisia and Morocco yielded six well-defined site-mean characteristic remanence directions of magnetization and six new robust and highly reliable archaeointensity data, corrected for both the effect of TRM anisotropy and cooling rate upon TRM intensity acquisition. The results showed that TRM anisotropy and cooling rate corrections are significant in the determination of the mean site intensities in two of the six structures studied, with differences of 4.4 and 5.8 per cent between uncorrected and corrected mean intensities indicating that these corrections must be investigated when studying archaeomagnetic materials if accurate archaeointensity determinations are to be obtained.

Rock magnetic analysis showed that the principal magnetic carriers are magnetite and low Ti titanomagnetite, along with thermally stable maghemite and a high coercivity phase with low unblocking temperature. Both this high coercivity phase and the thermally stable maghemite contribute to the characteristic magnetization of the kilns and behave ideally during archaeointensity experiments.

The comparison between the new data obtained, previously available data from nearby regions and geomagnetic field models results (Korte et al. 2009; Pavón-Carrasco et al. 2009) show a general broad agreement. However, some differences are observed between intensity data and models results for the Moroccan region. The results also suggest that high intensities of about 65 μT occurred in Tunisia during the 9th century, in agreement with European archaeomagnetic data that indicate that a rapid intensity fluctuation occurred in Europe around 800 AD. The results highlight the need to obtain more data for North Africa if a reference SV curve valid for this region wants to be obtained, such as the new data obtained here. They are also suggestive of the requirement of performing

Figure 11. Comparison of the data obtained in this study (in red) with geomagnetic field models results. Values predicted by the regional SCHA.DIF.3 (Pavón-Carrasco et al. 2009) and global ARCH3k.1 (Korte et al. 2009) are showed in grey and blue, respectively.
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SUPPORTING INFORMATION

Additional Supporting Information may be found in the online version of this article:

Table S1. Archaeomagnetic directional data for the last two millennia compiled within a restricted region of 800 km of radius around Rirha.
Table S2. Archaeointensity data for the last two millennia compiled within a restricted region of 800 km of radius around Rirha.

Table S3. Archaeomagnetic directional data for the last two millennia compiled within a restricted region of 800 km of radius around Sabra al-Mannsuriya.

Table S4. Archaeointensity data for the last two millennia compiled within a restricted region of 800 km of radius around Sabra al-Mansuriya.

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