Hyperfine Interact (2008) 186:89–98
DOI 10.1007/s10751-008-9860-0

# Mössbauer and mineral magnetic studies on archaeological potteries from Adhichanallur, Tamilnadu, India

**R. Venkatachalapathy · T. Bakas · N. Basavaiah · K. Deenadayalan**

Published online: 9 October 2008


**Abstract** Megalithic potteries collected from Adhichanallur, Tamilnadu, India (Lat. 8°44′ N; Long. 77°42′ E) have been subjected to various spectroscopic and rock magnetic studies. The type of clay, their origin, level of structural deformation due to firing, firing temperature and atmospheric condition followed during making the potteries are analyzed. The potteries were subjected to Mössbauer and X-ray diffraction studies to analyze the iron phases in them. It is found that the samples were made of local clay (red clay), fired above 600°C under open atmospheric and/or reduced atmospheric conditions and air has been allowed during cooling. The Mössbauer spectra reveal the presence of $Fe^{3+}$, $Fe^{2+}$ and iron oxides of hematite and magnetite. The firing temperature and firing conditions established from Mössbauer studies are similar to the observation made from FT-IR studies. The magnetic mineral types, the mass fractions and the domain states of the constituent magnetic grains were elucidated from a range of rock magnetic measurements including variation of susceptibility with low field, frequency and temperature, hysteresis parameters and isothermal remanence acquisition data. The magnetic mineralogy of most pottery samples was dominated by magnetite/(titano) magnetite, while magnetic grain size spectrum varies from very fine (super paramagnetic) to fine (stable single domain, pseudo single domain). The reversible thermo magnetic behavior reflects no mineralogical transformations during reheating and all the samples show same Curie temperature 580°C due to magnetite. From the above information it is demonstrated that these samples are suitable for determining the reliable ancient geomagnetic field intensity values existed during that period.

R. Venkatachalapathy (✉)
C.A.S in Marine Biology, Annamalai University, Parangipettai 608 502, India
e-mail: venkatr5@rediffmail.com

T. Bakas
Department of Physics, University of Ioannina, 45110 Ioannina, Greece

N. Basavaiah · K. Deenadayalan
Indian Institute of Geomagnetism, New Panvel, Navi Mumbai 410 218, India

**Keywords** Archaeological pottery · Mössbauer spectroscopy · Rock-magnetism

## 1 Introduction

Archaeological pottery is considered to be the most important remains of ancient civilization. They are made of clays which consist of a mixture of different minerals mainly of hydrous aluminium silicates, iron oxides and oxyhydroxides. During firing the pottery, these minerals change their structure, they decompose and finally new minerals are formed. Different minerals in the ancient pottery carry information about the technique of processing, the firing temperature and the firing atmosphere. The combination of Fourier Transform Infrared Spectroscopy (FT-IR), XRD and Mössbauer spectroscopy has a considerable potential in the study of the chemical and physical transformation occurring in pottery clays during firing.

The magnetic properties of such pottery are used to establish curves for the temporal variation of the Earth's magnetic field in several geophysical regions. The magnitude of Earth's magnetic field in the historic and geological past provides essential data with which document the evolution of geomagnetic field. Rock magnetism is important for carrying out an archaeomagnetic study as well as palaeointensity experiment, because it gives valuable information for magnetic remanence carriers and provide tools for evaluating the suitability of the material for obtaining reliable results [1].

An attempt has been made to subject the megalithic burial urn pottery samples collected from Adhichanallur, Tamilnadu, India (Lat. 8°44′ N; Long. 77′42′ E) to FT-IR, XRD and Mössbauer spectroscopic studies.

To select the suitable samples for geomagnetic field intensity measurements range of rock magnetic measurements including variation of susceptibility with low field, frequency and temperature, isothermal remanence and hysteresis parameters were performed on the pottery samples.

## 2 Experimental procedures

FT-IR spectra of powdered pottery samples were recorded in the region 4,000°C to 450°C $m^{-1}$ using Paragon 500, Perkin Elmer spectrophotometer by standard KBr pellet technique. The resolution of the instrument is $\pm 4$ $cm^{-1}$ region. The Room temperature Mössbauer measurements were performed on seven representative samples with a conventional constant acceleration spectrometer (M/s. WISSEL, Germany) equipped with a $^{57}Co(Rh)$ source. The spectrometer was calibrated using a standard $\alpha$-Fe foil at room temperature. The spectra were fitted on a PC with a least squares minimization procedure assuming Lorentian line shapes.

To compliment the Mössbauer analysis the XRD spectra were recorded on X-pert MPD from Philips using Cu $K_\alpha$ radiation in the $2\theta$ range from 10° to 40° in steps of 0.02°.

Mass specific magnetic susceptibility $\chi$ was measured with the Bartington MS2B dual frequency meter ($\chi_{LF}$ at 0.47 KHz and $\chi_{HF}$ at 4.7 KHz) with measuring accuracy of $1 \times 10^{-5}$ SI. Percentage frequency dependent magnetic susceptibility ($\chi_{FD}\%$) defined as: $\chi_{FD}\% = \frac{(\chi_{LF}-\chi_{HF})}{\chi_{LF}} \times 100$, as well as mass specific frequency dependent susceptibility $\chi_{FD}$ were measured. Temperature behaviour of magnetic susceptibility was studied using Bartington MS2WFP in air atmosphere.

Anhysteretic Remanence magnetisation (ARM) was produced by a direct field (50 μT) and a maximum alternating field (AF) of 100 mT. AF demagnetisation was performed on a laboratory built tumbling AF demagnetiser and IRM using a Pulse magnetiser model MMPM9. Remanences were measured with an Agico JR6 spinner magnetometer. The magnetic hysteresis loops in a maximum field of 1,000 mT were obtained using a Molspin Vibrating Sample Magnetometer (VSM).

## 3 Results and discussion

### 3.1 FT-IR study

Infrared spectroscopy is considered to be an important tool in the study of structure of clay minerals and their deformation due to thermal effects. Infrared spectra on archaeological artifacts reveal the type of clay, temperature of firing and firing conditions (open/closed) adopted by artisans during making [2]. To establish the above parameters one can compare the infrared spectra of pottery finds with those of reference clay (local clay exploited by the ancient potters) fired in the laboratory under controlled conditions. The characteristic absorption bands due to clay minerals and their level of deformation due to firing were well established and compared with the fired archaeological artifacts [3].

For the present study FT-IR spectra of representative pottery samples (13 nos) from Adhichanallur in as received state along with local clay fired at 450°C, 600°C and 700°C under laboratory conditions were recorded.

The infrared spectra of archaeological pottery samples in as received state is well compared with the local clay and inferred that all the pottery were made up of local clay (disordered kaolinite) fired above 600°C under open atmospheric condition. Some samples ADC-14, 15, 16, 17, 18 & 20 were fired around 600°C and air has been admitted during cooling. The red colour of the pottery reflects the open firing and red and black pottery show the reduced atmospheric condition prevailed during heating and air has been allowed during cooling which results the inner black and red surface of the pottery [4].

### 3.2 XRD and Mössbauer studies

X-ray diffraction and Mössbauer spectroscopy complement each other particularly well because the former yields information on the major minerals in clays and pottery while Mössbauer spectroscopy looks selectively at the iron bearing compounds, which often are difficult or even impossible to detect by X-ray diffraction [5].

For the present study XRD spectra of Adhichanallur local clay in the as received state and fired at 450°C, 600°C and 700°C under laboratory conditions were recorded in order to identify the clay mineral present, their structural deformation due to firing. The presence of quartz, Anorthite, Albite, orthoclase, koalinite and Goethite were identified in as received state and above 450°C the absence of kaolinite and Goethite reflects the destruction of kaolinite clay structure and transformation of magnetic minerals. (Goethite to hematite/magnetite).

Representative Mössbauer spectra of Adhichanallur pottery samples are shown in (Fig. 1). The Mössbauer parameters like isomer shift, quadrupole splitting and magnetic hyperfine interaction derived from the peak position of the spectra are given in (Table 1). Mössbauer spectral studies on archaeological artifacts were elaborately carried out by many authors [6–9] in order to establish the presence of magnetic components, and their transformation due to firing, firing conditions and colouring mechanism.

Room Temperature Mössbauer spectra of as received state Adhichanallur pottery samples show similar patterns with one ferric doublet ($Fe^{3+}$), two ferrous doublets ($Fe^{2+}$) along with sextets arises due to $Fe_2O_3$ and $Fe_3O_4$, Which reflects that all the pottery samples contain perceptible amount of iron/iron oxide components. The presence/absence of paramagnetic $Fe^{2+}$ and $Fe^{3+}$ the firing and colouring mechanism of the archaeological pottery have been deduced [7]. The Mössbauer parameters like Isomer shift ($\delta$) value 0.189 to 0.342 mm/s, quadrupole splitting ($\Delta$) value 0.830 to 0.995 mm/s are attributed to the presence of $Fe^{3+}$ ions present in all the samples.

The $\delta$-values of 0.926 to 0.956 mm/s, 1.815 to 1.930 mm/s, and $\Delta$-values of 2.298 to 2.725 mm/s, 2.222 to 2.455 are attributed to the presence of two $Fe^{2+}$ species respectively. The lower percentage value of $Fe^{2+}$ and higher percentage of $Fe^{3+}$ indicates that the samples were not fired under strong reducing atmosphere even though they were fired to a moderate temperature of around 600°C. From the above observation it is confirmed that the samples were fired under changing atmosphere, air was admitted at the end of the firing cycle following the reduced atmosphere, and is the reason for red colour surface of the samples.

The $\delta$-value of 0.350 to 0.374 and $\Delta$-value of −0.189 to −0.239 mm/s with hyperfine magnetic field value of 508 to 510 kOe are attributed to the presence of hematite in all the samples.

Two sextets with $\delta$ value of 0.232 to 0.394 mm/s and 0.520 to 0.388 mm/s, $\Delta$ value of −0.089 to −0.217 mm/s and −0.012 to 0.050 mm/s with hyperfine field value of ∼ 490 and ∼460 kOe are attributed to the presence of magnetite in all the samples.

Iron oxides that do not normally occur in natural clays may form during firing the pottery under reducing conditions [4].

Similar Mössbauer parameters on archaeological artifacts were observed and possible firing temperature, firing conditions and colouring mechanism were well established [8].

Based on the above observations it is well established that all the Adhichanallur pottery samples were fired above 600°C under changing condition. The red colour of the pottery confirms the above statement. The sample ADC-17 & 18 having higher $Fe^{2+}$ value reflects that they are not fully oxidized. The air was admitted only at low temperature during cooling which is not sufficient to convert $Fe^{2+}$ into $Fe^{3+}$. The dull red colour of the pottery confirms the above statement

## 4 Mineral magnetic studies

The detailed mineral magnetic investigations are seldom employed in archaeointensity studies and however such investigations can be useful to screen the samples before the archaeomagnetic measurements are performed. The screening would be to identify the samples with thermally stable magnetic minerals occurring in a

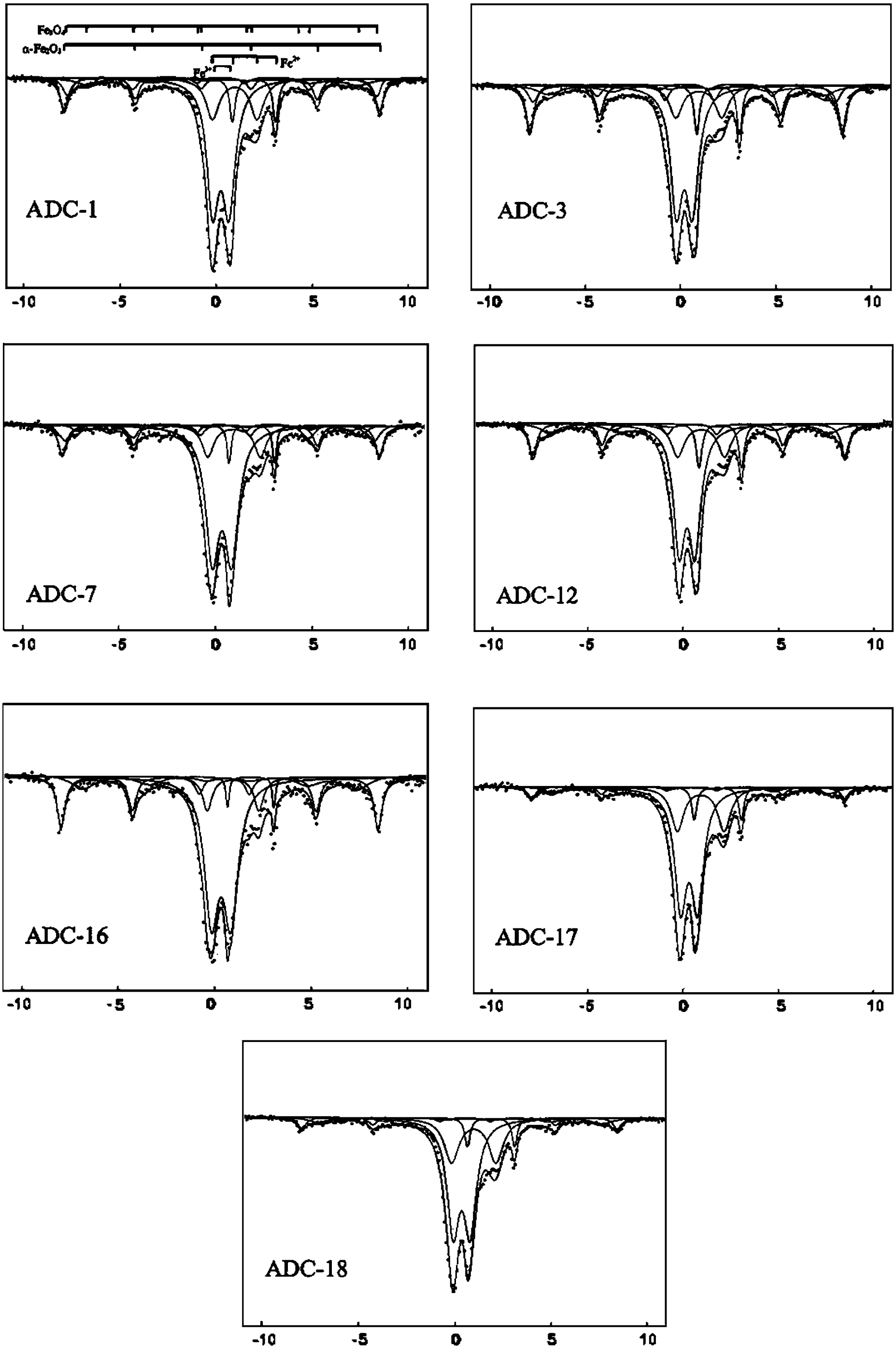


**Fig. 1** Room temperature $^{57}$Fe Mössbauer spectra of Adhichanallur potteries in the as-received state

**Table 1** Mössbauer parameters of Adhichanallur pottery samples in the as-received state

| Sample | $Fe^{3+}$ | | $Fe^{2+}$ | | $A-Fe_2O_3$ | | | $Fe_3O_4$ | | |
|---|---|---|---|---|---|---|---|---|---|---|
| | $\delta$ (mm/s) | $\Delta$ (mm/s) | $\delta$ (mm/s) | $\Delta$ (mm/s) | H (kOe) | $\delta$ (mm/s) | $\Delta$ (mm/s) | H (kOe) | $\delta$ (mm/s) | $\Delta$ (mm/s) |
| ADC-1 | 0.227 | 0.842 | 0.926 | 2.325 | 509 | 0.350 | −0.207 | 490 | 0.269 | −0.107 |
| | | | 1.923 | 2.247 | | | | 460 | 0.472 | −0.044 |
| ADC-3 | 0.189 | 0.845 | 0.926 | 2.415 | 509 | 0.350 | −0.205 | 491 | 0.292 | −0.217 |
| | | | 1.937 | 2.226 | | | | 462 | 0.388 | −0.029 |
| ADC-7 | 0.342 | 0.995 | 0.956 | 2.725 | 510 | 0.374 | −0.239 | 492 | 0.268 | −0.097 |
| | | | 1.869 | 2.355 | | | | 463 | 0.436 | −0.038 |
| ADC-12 | 0.206 | 0.830 | 0.926 | 2.458 | 508 | 0.355 | −0.189 | 490 | 0.292 | −0.091 |
| | | | 1.930 | 2.222 | | | | 458 | 0.482 | −0.017 |
| ADC-16 | 0.327 | 0.995 | 0.926 | 2.671 | 509 | 0.356 | −0.216 | 490 | 0.394 | −0.131 |
| | | | 1.834 | 2.403 | | | | 461 | 0.520 | −0.050 |
| ADC-17 | 0.315 | 0.894 | 0.926 | 2.454 | 510 | 0.366 | −0.192 | 491 | 0.232 | −0.089 |
| | | | 1.815 | 2.455 | | | | 451 | 0.458 | −0.012 |
| ADC-18 | 0.306 | 0.882 | 0.926 | 2.298 | 508 | 0.351 | −0.226 | 490 | 0.310 | −0.105 |
| | | | 1.820 | 2.453 | | | | 453 | 0.496 | −0.031 |

**Table 2** Summary of mineral magnetic parameters measured for Adhichanallur archaeological samples

| Sample | $\chi \times 10^{-7}$ $m^3$ /kg | $\chi_{FD\%}$ | NRM $\times 10^{-5}$ A.$m^3$ /kg | ARM $\times 10^{-5}$ A.$m^3$ /kg | SIRM $\times 10^{-2}$ A.$m^3$ /kg | $Q$ -ratio | ARM / SIRM $\times 10^{-3}$ | $S$-ratio$_{(-300\ mT)}$ | Soft IRM $\times 10^{-2}$ A.$m^3$ /kg | Hard IRM $\times 10^{-2}$ A.$m^3$ /kg | T °C | $M_{RS}$ / $M_S$ | $H_{Cr}$ / $H_C$ |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| ADC-1 | 35.89 | 6.18 | 46.18 | 31.47 | 3.31 | 3.23 | 9.51 | 0.6620 | 2.6092 | 1.1212 | 590 | 0.2108 | 2.8676 |
| ADC-2 | 31.44 | 5.38 | 68.99 | 46.25 | 3.16 | 5.51 | 14.64 | 0.7222 | 2.8015 | 0.8761 | 580 | 0.1713 | 2.8676 |
| ADC-3 | 75.06 | 3.71 | 205.73 | 146.89 | 8.37 | 6.89 | 17.55 | 0.9066 | 6.5382 | 0.7816 | 590 | 0.0782 | 2.8676 |
| ADC-4 | 64.21 | 6.87 | 55.59 | 58.60 | 4.38 | 2.18 | 13.38 | 0.9530 | 2.7160 | 0.2059 | 595 | 0.2597 | 2.7627 |
| ADC-5 | 72.43 | 7.09 | 53.25 | 60.59 | 4.44 | 1.85 | 13.65 | 0.9493 | 2.7647 | 0.2251 | 595 | 0.2818 | 2.7627 |
| ADC-7 | 42.15 | 7.37 | 61.28 | 44.88 | 2.36 | 3.65 | 19.02 | 0.8811 | 1.6829 | 0.2807 | 595 | 0.1604 | 3.5455 |
| ADC-12 | 52.70 | 6.84 | 72.31 | 30.61 | 2.65 | 3.45 | 11.55 | 0.8333 | 2.2084 | 0.4422 | 590 | 0.1015 | 5.4333 |
| ADC-13 | 35.81 | 8.71 | 32.57 | 18.45 | 0.92 | 2.29 | 20.05 | 0.7784 | 0.8028 | 0.2047 | 580 | 0.0538 | 5.9375 |
| ADC-14 | 13.19 | 9.17 | 11.52 | 11.77 | 0.75 | 2.19 | 15.69 | 0.7052 | 0.6618 | 0.2208 | 590 | 0.0769 | 5.8696 |
| ADC-16 | 25.86 | 5.94 | 84.89 | 38.55 | 2.87 | 8.25 | 13.43 | 0.6089 | 2.1040 | 1.1242 | NT | 0.1613 | 2.8261 |
| ADC-17 | 59.67 | 10.54 | 104.31 | 57.54 | 5.38 | 4.39 | 10.70 | 0.6144 | 4.1954 | 2.0734 | NT | 0.3257 | 3.0000 |
| ADC-18 | 55.62 | 10.46 | 98.63 | 50.64 | 4.34 | 4.46 | 11.67 | 0.6651 | 3.4045 | 1.4637 | 595 | 0.2419 | 3.3621 |
| ADC-19 | 16.46 | 6.41 | 13.11 | 8.21 | 1.73 | 2.00 | 4.75 | 0.7672 | 1.3416 | 0.4033 | NT | 0.1447 | 3.1452 |
| ADC-20 | 28.20 | 6.31 | 84.01 | 33.72 | 4.66 | 7.49 | 7.24 | 0.9982 | 3.1805 | 0.0082 | 580 | 0.2827 | 1.5116 |
| ADC-23 | 43.06 | 6.14 | 71.24 | 37.75 | 4.27 | 4.16 | 8.84 | 0.9902 | 3.5542 | 0.0414 | NT | 0.2704 | 3.3621 |
| ADC-24 | 41.97 | 6.42 | 64.97 | 46.35 | 4.88 | 3.89 | 9.50 | 0.9902 | 4.3563 | 0.0474 | 580 | 0.2772 | 2.2414 |
| ADC-25 | 29.82 | 8.35 | 26.09 | 28.29 | 1.95 | 2.20 | 14.51 | 0.9256 | 1.6197 | 0.1450 | 580 | 0.1421 | 3.3621 |
| ADC-26 | 50.38 | 7.54 | 45.91 | 28.13 | 4.59 | 2.29 | 6.13 | 0.9643 | 3.6846 | 0.1641 | NT | 0.2527 | 2.8261 |
| ADC-27 | 22.31 | 7.57 | 23.79 | 22.19 | 1.97 | 2.68 | 11.26 | 0.9822 | 1.7798 | 0.0351 | NT | 0.1563 | 2.7083 |

single domain (SD) and/or pseudo single Domain (PSD) state. Application of several magnetic parameters on archaeological artifacts and their implications for sample selection in archaeointensity measurements were elaborately carried out by many authors [10–13].

For the present study 19 pottery samples from Adhichanallur were subjected to a range of rock magnetic measurements. Summary of mineral magnetic parameters measured for Adhichanallur pottery samples are given in (Table 2). It is observed that the $\chi_{LF}$ values more evenly spread over a range of 13.19 to 75.06 $\times$ $10^{-7}$ $m^3/kg$ pointing to higher magnetic enhancement. Archaeological pottery generally show high susceptibility due to high temperature achieved during the baking procedure since $\chi$ is depends mainly on the concentration of ferrimagnetic minerals [14–16]. Our measurement suggests that the pottery samples are magnetically most enhanced in terms of concentration and degree of crystallinity of magnetite. High temperature behavior of magnetic susceptibility is carried out for all the pottery samples in order to determine the main magnetic minerals responsible for magnetic enhancement. Similar reversible curves are obtained for all the samples with a magnetite curie temperature (580°C).

All the pottery samples show Q-ratio in the range from 1 to 8 and have a high NRM values. This can be seen in terms of their ability to retain a very large remanent component that dominates the magnetic response and indicating a PSD magnetite contribution.

S-ratio (−300 mT) is very useful to determine the concentration of magnetic minerals of magnetite/hematite in the samples. Blomendal et al. [17] reported the *S*-ratio value of 1 for pure magnetite and it decreases with increasing proportion of antiferromagnetic particles such as hematite. From the above suggestion it is evident that all the samples studied presently shows the presence of both magnetite and hematite in varying concentration. The calculated Soft IRM and Hard IRM for all the samples indicative of the presence of high concentration of ferrimagnetic grains.(Basavaiah and Khadkikar [18]). Stepwise SIRM acquisition curves are useful in identifying magnetic mineral species [19]. Sample ADC-1 is saturated by 2 T and showed remanence coercivity ($H_{cr}$) of about 45 mT, indicating that hematite is probably the main magnetic carrier. ADC-7 and 26 are almost saturated at 700 mT and the $H_{cr}$ values are around 35–40 mT indicating a mixture of magnetite and antiferromagnetic minerals in the samples.

Hysteresis properties are helpful in identifying the magnetic mineralogy and their grain sizes [20]. The samples analyzed in the present study indicate that it comprise predominance of the PSD size, probably indicating a mixture of MD and SD grains. Narrow loops obtained for all the samples indicating only the presence ferrimagnetic minerals.

## 5 Conclusions

From the FT-IR studies it is established that all the Adhichanallur pottery samples were made up of local clay contain disordered kaolinite and fired above 600°C under varying atmospheric conditions (open/closed).

Major minerals like quartz, anorthite, albite and orthoclase, the minor quantity of iron oxides were identified from the XRD spectra.

The oxidation states of magnetic minerals present in fired archaeological pottery were well established from their Mössbauer spectra and the firing temperature and firing conditions adopted by the artisans are dealt with.

The detailed mineral magnetic studies of all the archeological artifacts reflect the magnetic mineralogy and their grain size. Consistent results were obtained from all the methods. Ferrimagnetic components (magnetite) in SD/PSD state are predominantly found in all the samples, and are found to be suitable for archaeointensity measurements.

**Acknowledgements** We thank Dr. T. Sathyamurthy, Archaeological survey of India, Chennai circle for providing the samples. Our thanks are due to The Director, Indian Institute of Geomagnetism (I.I.G), Mumbai for rock magnetic measurements.

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