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SPECTROSCOPIC AND ANCIENT GEOMAGNETIC FIELD INTENSITY STUDIES ON ARCHAEOLOGICAL POTTERY SAMPLES, INDIA

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Spectroscopic and paleointensity studies have been performed on archaeological pottery samples from Mayiladumparai, Tamilnadu, India. The clay mineral type and its level of structural deformation due to firing were studied from their Fourier Transform Infrared (FTIR) Spectra. The maximum firing temperature attained during baking, firing conditions (open / reduced atmospheric) and iron mineral phase changes were well established. Intensive rock magnetic properties on these samples were carried out in order to select the samples for paleointensity measurements. The results showed that all the samples were magnetically enhanced having superparamagnetic grains with Curie temperature of magnetite (580 °C) and yielded mean paleointensity value of 48.71±0.16 μT.

Keywords: archaeological artifacts, FTIR and paleointensity

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1. Introduction

Pottery occupies a unique position by its distinguishing and characteristic, features as well as by its distribution in different areas and periods. Pottery is helpful for identification of a civilisation, inasmuch as it is an essential element to characterize it [1]. The value of pottery for studying the culture of a particular groups of people in a particular region and period is considerable. A careful study of pottery helps one to identify the different cultural groups and their influence on one another. Pottery, being the common man's property and a popular industry among the society, throws enough light on everyday life. The richness of pottery, judged by its fabric, polishing, glazing, slips, paintings, and decorative designs, reflects the economic conditions and the aesthetic tastes of the people who used it. The beginning and end of a particular culture or civilisation may also be determined with the help of pottery.

Clays, in fact, very rarely used as they are. The aim of the potter is to obtain a material characterised by a plastic state. This state occurs when the mixture can be moulded and formed without breaking the paste material. Once the forces of shaping are relaxed, the material maintains its structure and shape. The plastic state depends upon the proportions of clay, water, and non-plastic material (temper grains). Temper grains can be naturally present in the clay or added by potter during process of making a paste. If clays contain too much non-plastic temper, the potter will decant out of the coarse material concentrating the clay fraction and, in the end, he will need to add some temper again. Furthermore, a more complicated but quite common practice is to use two or more clay sources (characterised by different plasticity) as a base for a paste.

Clay, the chief ingredient used in pottery, is a fine-grained material that develops some ductile behaviour when mixed with water. The main components of clay are Si, Al, and water, which also contains small amounts of feldspars, biotite, carbonates, Fe-Ti oxides, as well as soluble salts and organic matter. The most common Fe minerals in clays especially important in archaeomagnetic studies are hematite, goethite, limonite, magnetite, and siderite [2]. The making of pottery in-

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volves different steps [3] – collection and preparation of the clay, modelling of the artifacts, surface finishing, decoration, drying, and hardening by heat. Clay firing is one of the earliest technological operations of mankind. Clay firing, if properly conducted with respect to material and process, produces exceedingly durable artifacts. Scientific studies are used to explore the technological development of the prehistoric man. The clay intended for making vessels is devoid of impurities and possesses fine plasticity, the vessels made out of it will be of a better quality.

The clay minerals as well as associated minerals including iron oxides undergo characteristic chemical and physical changes during firing which can be followed by different techniques like X-ray diffraction, scanning electron microscopy, differential thermal analysis, optical microscopy, Mössbauer studies, FTIR, etc. Of the various well-known methods of analysis, infrared absorption spectroscopy is a rapid, economical, and non-destructive physical method universally applicable for structural analysis of clay minerals. Infrared spectroscopy is a sensitive technique to monitor dyhydroxylation and dehydration of clay minerals [4]. On firing the clay materials the structural deformation of the clay minerals and associated minerals depend upon firing temperature and atmosphere, which can be followed by FTIR [5–8]. Knowledge of the firing temperature can be of value in other scientific investigations of ancient ceramics and kilns, such as thermo-luminescence and magnetic dating.

Archaeomagnetic study is primarily based on the determination of the paleointensity using baked clays. These clays usually carry a stable remanent magnetisation which has “fossilized” the direction and intensity of geomagnetic field at the time and in the place of its last firing [9, 10]. The necessary condition for determining the suitability of the materials for archaeomagnetic investigation is that they must be heated to high temperature, i. e. above Curie temperature of the respective minerals, which fossilize the earth’s magnetic field and its direction. The type of magnetic minerals (remanence carriers), their concentration and domain states are important factors in determining the reliability of the results possessed by the artifacts [9–11]. The artifacts are subjected to rock magnetic studies like magnetic susceptibility (frequency and temperature) and isothermal remanent magnetisation (IRM) acquisition in order to characterise the main magnetic phases and to select the most suitable samples for paleointensity measurements.

For the present study the archaeological pottery samples (broken body pieces of red ware / black and red ware) belonging to Iron Age were collected from Mayiladumparai (MPI) (lat. 12°32′N, long. 77°32′E) Krishnagiri District, Tamilnadu, India. Samples were subjected to FTIR and archaeomagnetic studies in order to reveal the firing temperature, firing conditions, mineral magnetic and paleointensity values.

2. Experimental methods

The FTIR absorption spectra were recorded in the frequency region of 4000–400 cm^{-1}, using model Paragon 500, Perkin–Elmer spectrophotometer with 16 scan mode by using standard KBr pellet technique. The accuracy of the measurement was ± 4 cm^{-1} in 4000 to 2000 cm^{-1} region and ± 2 cm^{-1} in 2000 to 400 cm^{-1} region. Magnetic susceptibility measurements were carried out using a dual frequency Bartington MS2B susceptibility meter at two frequencies (χ_{LF} at 0.47 kHz and χ_{HF} at 4.7 kHz).

The parameters, mass-specific frequency-dependent susceptibility χ_{FD} defined as $\chi_{\mathrm{FD}} = \chi_{\mathrm{LF}} - \chi_{\mathrm{HF}}$ and percentage frequency-dependent susceptibility $\chi_{\mathrm{FD}}\%$ defined as $\chi_{\mathrm{FD}}\% = (\chi_{\mathrm{LF}} - \chi_{\mathrm{HF}})/\chi_{\mathrm{LF}} \cdot 100$, are used to detect ultrafine (<0.03 μm) ferrimagnetic minerals lying in the superparamagnetic grain size [12, 13]. Temperature dependence of magnetic susceptibility χ–T (in air atmosphere) was measured using Bartington MS2WFB with high temperature furnace by heating samples up to 700 °C and cooling down to 100 °C in steps of 2 °C. Saturation isothermal remanent magnetization (SIRM) studies were conducted using pulse magnetizer (IM-10-30 Impulse Magnetizer, ASC Scientific, USA). Remanence intensity was measured with an Agico Molspin spinner magnetometer with a measuring accuracy of $\pm 2.4 \cdot 10^{-6}$ A/m. The paleointensity values are derived by subjecting the suitable samples to modified Thellier and Thellier method using Magnetic Measurements Thermal Demagnetiser (MMTD, UK).

3. Results and discussion

3.1. FTIR studies

Infrared absorption bands characteristic of clay minerals are observed in all the samples with deformed state. To study the level of deformation, the spectra of heated samples in laboratory furnace at 300, 500, and 800 °C is compared with the spectra of the received state. FTIR spectra of selected pottery samples MPI-1

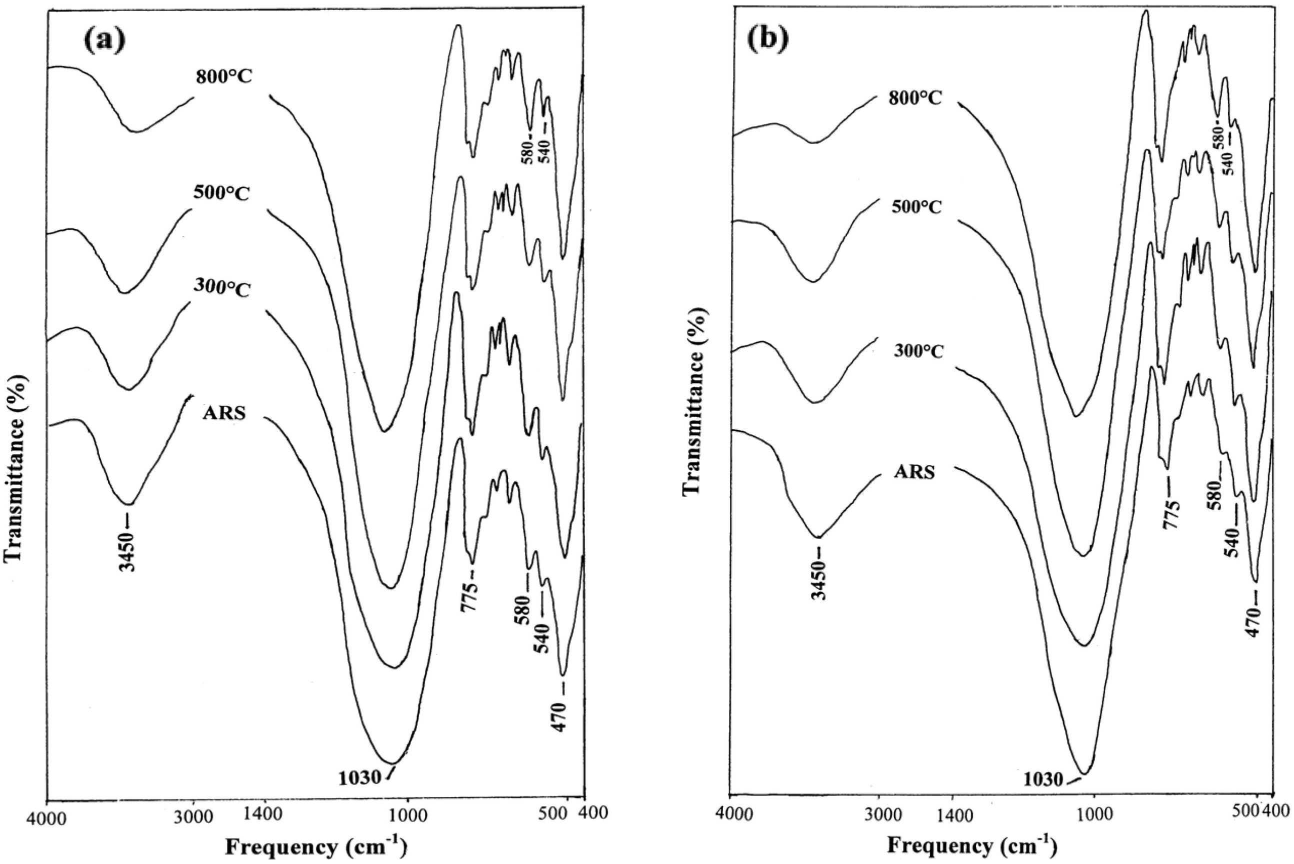

Fig. 1. FTIR absorption spectra of Mayiladumparai pottery samples: (a) MPI-1, (b) MPI-2.

and MPI-2 in as received state and refired at 300, 500, and 800 °C are shown in Fig. 1.

When clay is fired between 300–500 °C, dehydroxylation of octahedral layers of most clay minerals takes place [14]. The onset of dehydroxylation is reflected by marked attenuation of the bands due to Al Al–OH in-plane vibration at 920 cm^{-1} and translational vibrations involving OH at 522 cm^{-1} [7]. At 600 °C, the silicate structure collapses and a broad symmetry band is observed at 1030 cm^{-1} for red clay and 1080 cm^{-1} for white clay type [15]. Venkatachalapathy et al. [6] have established the dehydroxylation and destruction of layer silicates of clay minerals by firing model clay (Nathikudi) in steps from 50 up to 800 °C. The intensity of hydroxyl bands at 3700 region, 1100, and 920 cm^{-1} decreases and disappears in between 400–600 °C, due to the onset of dehydroxylation. The appearance of the 540 and 580 cm^{-1} bands attributed to iron oxides at 600 °C, with marked attenuation of the 530 cm^{-1} band in between 400–600 °C is well established. Wagner et al. [14] reported that the formation of crystalised hematite in clays have been observed in between 700 and 900 °C, from the Mössbauer spectral analysis.

The presence of iron oxides in the region of 700–400 cm^{-1} formed during the firing process of clay is due to the replacement of aluminium by iron. Some iron oxides that do not normally occur in natural clays may form during firing of pottery. Magnetite may form during firing under reducing condition [8]. On refiring the pottery in laboratory (oxidising) condition, reoxidation of iron oxides takes place in between 400 and 600 °C, which is reflected by the changes in intensity of the bands at 540 and 580 cm^{-1}. The formation of crystalline hematite is observed above 800 °C. Ishii and Nakahira [14] point out that the peak appearing at 572 cm^{-1} is due to Fe–O vibration of iron oxide, but exact nature of oxide could not be determined from the IR spectrum.

The presence of sharp band around 790 along with 695 cm^{-1} in all samples is due to the presence of quartz (Si–O) [4]. Quartz and feldspar are often present in pottery clays either because they are present already in the raw clay, or because they are added as temper. The bands occurring at 462 and 514 cm^{-1} are assigned to mixed Si–O–Si and O–Si–O bending mode [17]. The band at 470 cm^{-1} due to Si–O band is free from any temperature effects.

The absence of hydroxyl bands and the presence of broad symmetry band centred around 1030 cm^{-1} in the as received state spectra indicate that the samples MPI-1 (red ware) and MPI-2 (black and red ware) have been fired above 600 °C and made up of disordered clay [6, 7]. The well resolved and distinct peaks at 540 and 580 cm^{-1} in the spectra of as received state in both samples reveal the presence of iron oxides. It also confirms the firing temperature as above 600 °C [6]. The prominent peak at 540 and 580 cm^{-1} in sample MPI-1 reveals that the sample is fired under reduced atmosphere. At the same time, air has been allowed at a higher temperature during cooling which has enabled the oxidation of iron components formed during reduced atmosphere, the reason for the red colour of the pottery. Allowing air during cooling is a common practice for coloration of the baked clays. In the case of as received state spectra of sample MPI-2, the band at 540 and weak shoulder at 580 cm^{-1} indicate that the sample is fired under strong reduced atmosphere and air is admitted only at lower temperature, the reason for inner black and outer red colour surface of the sample.

On refiring the above samples, no appreciable changes are observed up to 500 °C and an increase in intensity of the bands at 540 and 580 cm^{-1} at 800 °C, indicating further oxidation and crystallisation of iron components.

From the FTIR studies, it is concluded that the samples MPI-1 and MPI-2 have been fired above 600 °C under different atmosphere (reducing followed by oxidising at higher temperature) and the clay used is of a disordered type.

3.2. Mineral magnetic studies

3.2.1. Frequency dependent susceptibility

Magnetic susceptibility χ describes the magnetic response of a sample when exposed to a (generally weak) magnetic field, and is mainly a function of the concentration, sample size, and mineralogy of the ferrimagnetic (magnetite, maghemite, Fe-sulphides) minerals present. It is customary to present susceptibility as mass-normalized susceptibility χ [18, 19].

In the present study, magnetic susceptibility χ_{LF} of pottery samples are more evenly spread over a wide interval ($15\text{–}73\cdot10^{-7}\,\mathrm{m^3/kg}$) pointing to higher magnetic enhancement. Higher magnetic susceptibility values are most probably due to high degree of firing. Magnetic susceptibility is a magnetic characteristic which depends mainly on the concentration of ferrimagnetic minerals [18, 20]. High values of χ_{FD} indicate the presence of very fine-grained metastable magnetic grains spanning the superparamagnetic–stable single domain (SP–SSD) boundary [21]. All the samples show $\chi_{\mathrm{FD}}\% > 2$ but most of the samples fall in between 4–10% suggesting the presence of significant amount of the superparamagnetic magnetite grains. Jordanova [9] has reported that burnt clay samples with $\chi_{\mathrm{FD}}\%$ values of 6–10% contain significant content of fine superparamagnetic magnetite grains of sizes 0.012–0.023 μm [22]. The investigator's results suggest that the pottery samples under investigation are magnetically enhanced materials in terms of concentration and degree of crystallinity of magnetite.

3.2.2. Temperature dependent susceptibility

Temperature dependent susceptibility measurements are widely used for the determination of main magnetic minerals (using their Curie temperatures T_{C}), which are responsible for acquiring ancient geomagnetic field, and the nature of mineral phase transformation, if any. Samples with basic reversibility of heating and cooling curves suggest no distinct mineralogical alteration occurred during heating, which are durable for paleointensity studies. Curves with irreversible behaviour are not suitable for paleointensity measurements [23, 24]. Jordanova et al. [9] have reported that the enormous increase in susceptibility values during cooling is most probably due to breakdown of clay minerals and formation of new strong ferrimagnetic phase indicating that these materials are not burnt to higher temperatures. The temperature dependent susceptibility curves for two representative samples are given in Fig. 2.

In the present investigation, the reversibility of the heating and cooling curves of all the MPI samples reveal that there is no distinct mineralogical changes and that magnetite, the main magnetic mineral, with Curie temperature around 580 °C is found. Hence the samples are suitable for paleointensity measurements. The presence of magnetite is also revealed by the drop of the initial susceptibility observed in the cooling curve, which is due to the oxidation of magnetite during heating to higher temperatures of 700 °C.

3.2.3. IRM acquisition curves

IRM is the remanent magnetization acquired by a sample after exposure to, and removal from a steady (DC) magnetic field. IRM depends on the strength of the field applied, which is often denoted by a subscript, and is also a function of the magnetic mineralogy and grain size. The maximum remanence that can be produced in a sample is called Saturation Isothermal Remanent Magnetization. IRM is often used as an indicator

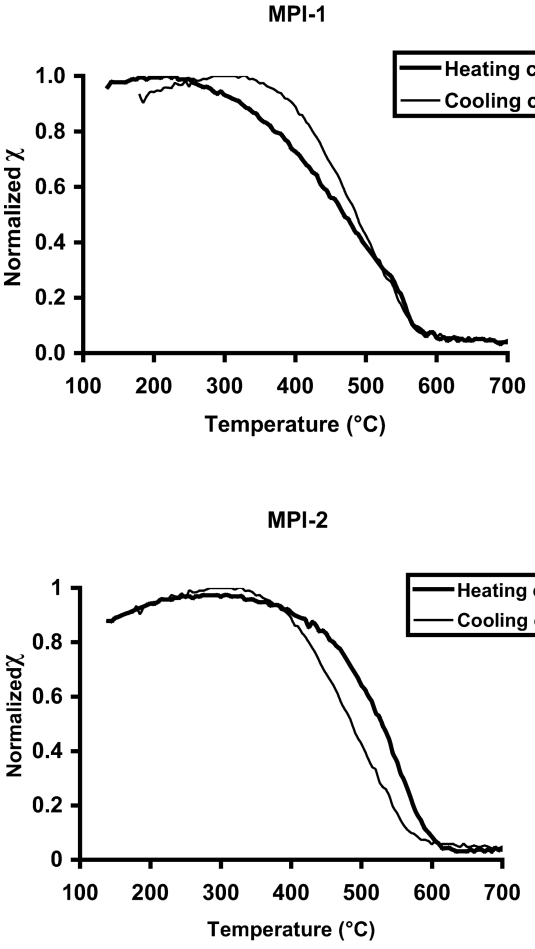

Fig. 2. Temperature dependent magnetic susceptibility.

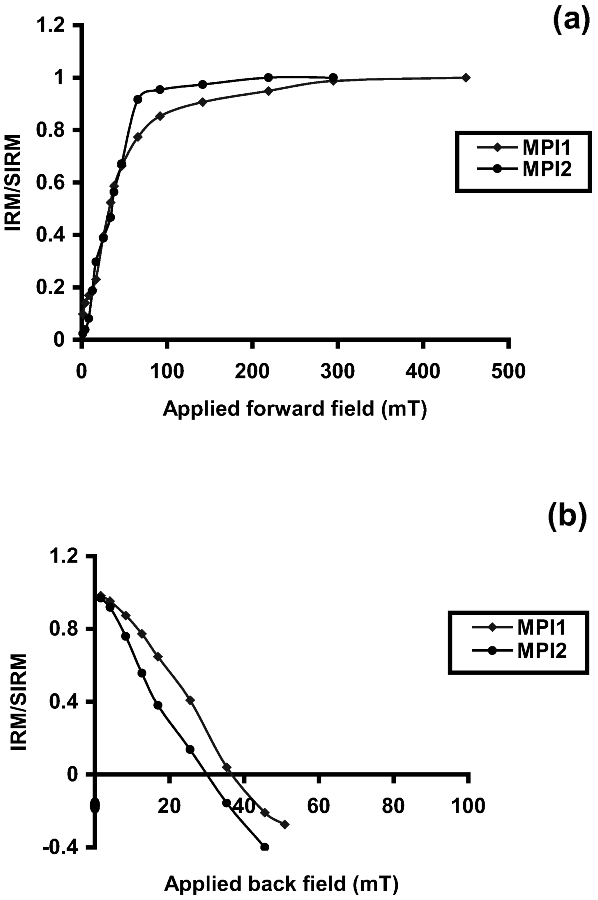

Fig. 3. (a) IRM acquisition and (b) back field DC demagnetisation curves.

for the presence of ferrimagnetic minerals, but also antiferromagnetic minerals, such as hematite and goethite, are also capable of acquiring an IRM. After a sample has acquired an IRM, it is often possible to (partially) demagnetize the sample by exposing it to a magnetic field in the reverse direction. Such a partial demagnetization can yield information about the ease of remanence acquisition, or the coercivity of a sample [19]. IRM acquisition curves give information about the coercivity distribution of a sample indicating the field at which the sample acquires its remanence. Tian et al. [23] and Zhu et al. [24] reported that the samples saturated by 300 mT and showed a remanence coercivity of about 30–40 mT, indicating magnetite, probably the main magnetic carrier. The IRM acquisition curves for two representative samples are given in Fig. 3. In the present investigation all the samples show remanence coercivity of about 30–40 mT suggesting that magnetisation is carried by low coercivity magnetic mineral such as magnetite / titanomagnetite with low Ti content. The S-ratio is $S_{100} = \mathrm{IRM}_{-100}/\mathrm{SIRM}$, where IRM_{-100} denotes an IRM acquired in a reverse field of 100 mT after SIRM acquisition. S-ratios can be used to gain information about the magnetic mineralogy [25]. S-ratios close to $+1.0$ are indicative of ferrimagnetic minerals while low S-ratios (<0.6 or even <0) are caused by the presence of antiferromagnetic minerals. The studied samples show S-ratio values >0.6, which reflect the presence of ferrimagnetic minerals.

The magnetic parameter Königsberg ratio, Q-ratio $= \mathrm{NRM}/\chi_{\mathrm{LF}} \cdot 0.5$ Oe, gives the type of mineral and its domain state that produce a dominantly induced remanent magnetization [26]. The magnetization value of 0.5 Oe corresponds to a magnetizing force of 39.79 A/m. The high Q-ratio values are characteristics of stable (thermoremanent) origin of NRM while low values ($Q < 1$) are found for non-stable remanence [21, 27]. The Q-ratios provide a relative importance of remanent and induced magnetization, being remanence-dominant for $Q > 1$. Variations in remanent intensity and susceptibility depend on the volume content of magnetite. The Q-ratio > 1 indicates the presence of single domain / pseudo single domain (SD / PSD) magnetite grains in the samples. Cui and Verosub [28] have

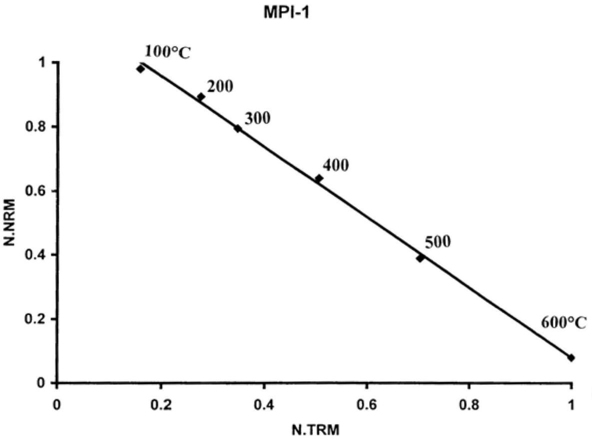

Fig. 4. Arai diagram of Mayiladumparai pottery MPI-1.

pointed out that in pottery samples a broad unblocking temperature distribution can arise from a grain size distribution that spans from superparamagnetic to single domain, and may be even to a pseudo-single domain. The broader the unblocking temperature distribution, the better the chances of retrieving the paleointensity before mineralogical changes occur.

3.2.4. Paleointensity measurements

Most paleointensity determinations using archaeological materials and lava flows are based on the modified Thellier and Thellier method [29] or modification of it [30, 31]. The modified Thellier and Thellier method involves heating the sample in a zero field to a number of increasing temperature stages and measuring the intensity of magnetization remaining after each stage – its natural remanent magnetization (NRM). Once all the magnetization is removed, the process is repeated but with the sample exposed to a known reference magnetic field and the magnetisation measured is thermoremanent magnetisation (TRM). No chemical alterations are observed during Thellier double heating experiments. Thellier [32] suggests that the baked earth is the most suitable material for paleointensity determinations. A unique advantage of the Thellier method is that, as the heating temperature is progressively increased, magnetomineralogical alteration can be detected directly during the experiment using standard partial thermoremanent magnetisation (pTRM) checks [33]. The Arai plot has been drawn with the measured NRM and TRM magnetisation values. The Arai plot for one representative sample is shown in Fig. 4. The slope of the best fitting line to the linear part of the Arai diagram multiplied by the value of reference field (laboratory field) gives the intensity of ancient geomagnetic field $B_{\mathrm{anc}} = \mathrm{NRM/TRM} \cdot B_{\mathrm{lab}}$ (μT), the value at the time of firing the samples.

The mean paleointensity value of the sample is found to be 48.71±0.16 μT. From the archaeologist point of view these samples belong to Iron Age (1000–500 BC), to obtain a more precise dating of the samples they can be subjected to ^{14}C dating.

4. Conclusion

Infrared spectroscopy has the potential to provide useful information on firing technique of pottery. The analysis of the samples shows that the shreds are fired above 600 °C under reducing / oxidising atmosphere and the type of clay used is disordered type. The main ferrimagnetic iron oxide minerals responsible for the magnetic susceptibility of burnt clay are magnetite with significant amount of superparamagnetic / single domain magnetite particles and hence suitable for paleointensity measurements. Remenance measurements, Q-ratio and S-ratio, point out the presence of ferrimagnetic mineral magnetite with SD / PSD state. The paleointensity value is found to be 48.71±0.16 μT.

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ARCHEOLOGINIŲ INDIJOS PUODŲ ŠUKIŲ SPEKTROSKOPIJA IR SENOVĖS GEOMAGNETINIO LAUKO STIPRIO TYRIMAS

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Santrauka

Atlikti archeologinių puodų šukių pavyzdžių (Mayiladumparai, Tamilnadu, Indija) spektroskopiniai ir senovės magnetinio lauko stiprio tyrimai. Apie molio mineralinį tipą ir sandaros pokyčius dėl degimo spręsta iš Furje transformuotų infraraudonųjų (FTIR) spektrų. Patikimai nustatyti maksimali degimo metu pasiekta temperatūra, degimo sąlygos (atmosferinės ar riboto sąlyčio su oru) ir geležies mineralinių fazių kitimai. Intensyviai tirtos šių pavyzdžių uolienų magnetinės savybės, siekiant atrinkti pavyzdžius senovės magnetinio lauko intensyvumo matavimams. Rezultatai parodė, kad visi pavyzdžiai buvo magnetiškai aktyvūs, su superparamagnetinėmis granulėmis, kurių Kiuri temperatūra (580 °C) tokia, kaip magnetito. Iš to seka vidutinė $48{,}71\pm0{,}16$ μT senovės magnetinio lauko stiprio vertė.