REMANENT MAGNETIZATION IN PORTLAND-CEMENT-BASED MATERIALS

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Summary: The purpose of this research is to demonstrate the possibility of applying one geophysical method to studying untraditional systems as is the case with Portland-cement-based materials. The research demonstrates how conventional paleomagnetic methodology can be employed in studying the mode of magnetic recording in present-day industrial materials. Portlandcement admixtures such as fly ashes and furnace slags should be discriminated, because those particles interact in soils and sediments in nature. Moreover, a better undertanding of magnetic remanent acquisition in model materials can serve to improve the interpretation of magnetic remanent acquisition in natural rocks formed a long time ago. The magnetic constituents of Portland-cement paste and mortar acquire a magnetic remanence due to their alignment with the earth's magnetic field at the casting place. This magnetization can be measured using ordinary paleomagnetic techniques. The alignment of the individual magnetic particles accounts for the intensity of the magnetic remanence, which can be increased by adding water and by vibration before setting and hardening. Blast furnace slag admixtures also add to the enhancement of the intensity of remanence. The magnetization of Portland-cement-based materials shows a near linear relationship with the water /cement (w/c) ratios employed in the experimental work; the w/c ratios range between 0.2 - 0.6 in pastes and 0.3 - 0.6 in mortar. Stable remanent magnetization was obtained during the first seven days of setting and hardening, a period necessary for magnetic particles to become locked parallel to the earth's magnetic field. The stability of magnetic remanence predicts the usefulness of the methodology in studying the properties of Portland cement and particularly in the control of ironbearing admixtures.

Keywords: Paleomagnetic methods, industrial materials, chemistry, iron-bearing admixtures, water/cement ratio, furnace slag, magnetic susceptibility, remanent magnetization

1. INTRODUCTION

There are few works concerning the study of remanent magnetization acquisition in industrial materials such as Portland cement, but the characteristics of the remanent magnetization and the possibility of measuring this parameter by conventional paleomagnetic techniques may be useful in studying industrial materials.

Portland cement used in building and civil engineering is essentially a calcareous cement. It is made primarily of calcareous material, such as lime-stone or chalk, and from alumina and silica found as clay and shale. Marl, a mixture of calcareous and argillaceous materials is also used. These raw materials interact in the kiln, between 1300 and 1400°C, to form series of more complex

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products, and chemical equilibrium is reached, with the exception of a small residue of uncombined lime, which has insufficient time to react. However, equilibrium is not maintained during cooling, and the rate of cooling affects the degree of crystallization and the amount of glass in the cooled product (*Neville*, 1963). The properties of glass differ from those of crystalline compounds of a similar chemical composition; hence, the rate of cooling is very important in manufacturing Portland cement. According to the same author, there is another difficulty due to the interaction of the liquid part of the clinker with the existing crystalline compounds. The calculation of the compound composition of commercial cement assumes, in fact, that the cooled resulting product reproduces the existing equilibrium at the clinker temperature. Consequently, the composition is referred to as a potential composition, and it is calculated from the measured quantities of oxides present in the clinker, assuming full crystallization of the equilibrium product. The major constituents of Portland cement, determined as oxides of elements, are shown below with the abbreviation, used by cement chemists, in parentheses.

Tricalcium silicate	$3CaO.SiO_2$ (C ₃ S)
Dicalcium silicate	$2CaO.SiO_2(C_2S)$
Tricalcium aluminate	$3CaO.Al_2O_3$ (C_3A)
Tetracalcium aluminoferrite	4CaO.Al2O3.Fe2O3 (C4AF)

The clinker is cooled and ground into fine powder, and gypsum is added, the resulting product being the commercial Portland cement widely used throughout the world. Since hydration starts at the surface of the cement particles, it is the total surface area of the cement that represents the material available for hydration and the rate of hydration depends on the fineness of the cement particles. Besides, others technical properties of Portland cement are related to its grade of fineness. According to *Neville (1963)* the finer the cement, the more rapidly it deteriorates on exposure to the atmosphere. On the other hand finer cement leads to a stronger reaction with alkali-reactive aggregate, and pastes exhibit higher shrinkage and greater proneness to cracking.

Concerning the subject of this paper, it must be pointed out that the limestones and clays, used as raw materials, are particularly poor in magnetic minerals, but iron is present in the composition of one of the major compounds of Portland cement, i.e. tetracalcium aluminoferrite. Besides, blast furnace slags, used as admixtures, seem to contribute to the acquisition of remanent magnetization.

Paris (1973) considers that elemental iron can be introduced by the steel balls in mills during the clinker grinding process, being thus added to the raw material. He claims that iron particles could be free to rotate and align with the earth's magnetic field direction while the Portland-cement paste and mortar are essentially in a fluid state. According to the mentioned author and *Farrel et al (1991)*, the grains will no longer be free to rotate when the material becomes dry and hard. At some time during the setting process, particles are locked in place in a way similar to natural conditions in sediments.

Farrel et al. (1991), in agreement with Paris (1973), consider that the magnetic carrier of remanence in Portland cement is elemental iron introduced during clinker grinding. These authors also point out that the torque which aligns the magnetic grains, while the material is still fluid, is produced by the ambient magnetic field, and that this field must overcome inertia, viscose and electrostatic forces.

Games (1970) working with adobe bricks (unburned bricks dried in the sun) found that their ferromagnetic minerals were capable of recording the earth's field direction. He claims that mineral particles in clays could rotate while the material has appreciable fluidity.

2. MATERIAL CHARACTERISTICS

Two different types of normal Portland cement were employed in the preparation of pastes and mortar for the present research. The first (C1) without blast furnace slag and the second (C2) with a content of 9,5% of blast furnace slag type C. Magnetic measurements have also been carried out on pastes of C2 with higher slag contents.

2.1. Chemistry

Table 1 shows the chemical composition of the employed Portland cements (C1 and C2) expressed as oxide weight percentages according to the standard method for chemical analysis of hydraulic cement ASTM C114-85. The total iron determination, calculated as Fe_2O_3 , indicated a higher concentration in C1 than in C2. In addition, the concentration of FeO was calculated using the Wilson method (*Wilson*, 1955), modified by *Bidegain et al* (1995). The FeO percentage in C1 is lower than in C2 (0.02% and 0.13%, respectively). The results of the chemical analysis carried out on the blast furnace slags employed in the present study are also indicated.

2.2. Mineralogy and Grain Size of Materials

Microscopic studies of clinkers from different factories in the Buenos Aires Province have been carried out by *Cortelezzi et al. (1965)*. The authors pointed out the low content of iron oxides in the analyzed material and the presence of $Ca_3Al_2O_6$, Ca_3SiO_5 , Ca_2SiO_4 with a preponderance of the last compound.

No microscopic studies have been performed in the present study. However, micrometric microscopy was employed in order to determine the grain size of the blast furnace slags, added in the preparation of C2 cement. The most representative sizes for small and large slag grains were 13 μ m and 22 μ m, respectively. Detailed counting of grains, in order to evaluate the prevalence of one size with respect to the other, has not been carried out yet, however, it must be pointed out that the small fraction is clearly dominant. XDR diffractograms of the C1 and C2 pastes indicated that C1 contains gypsum, calcite, Ca₂SiO₄, hematite and Ca₅Si₂O₇(CO₃)₂, while C2 contains Ca₂SiO₄, gypsum and Ca₅(SiO₄)₂CO₃. Calcite formation in C2 has not been determined using this method.

Oxides in %	Normal Portland cement (C1)	Portland cement with 9.5% of blast furnace slags (C2)	Blast furnace slag
SiO ₂	21.6	21.6	37.56
Al ₂ O ₃	4.1	6.2	16.00
Fe ₂ O ₃	4.2	3.7	0.84
CaO	63.7	58	39.16
MgO	0.52	3.6	4.12
K ₂ O	1.08	0.82	0.59
Na ₂ O	0.13	0.60	0.59
SÕ3	2.6	2.00	-
Sulfuro	-	-	0.30
UM*.	0.60	2.18	-
LI**	1.47	1.30	0.70
Totals	100	100	100

Table 1. Chemical composition of Portland cements C1, C2 and blast furnace slag.

Unsoluble matter

** Loss on ignition

The standard method for testing the fineness of hydraulic cement, the Blaine Method (ASTM 204-96a) was also performed on C1 and C2. Values of $296m^2/kg$ and $311m^2/kg$, respectively, were obtained.

In the preparation of mortar, Argentine river sand has been used. The mineralogical composition of the Argentine river sand is: I) quartz: 95.5%; II) potassic feldspar: 1.5%; III) chalcedony: 0.5%; IV) limonite: 1.5%; V) opaque minerals 1%, integrated by magnetite partially altered to hematite. The grain size of the employed Argentine river sand is shown in Table 2.

3. PASTE AND MORTAR PREPARATION

Hydrated samples of C1 and C2 were prepared in plastic molds of 13x11x 6 cm using distilled water in different water/cement (w/c) ratios. These ratios represent the weight percentage (i.e. w/c = 0.2 means 200g water per 1000g cement). Blocks of paste of C1 and C2 were prepared using w/c ratios of 0.2, 0.3 and 0.4 for C1, and 0.2, 0.3, 0.4 and 0.5 for C2. These w/c ratios are the most common proportions in industrial tests of Portland cement. Suitable samples for magnetic measurements were also obtained from blocks of pastes with 19.5 and 44.5% furnace slag.

Portland cement C2 with 0.4, 0.5 and 0.6 w/c ratios were used to prepare the mortar. Mortars were prepared employing Argentine river sand at the standard ratio of 1/3; i.e. one part cement to three parts sand.

The pastes and mortar were mixed according to the standard practice for mechanical mixing of hydraulic cement pastes and mortars of plastic consistency ASTM C 305-94. The mixture was put in the plastic molds mentioned and vibrated for 2 minutes on a vibrating table.

Setting in place and the subsequent hardening took seven days. The mixture, which undergoes an exothermic reaction (characteristic of Portland cement materials), was kept hermetically sealed for curing under normal pressure and room temperature, during this period. The magnetic north direction was determine with a Brunton compass and marked on the upper surface of the blocks; this mark was translated to all cores and samples.

After hardening for seven days the blocks of paste and mortar were drilled, using a 25 mm inner diameter drill core. Six cores were obtained from each block.

Sieve Number ASTM C897-83	Sieve Size (mm)	% Cumulative
4	4.75	
8	2.38	1
16	1.18	2
30	0.60	4
50	0.30	50
100	0.15	98
	<0.15	2

Table 2.	Grain size	determination	performed	on dry	Argentine	river	sand
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Fig. 1 shows an oriented core cut from one block and a sample obtained from the central part of the core. The central part of the core was sampled because it is less disturbed and thus more suitable for magnetic measurements.

It is worthwhile mentioning, that it was impossible to obtain suitable samples for paleomagnetic measurement from the C2 blocks with a w/c ratio of 0.2. The material becomes friable with this water/cement ratio, and the cores were destroyed by drilling.

In order to obtain a stronger magnetic field to get a higher signal, cement molds of C2 Portland-cement paste were placed in Helmholtz coils, oriented parallel to the earth's magnetic field. A current of 2 A was applied and a magnetic field of $126.2 \pm 0.2 \,\mu\text{T}$ was achieved. The molds remained hermetically sealed within the coils for 24 hours; they were then taken apart for curing and hardening, under the influence of the earth's magnetic field for six days. This method was applied in order to demonstrate that the magnetic remanence is acquired during the first day of casting.

4. MEASUREMENT OF MAGNETIC PARAMETERS

The intensity and direction of the earth's magnetic field at the casting place of pastes and mortar (La Plata, Buenos Aires, Argentine), at the moment of preparation of the molds were: intensity = $23.7 \,\mu$ T; declination = -5.7° ; and inclination = -36.3° .

Magnetic susceptibility was measured with a Bartington Susceptometer, model MS2. The susceptibility values obtained for the different samples are shown in Table 3. The susceptibilities of some samples were measured at low (0.47 kHz) and high (4.7 kHz) frequencies, but the results do not show differences larger than 2%.

The remanent magnetization (RM) was measured with a spinner magnetometer (Digico) and a cryogenic magnetometer (2G). Both of them have an accuracy as high as



Fig. 1. Sketch of the blocks of cement and the sampling method.

Cement Type	Sample	w/c ratio	Susceptibility (10^{-5} SI)
C1	C1-2-7	0.2	54
	C1-2-6	0.2	51
	C1-3-4	0.3	45
	C1-3-5	0.3	44
	C1-3-7	0.3	46
	C1-3-8	0.3	46
	C1-4-5	0.4	38
	C1-4-6	0.4	38
	C1-4-7	0.4	38
	C1-4-8	0.4	36
C2	A1-3-2	0.3	61.6
	A1-3-3	0.3	61.6
	A1-3-5	0.3	60.3
	2A2-3	0.4	55.3
	2A2-4	0.4	55.3
	2A3-2	0.5	50.3
	2A3-4	0.5	49.0
With blast furnace slag (19.5%)	E1-10-1	0.3	104.3
With blast furnace slag (19.5%)	E1-10-4	0.3	103.0
With blast furnace slag (19.5%)	E1-10-6	0.3	105.6
With blast furnace slag (44.5%)	E3-3	0.3	143.3
With blast furnace slag (44.5%)	E3-4	0.3	226.2

 Table 3. Susceptibility values for C1 and C2.

 10^{-4} Am⁻¹, suitable for low intensity samples. Values of intensity of *RM* (*M*), declination (*D*) and inclination (*I*) were obtained for each sample.

RM and stepwise alternate field (AF) demagnetization was performed using the static three orthogonal axis method with a demagnetizer enclosed in the cryogenic magnetometer up to a peak field intensity of 140 mT. Besides, stepwise thermal demagnetization was also carried out up to 600°C using a Schonstedt furnace. Both demagnetization were carried out on 10 samples.

5. RESULTS

Table 3 shows that the susceptibility values decrease with increasing w/c ratio for C1 and C2 samples and increase with addition of furnace slag for samples with the 0.3 w/c ratio corresponding to C2. The difference between the susceptibilities at low and high frequency is negligible (about 2%).

The material with a w/c ratio of 0.4 displays better behavior with less scattering of results. For this reason this kind of material was studied more exhaustively.

Table 4. I	ntensity v	alues of <i>RM</i>	for C2	samples	with an	0.30 w/o	ratio.
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Material	RM (Am ⁻¹)
	In the earth's magnetic field
9,5% furnace slag admixture (C2)	$7 - 13 \times 10^{-3}$
	In a field of 126.2 µT
9,5% furnace slag admixture (C2) 19.5% furnace slag admixture 44.5% furnace slag admixture	$34 - 46 \times 10^{-3}$ 92 - 110 × 10^{-3} 105 - 139 × 10^{-3}

Different times of vibration were used in mold preparation, in order to study the effect of vibration on the obtained RM. Fig. 2a shows log(M) vs. the corresponding vibration time applied to the C2 Portland-cement paste with a w/c ratio of 0.4. The M values are lowest (8 to $18 \times 10^{-3} \text{Am}^{-1}$) at zero vibration time, they increase drastically up to $80 \times 10^{-3} \text{Am}^{-1}$ after 2 minutes of vibration, and reach the highest value of M for the applied field around this time. The directional parameters D and I are shown in Fig. 2b, where stereographic projections were made. The stereoplots show the position of the RMvector in the Northern Hemisphere, in agreement with the present earth's magnetic field direction at the setting place. According to the results, it is unnecessary to vibrate for more than 2 minutes because the lowest standard deviation is obtained at this time. Besides, with higher vibration times, the scattering is similar and M values do not increase (Fig. 2). For this reason 2 minutes of vibration time were used in this study. The directions were averaged using Fisher Statistics (*Fisher*, 1953).

Fig. 3 shows stereographic projections and Cartesian representations of intensity M of the remanent magnetization of samples of Portland cement C1 (a and b) and C2 (c and d) for different w/c ratios. RM intensities varied between 6 and 16×10^{-3} Am⁻¹ in the analyzed C1 Portland cement, and, the RM directions are close to the local present earth's magnetic field direction. The lower scattering of values in relation to those obtained in Fig. 2 is not yet clear. This might be due to the position of the mixing device being closer to the setting place and, consequently, the shorter time between mixing and setting. The best fitting for C1 was obtained with a linear relationship between M and w/c (slope: 45.1 Am^{-1} , R: 0.96).

It should be mentioned that for C2 it was possible to obtain cores from blocks with a w/c ratio of up to 0.5, because its higher specific surface favors higher water absorption capacity. The *RM* intensities varied between 5 and 40×10^{-3} Am⁻¹(Fig. 3d). The higher intensity may be caused by the higher content of furnace slag. Again a linear relationship fitted *M* vs. w/c ratios (slope: 143.2 Am⁻¹, *R*: 0.97).

It was difficult to cut blocks of mortar of C2 to obtain suitable samples for w/c ratios lower than 0.4; for this reason the molds were prepared with w/c ratios of 0.4; 0.5 and 0.6.



Fig. 2. Representation of intensity M (a) and the directions (b) of the remanent magnetization of samples of the C2 paste for different vibration times.

M of *RM* vs. w/c ratio is shown in Fig. 4a, and a higher concentration of the directions is observed in Fig. 4b. A linear relationship may also be fitted in Fig. 4a, (slope: 142.6 Am^{-1} , *R*: 0.97).

Fig. 5a shows the AF demagnetization behavior for one sample of the C1 paste with an 0.4 w/c ratio. The stereographic projection of directions of the remanent magnetization after each step of AF demagnetization is also indicated in Fig. 5b. It is easy to see that the progressive decay in remanence intensity does not affect the directional parameters causing an appreciable high concentration of directions.

Thermal demagnetization carried out on a sample of C2 paste with an 0.4 w/c ratio is also indicated in Fig. 5c. The intensity of remanent magnetization in each step, in relation to the initial intensity (M/M_0) , falls below 10% at around 500°C, which does not agree with the behavior of magnetite, probably due to the generation of other iron oxides, not yet determined by this method. Both demagnetization procedures suggest the presence of magnetic carriers with coercitive fields and Curie temperatures lower than magnetite. Unfortunately, it was impossible to heat the samples to temperatures higher than 600°C, because they were destroyed during the heating. Further research must be carried out in order to differentiate the iron oxides mentioned.

Fig. 6a shows M for those samples of the C2 paste, which were placed in a field of 126.2 μ T, vs. w/c ratios. These M values are higher than those observed in Fig.3 and 4, because M reflects the higher intensity of the applied field during the first hours of setting. The relationship is also linear (slope: 145.2 Am⁻¹, R: 0.96) and the scattering of directions is lower than in the previous figures, as indicated in the stereographic projection.



Fig. 3. Stereographic projections and Cartesian representations of intensity M of the remanent magnetization of samples of Portland cement C1 (a and b) and C2 (c and d) for different w/c ratios.



Fig. 4. Cartesian representation of intensity M (a) and stereographic projection (b) of the remanent magnetization of samples of the C2 mortar for different w/c ratios.

As mentioned above, higher values of M were observed in the C2 samples, probably due to the furnace slag addition. This property could also be appreciated by adding a higher amount of blast furnace slag to the C2 Portland cement; although no clear trend can be established yet. The M values obtained for samples with an 0.3 w/c ratio are summarized in Table 4.

6. DISCUSSION

Similarities and differences between remanent magnetization in sediments and in Portland cement materials have been found. The similarities concern the alignment of magnetic particles under normal pressure and room temperature conditions, and the differences appear in the mechanical process due to the vibration that Portland cement pastes and mortar undergo in the laboratory. A remarkable difference is also the exothermic nature of the chemical reaction in Portland cement. No experience, concerning this property, has been gained in relation to remanent acquisition in Portland cement. However, the intensity of remanence does not experience any measurable change over a period of months. It is important to point out this fact, since the release of the total heat produced during hydration takes a long time after setting.

The magnetic behavior of Portland cement paste and mortar under vibration is consistent with the results reported by *Farrel et al (1991)*. According to these authors, the magnetic torque acting on mineral particles is conditioned by opposite forces of inertia

and viscosity of Portland-cement materials from the beginning of the setting. On the other hand, if water is added, hydrodynamic forces favoring the alignment of the magnetic particles are generated.



Fig. 5. AF demagnetization behavior for one sample of C1 paste with an 0.40 w/c ratio (a) and its stereographic projection of the directions of the remanent magnetization (b). Representation of M/M_0 vs. temperature during thermal demagnetization for samples of C2, placed in a field of 126.2 μ T (c).



Fig. 6. Intensity (a) and directions - stereographic projection - (b) of remanent magnetization M for samples of the C2 paste placed in a field of $126.2 \,\mu\text{T} \pm 2.0 \,\mu\text{T}$, vs. w/c ratios.

In fact, hydrodynamic forces favor the magnetic orientation of grains parallel to the local field up to a w/c ratio, which is different for each employed material, and the scattering of directions becomes lower with higher w/c ratios (Fig. 3 and 4). Directional parameters D and I confirm that the preferential orientation of magnetic grains of Portland-cement paste and mortar is controlled by the earth's magnetic field.

As long as susceptibility decreases and RM increases with increasing w/c ratio, it is evident that the favoring of the alignment of the magnetic particles by adding water is a very important effect.

The decrease of susceptibility values with increasing w/c ratio for the C1 and C2 samples (Table 3) can be explained by the diamagnetic characteristic of water. The increasing proportion of furnace slag can explain the increase of susceptibility values with the addition of furnace slag to C2 (Table 3).

According to conventional paleomagnetic techniques employed in this research, a stable remanent magnetization exists; the magnetic carriers may be either ferromagnetic composites of the clinker, or particles, which are added during the grinding in the mills. This magnetization constitutes a magnetic memory, characteristic of each employed Portland cement, and varies with the preparation method, the w/c ratios and the intensity of the applied magnetic field. The negligible difference between the susceptibilities at low and high frequency suggests the absence of superparamagnetic materials.

Concerning the mineral carriers of remanence, more research into rock magnetism has to be carried out. Neoformation of ferromagnetic particles, closely related to the behavior of magnetite or some other iron oxide in a possibly reduced state, must be taken into

account. It must also be considered that in one environment with low susceptibility components, as in Portland-cement-based materials, both iron states (Fe^{3+} and Fe^{2+}) carried by the admixtures - although in low proportions - can contribute to the enhancement of magnetic parameters. However, in the Portland cements analyzed, the increase in susceptibility seems to be correlated with the higher amount of FeO. Chemical determinations indicate that FeO concentrations were 6.5 times higher in C2 than in C1.

On considering the results from Fig. 3, 4 and 6, it is evident that a proportionality exists between M and the w/c ratio. The slopes of linear regressions obtained from paste (Fig. 3d) and mortar (Fig. 4a) of C2, placed in the earth's magnetic field, and from the C2 paste placed in a field of 126.2 μ T (Fig. 6a) are similar, whereas the fitted slope for the C1 paste samples is lower. These results suggest that the slopes depend only on the material.

Besides, since the molds placed in an external field were removed from the Helmholtz coils after the first 24 hours and the curing and hardening continued during the subsequent days, the results suggest that the remanent magnetization was acquired during the first day of setting and of the early hardening.

7. CONCLUSIONS

This type of research, employing Argentine Portland cements, shows good agreement with an earlier study of *Farrel et al. (1990)* and introduces new technological and methodological aspects considered as appropriate for the study of the magnetic behavior of Portland-cement paste and mortar. This can be summarized as follows:

- 1. Ordinary Portland-cement paste and mortar become weakly magnetized with directions of magnetization parallel to the earth's magnetic field at the casting location.
- 2. Induced vibration of paste and mortar favors alignment of the magnetic particles with the ambient magnetic field. A vibration table was used at different times, and the highest values of intensity of remanent magnetization were observed during the first 2 minutes of vibration.
- 3. Portland-cement paste and mortar containing a higher amount of blast furnace slag acquire a significantly stronger magnetization.
- 4. Bulk susceptibility decreases when water is added, and increases with the increase of the content of blast furnace slags.
- 5. The w/c ratio in both paste and mortar maintains a near linear relationship with the intensity of remanent magnetization. The constant of proportionality between these two parameters depends on the material.
- 6. By increasing the magnetic field using Helmholtz coils, the intensity of remanence carried by Portland-cement paste increases appreciably. The relationship between intensity and w/c ratios remains.
- 7. The research shows the employment of conventional paleomagnetic methodology in studing the mode of magnetic recording in present-day industrial materials. Portland cement admixtures such as fly ashes and furnace slags should be discriminated because these particles interact with soils and sediments in the nature. Therefore, the work is an approach to studying magnetic remanence in sediments in relation to environmental and paleoenvironmental problems.

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8. The methology employed can also be developed for application in studying the archaeomagnetic aspect, as with ancient buildings. The better understanding of magnetic remanent acquisition in model material can contribute to a better interpretation of magnetic remanent acquisition in natural rocks formed a long time ago.

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