LUMINESCENCE AND ELECTRON PARAMAGNETIC RESONANCE PROPERTIES OF PREHISTORIC CERAMICS FROM AL-KHIDAY EXCAVATION SITE, SUDAN

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ABSTRACT

Direct dating of prehistoric pottery from desert environment is complex, due to the lack of the attributes required for the archaeological chronological methods. Pottery samples from Sudan were therefore studied by means of Thermoluminescence (TL) and Optically Stimulated Luminescence (OSL) to establish the best measurement protocol for dating such ceramics. These techniques exploit the time-dependent accumulation of charge carriers in defects present in mineral components of ceramics, like quartz and feldspars. To evaluate the amount of such charges, which is a measure of the energy released in matter by natural ionizing radiation, two different protocols were firstly tested: the Multiple Aliquot Additive Dose protocol (MAAD) for TL and the Single Aliquot Regenerative dose protocol (SAR) for TL and OSL. Both methods allowed the discrimination of Neolithic from Mesolithic pottery, even if the data were generally affected by high uncertainties, and the presence of a few aberrant results was observed.

Moreover, a further independent technique for measuring the population of defects was attempted: the Electron Paramagnetic Resonance (EPR), in both Continuous Wave (CW-EPR) and Pulsed methods (EDEPR). This application was problematic in naturally irradiated samples, due to the low EPR sensitivity and the high Fe (III) background spectrum observed under Continuous Wave measurements. Preliminary encouraging results were instead obtained with the Pulsed technique, which was only tested before on few artificial irradiated samples.

INTRODUCTION

The El Salha archaeological project promoted by the IsIAO (Istituto Italiano per l'Africa e l'Oriente) and directed by D. Usai and S. Salvatori since 2000 is focused on the study of the prehistory of Sudan. This study began in the mid twentieth century with the excavations of J. Arkell (Arkell, 1947). Since then, many activities have been performed but there are still some gaps in the prehistoric reconstruction of the area.

The specific scientific topic of the Italian project is primarily the transition from Mesolithic hunter-gatherer-fisher to Neolithic pastoral societies, a key stage in human history of Sudan. The main feature of the Mesolithic of the area is pottery technology, usually typical of Neolithic societies.

The antiquity of pottery production along the Nile valley and in the Sahara region has been a recurring theme of research in the last years, but it is not fully understood yet. The Italian mission made headways in studying Khartoum prehistory, especially thanks to the localization and the excavation of the site called Al Khiday 1, at about 25 km south of Omdurman (Fig. 1). A test trench (5 x 5 m) revealed compact and apparently undisturbed archaeological deposits. It is very difficult to find such condition in sites along the Nile because of the strong erosion and of the action of human and animal post-depositional events typical of the sub-Saharan regions. Nile floods and wind erosion alter the matrix of the soil in powdered deposits without cohesion where archaeological findings can easily move. For this reason the original context of deposition is often lost.
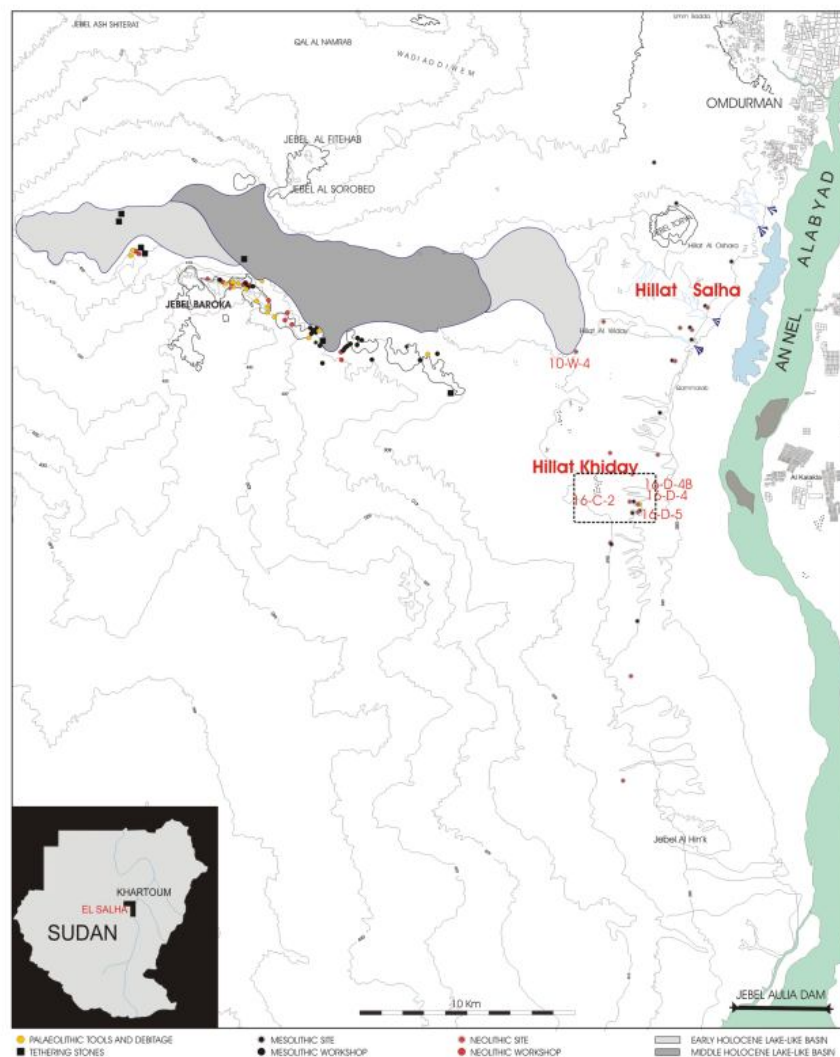

Figure 1. Prehistoric archaeological sites distribution in the El-Salha region.

The stratified deposit found at 16-D-5 is a valuable tool in order to define a first chronological reference for different types of desert pottery covering the period between approximately 7000 BC and 6000 BC. Geoarchaeological studies permitted definition of the features of the stratigraphic context, which can be divided in two macro-units (Zerboni, 2011). The upper is heavily affected by post depositional processes, while the lower (where the selected potteries were found) shows evidences of the preservation of the living floor and interpreted as an anthropogenic layer.

The unit starts at about 70 cm in depth from the surface and it is up to 80 cm thick. It is characterized by a sandy to silty-clayey matrix rich in organic matter. The micromorphological and sedimentological analysis revealed a preserved lamination with alignment of bones and shells fragments, indicating a prolonged use of the floor, trampling and absence of later bioturbation.

The abundance of amorphous organic groundmass was also related to human activity. Radiometric determinations on charcoal and shells from different features and layers fixed the chronology of the lower phase to 7th millennium cal. BC, and the upper phase to the second and third quarters of the same millennium (Salvatori et al., 2011). An extension of the excavation area reveal a 10-cm-thick deposit of packed Neolithic pottery fragments intermixed with shell and faunal remains dated to the 4th millennium cal. BC.

While the development of an absolute chronology based on prehistoric potsherds found in their original stratigraphy would help in the chronological arrangement of pottery from surface recognition and from unstratified deposits, the traditional methods of creating chronologies are difficult to apply to ceramic found in the desert, because of the lack of the attributes that are required for the common approach to dating.

In the specific case, well attested decoration motifs and almost unknown pottery types were found, and poor chronological information can be deduced from stylistic analysis (Salvatori, 2012). It is worth mentioning the results of a former absolute dating program on Neolithic pottery from Central Sudan has before performed (Guibert et al., 1994). TL ages from archaeological samples where intercompared with radiocarbon ages of associated shells. Good agreement between the two independent methods where found, allowing to confirm the chronology established on the basis of the archaeological assumption. To our knowledge, no other cases of pottery dating are available, while OSL ages were performed to support palaeoenvironment reconstruction (Williams et al., 2010).

We present here an application of Thermoluminescence (TL) and Optically Stimulated Luminescence (OSL) dating techniques on mesolithic and neolithic ceramics from al Khiday, on which also Electron Paramagnetic Resonance (EPR) dating was attempted (see Liritzis et al., 2002).

As a brief reminder, we summarize here the basis on which these dating techniques rely.

For pottery, TL and OSL dating techniques (Aitken, 1985; Aitken, 1998) allow to determine the time elapsed since the last high temperature heating experienced by a clay artefact, that usually coincides with its firing in kiln. It relies on TL or on OSL, similar physical mechanisms exhibited by quartz, feldspars and other clay components, consisting in the trapping of electron charges as a consequence of the interaction with radiation, and subsequent light emission upon heating at high temperature for TL, or illumination with light of specific wavelength for OSL.

These physical phenomena allow to measure the amount of energy absorbed (radiation dose) by matter as a consequence of the exposure to ionizing radiation, and are widely used in dosimetric applications, of which dating is just an example. Through the measurement of the amount of electron charges stored in the minerals present in a ceramic shard and by measuring the natural radioactivity field that caused this accumulation (i.e., the concentration of U, Th and K of ceramics and environment, plus a cosmic rays contribution), two main quantities can be determined: the total absorbed dose (palaeodose) and the rate at which the dose was absorbed (annual dose-rate). The age results from the equation

Age (a) = Palaeodose (Gy) / Annual Dose-rate (Gy year^{-1})

EPR is also based on the fact that ionizing radiation causes electrons to dislodge from their normal positions in atoms and get trapped in the crystalline lattice of the material, producing paramagnetic centres with long lifetimes in a number of materials.

The concentration of these centres in a given sample is therefore a measure of the total radiation dose to which the sample was exposed (Jonas, 1997).

EPR is used to date calcium carbonate in limestone, coral, fossil teeth, mollusks and egg shells as well as quartz-bearing volcanic rocks, heated sediments and stone, optically bleached quartz sediments, burn chert and flint (Rink, 1997). Its application to ceramics is not yet routinely applied (Bartoll and Ikeya, 1996).

EXPERIMENTAL

Six pottery shards of the Mesolithic period and six of the Neolithic were selected for dating (Fig. 2).

The assumed age is derived from AMS radiocarbon dating of the archaeological layers of provenance (Mesolithic 7050-6500 BC; Neolithic 4450-4230 BC; BETA Analytic Laboratory, USA; INTCAL04, OxCal 3.10; Usai et al., 2010).
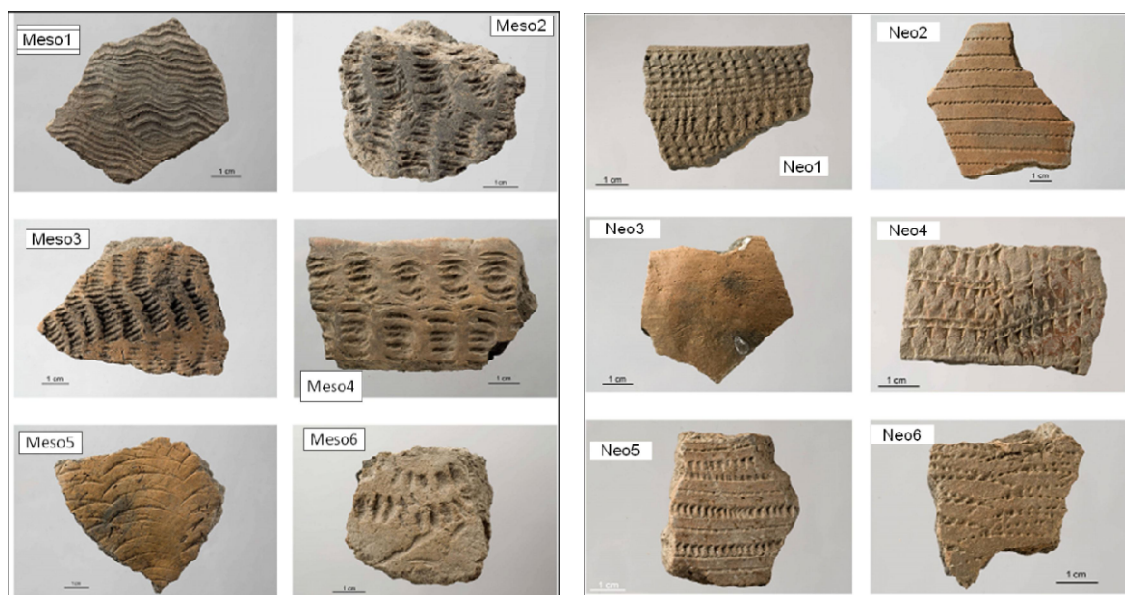

Figure 2. Fragments of pottery from Sudan. The six Mesolithic shards (Meso1-6) are reported on the left while the six Neolithic ones (Neo1-6) on the right.

The scarce amount of available material prevented any selection of luminescent minerals. The fine-grain dating technique (Zimmermann, 1971), requiring relative small amount of material, was therefore used for dating. The samples were prepared under dim red light, using the standard procedure and the polymineral fine grain (4-11 μm) fraction was deposited on stainless steel discs.

For the evaluation of the palaeodose, the TL Multiple Aliquot Additive Dose protocol (MAAD, Aitken, 1985, Liritzis et al., 2013) was first applied. In this case many aliquots (from twelve to sixteen) of the same sample are used for the construction of the luminescence vs. dose growth curve. Four aliquots are measured to obtain the natural signal, others are given different artificial doses superimposed to the natural signal. Plotting the luminescence signal versus the imparted dose, the palaeodose is obtained by extrapolation of the linear portion of the curve. The Single Aliquot Regenerative dose protocol (SAR; Murray and Roberts, 1998) was also applied. After the natural luminescence signal of the sample is registered, the very same aliquot is irradiated in laboratory with increasing doses.

To overcome the problem of sensitivity changes due to the repetition of irradiation and heating, a normalizing test dose measurement is performed after each measure. The archaeological dose is obtained by interpolation of the natu-

ral signal, corrected for sensitivity changes. The SAR protocol was here applied for both TL and OSL. It is worth mentioning that SAR is only seldom applied to ceramics (Hong et al., 2001, Takano et al., 2003, Lamothe, 2004, Benea et al., 2007).

TL measurements were performed using a home-made system based on the photon counting technique with a photomultiplier tube (EMI 9235QB) coupled to a blue filter (Corning BG12). The samples were heated from RT to 480°C at 15°C s^{-1}. Artificial irradiations were carried out by a 1.85 GBq ^{90}Sr-^{90}Y beta source (dose-rate: 4.21 Gy min^{-1}), a 37 MBq ^{241}Am alpha source (dose-rate: 14.8 Gy min^{-1}).

OSL measurements were carried out using a Risø TL-DA-20 equipped with a $^{90}Sr/^{90}Y$ beta source delivering 0.23 Gy s^{-1} to the sample position. The OSL was stimulated by an array of blue LEDs (470 ± 30 nm) for 100 s at 125°C with a constant stimulation power of 54 mW cm^{-2}. Photons were detected by a bialkali photomultiplier tube (EMI 9235QB) coupled to a 7.5 mm Hoya U-340 filter.

EPR is based, like the luminescence techniques, on the time-dependent accumulation of trapped charges due to the exposure to ionizing radiation (Ikeya, 1993). It is able to detect the absorption of microwaves by unpaired electrons (as trapped charges) under the effect of an external magnetic field.

EPR is a non-interfering method dealing with spin transitions between states of different energies without the recombination of the electrons with the holes. EPR dosimetry of archaeological samples is quite a challenge, due to the low amount of radiation induced paramagnetic defects per unit of absorbed dose.

Furthermore, Continuous Wave (CW) EPR spectra of pottery show a strong Fe(III) background, often preventing the detection of any other signal.

To circumvent this problem, the Echo Detected EPR (EDEPR) method (Schweiger et Jeshke, 2001), a common Pulsed EPR method based on the Electron Spin Echo (ESE) signal, was applied. In the EDEPR method, the ESE intensity is acquired sweeping the magnetic field and a spectrum CW-EPR-like is obtained.

The ESE of the radiation induced signal decays with very long relaxation times while the ESE of iron is characterized by so short relaxation times that cannot be detected (Zoleo et al., 2011): in that way, the EDEPR spectrum shows just the signal from the radiation-induced defects. The sensitivity of the pulse spectrometer is lower than CW one, thus increasing the limit of the detectable dose.

EPR measurements were performed at 80 K with MAAD protocol on coarse-grained samples (75-250 μm) without mineral separation. CW and pulsed measurements were made with Elexsys Bruker spectrometer equipped with a dielectric resonator and Oxford CF 935 flow cryostat.

The annual dose rate, the denominator of the equation of the age, was indirectly derived from the measurement of the radioactivity of the sample and of its surrounding soil. The U and Th concentrations were obtained by total alpha counting using ZnS scintillator discs assuming a Th/U concentration ratio equal to 3.16 (Aitken, 1985).

The contribution due to ^{40}K content was deduced from the total concentration of K obtained by flame photometry.

In the light of the available information on the humidity of the site (extremely arid nowadays, but until about 4000 years ago characterized by humid conditions and frequent floods), the pottery were assumed to have been in saturation for 50% of the time, after measuring in laboratory the maximum water content of each shard (saturation water).

RESULTS AND DISCUSSION

The typical TL behaviour of the samples is represented in Fig. 3, where several TL curves (Fig. 3a) and the corresponding TL growth curve vs. dose (Fig. 3b) are shown for MAAD protocol. The artificially irradiated samples (doses ranging from 4 to 64 Gy) were preheated at 200°C for 10 s.

In all samples, both natural and laboratory irradiated aliquots showed a broad peak centered around 350°C.

On the basis of the plateau test, that allows to identify the thermally stable portion of the curves (Aitken, 1985), the TL signals were integrated between 350 and 400°C.
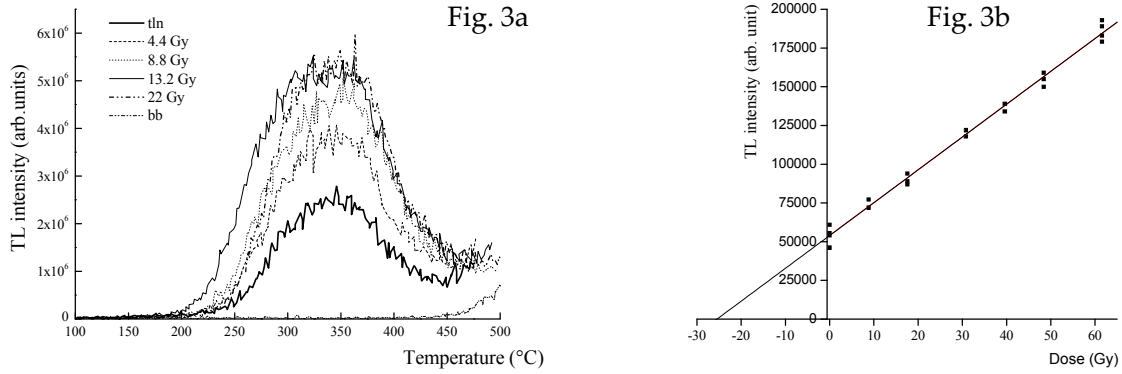
Figure 3. a) TL glow curves for the Neolithic sample Neo6 obtained with the MAAD protocol. b) TL growth curve with dose obtained from TL measurements reported in Fig. 3a.

The equivalent doses calculated with the SAR protocol were instead obtained from 3-7 aliquots for each pottery shard. The aliquots irradiated in laboratory (doses ranging from 4 to 70 Gy) were preheated at 200°C for 10 s, as for the MAAD technique. The samples did not show any change in sensitivity and TL emissions were therefore not corrected for the test dose. In Fig. 4 an example of a SAR TL growth as a function of dose is shown (Liritzis et al., 2002).
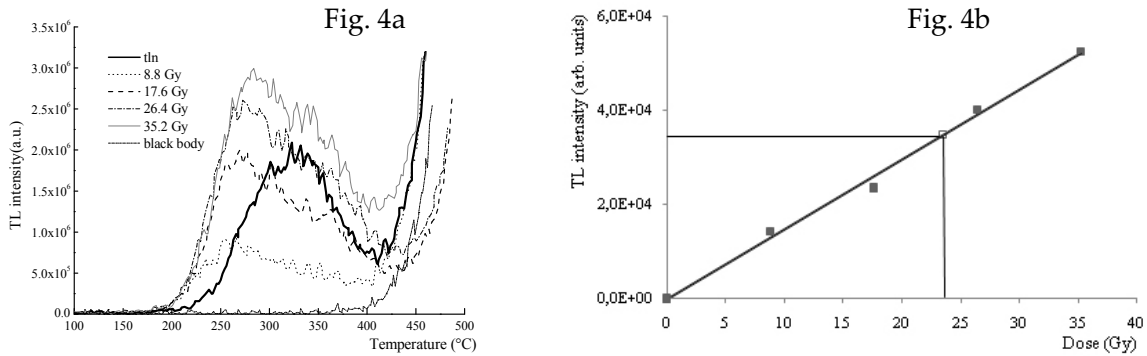

Figure 4. a) TL glow curves for the Neolithic sample Neo6 obtained with the SAR protocol. b) TL growth curve with dose obtained from TL measurements reported in Fig. 4a. Regenerated points are reported with filled squared while the natural signal with empty square.

Because in the SAR protocol the same aliquot of sample is repeatedly heated at high temperature (in our case at 480°C), a study of the dependence of luminescence characteristics on thermal treatments was made. To do this a comparison of the shape of the natural signal with that of the regenerated one was done.

No changes were detected and as a consequence the ratio of the natural TL over the regenerated TL as a function of temperature show a plateau in the temperature range 270 and 480°C (Roque et al., 2004a, 2004b; Fig. 5).
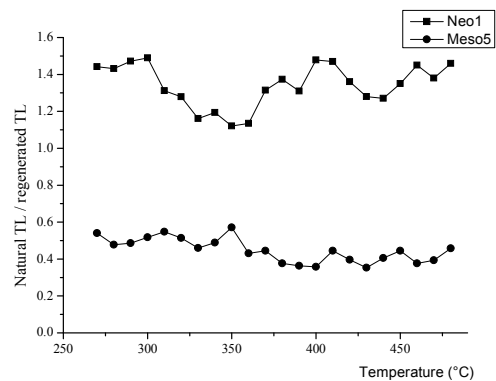

Figure 5. Ratio of natural TL to regenerated TL as a function of temperature.

The SAR protocol was also adopted for OSL measurements (Murray and Wintle, 2000). The samples were artificially irradiated with doses comprised between 11 and 40 Gy, while the test dose was 4.6 Gy.

The preheat temperature was 200°C for 10 s while the cut-heat was 180°C. For the palaeo-dose determination, the initial part of the OSL decay curve was used, specifically the first 1.6 s. The background was assumed as the average signal of the last 20 s of stimulation. Examples of shine-down curves and related growth curve are shown in Fig. 6.

The thermal transfer was systematically evaluated by calculating the recuperation point, which never exceeded 5% of the natural emission. All aliquots had acceptable recycling values, i.e. within the range of 0.90 and 1,10 (Armitage et al., 2000; Roberts and Wintle, 2001).
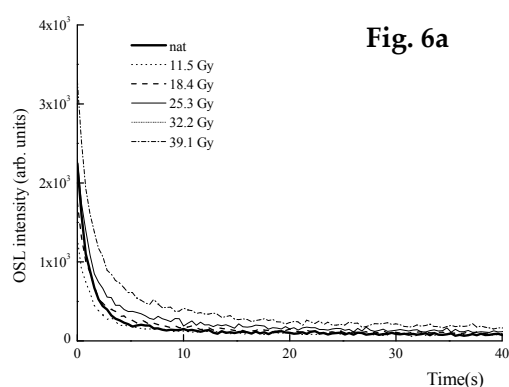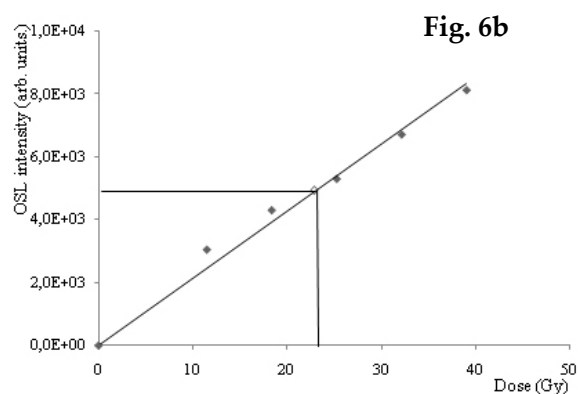

Figure 6. a) OSL shine-down curves for the Neolithic sample Neo6 obtained with the SAR protocol.
b) OSL growth curve with dose obtained from OSL measurements reported in Fig. 6a.
Regenerated points are reported with filled squared while the natural signal with empty square.

Such measurements were also repeated with alpha irradiation in order to obtain the a value, which takes into account the different efficiency of alpha particles in inducing TL (Aitken, 1985).

The fading rate (rate of luminescence loss in absence of source of stimulation) was measured for all samples (Huntley and Lamothe, 2001).

The effect of fading was almost negligible for all samples using all techniques, except for the OSL emission of the sample Meso1.

This sample is the only one with a significant content of K-feldspars, between 30 and 50%. This value was determined on thin section with a transmitted light microscope. Its g-value is 5.3%.

The mean values of palaeodose obtained with the different techniques and protocols are reported in Table 1, together with the errors. The results obtained with MAAD technique had, as expected, the highest errors, the extrapolation procedure being intrinsically less precise than interpolation.

For only 7 samples over 12 the palaeodoses measured with the different protocols were in agreement within 1 σ. In the remaining cases, high discrepancies were obtained, with no systematic trend.

Annual dose-rates are also listed in Table 1, with some detailed results of radioactivity measurements. The gamma dose rate was evaluated using the K, U and Th contents of soil (reported in the same table) that surround the pottery shards.

Repeated measurements on different fractions of the burial sediment reveal the homogeneity of distribution of radioactivity content. The cosmic component was evaluated following Prescott and Hutton (1988).

Table 1. List of samples, radioactivity data, calculated annual dose and estimated palaeodose (obtained with MAAD TL, SAR TL and SAR OSL). Sample Meso1 and Meso4 were not measurable with the MAAD protocols while for samples Meso6, Neo2 and Neo4 there were not enough samples to apply all measurements protocols.

Sample ID	H_2O sat % (± 10%)	Radioactivity			Annual dose (mGy a^{-1})	Palaeodose (Gy, mean ± 1σ)		
		% K_2O (± 3%)	ppm U (± 5%)	ppm Th (± 5%)		TL MAAD	TL SAR	OSL SAR
Meso1	7.8	4.00	3.72	11.77	8.9 ± 0.4	not measurable	52.6 ± 3.5	29.9 ± 0.7
Meso2	11.1	1.24	0.92	2.91	2.44 ± 0.12	16.2 ± 1.6	28.7 ± 0.9	25.1 ± 0.4
Meso3	12.0	0.83	1.33	4.20	2.77 ± 0.13	25.0 ± 2.3	27.1 ± 0.8	21.4 ± 0.4
Meso4	10.0	1.52	1.61	5.08	2.54 ± 0.12	not measurable	24.2 ± 0.6	20.8 ± 0.9
Meso5	8.5	0.29	1.17	3.68	2.86 ± 0.14	24.8 ± 2.6	26.1 ± 0.8	25.2 ± 0.5
Meso6	11.2	0.89	1.53	4.85	3.19 ± 0.15	26.0 ± 2.3	Not enough sample	23.1 ± 0.3
Neo1	6.7	0.56	1.80	5.70	2.23 ± 0.11	18.0 ± 1.6	14.8 ± 0.1	15.1 ± 0.7
Neo2	9.6	1.05	1.84	5.81	2.99 ± 0.14	18.3 ± 1.7	18.2 ± 0.3	Not enough sample
Neo3	9.7	1.20	1.01	3.20	2.82 ± 0.13	22 ± 2	22.0 ± 1.0	16.0 ± 0.5
Neo4	13.9	1.57	1.42	4.48	3.46 ± 0.17	26 ± 3	23.0 ± 0.5	Not enough sample
Neo5	9.6	1.29	1.10	3.48	2.97 ± 0.14	32.0 ± 2.1	31.4 ± 0.9	17.3 ± 0.4
Neo6	11.2	1.13	1.81	5.72	3.62 ± 0.17	22.0 ± 2.1	21.5 ± 0.6	20.0 ± 0.5
soil	20	0.47	1.50	4.75				

The dating results (years since firing in kiln) are obtained by dividing the palaeodose by the corresponding dose-rate. This was done for each technique, therefore obtaining 3 ages for each sample. They were corrected for fading and are reported in Table 2. Errors, calculated following the standard procedures (Aitken, 1985), ranged from 5 to 15 %. The unhomogeneity of palaeodoses obviously reflects into the unhomegeneity of the calculated ages.

Table 2. Ages of the samples obtained with the three luminescence methods. Radiocarbon ages of the layer of provenance are indicated.

Sample ID	US ^{14}C dating	Age (a)		
		TL MAAD	TL SAR	OSL SAR
Meso1	Mesolithic 8510-9060 cal BP		5930 ± 480	4760 ± 170
Meso2		6650 ± 730	11770 ± 690	10280 ± 530
Meso3		9030 ± 930	9780 ± 540	7730 ± 390
Meso4			9510 ± 500	8160 ± 520
Meso5		8680 ± 1000	9140 ± 530	8830 ± 460
Meso6		8160 ± 810		7240 ± 350
Neo1	Neolithic 6240-6460 cal BP	8070 ± 820	6640 ± 330	6760 ± 460
Neo2		6120 ± 630	6080 ± 300	
Neo3		7810 ± 790	7800 ± 500	5680 ± 310
Neo4		7510 ± 940	6640 ± 350	
Neo5		10760 ± 870	10560 ± 580	5830 ± 300
Neo6		6070 ± 620	5940 ± 320	5520 ± 290

In general OSL gives younger ages than SAR TL. For the two class of samples (Mesolithic and Neolithic) and for the sub-classes based on the technique used for palaeodose evaluation (TL-MAAD, TL-SAR and OSL-SAR) mean, standard deviation, weighted mean and error of the mean have been separately evaluated as reported in Table 3.

The mean attributed the samples to the correct prehistoric period, but with a high standard deviation (about 20% in most cases). The data obtained for the two groups well matched their Mesolithic and Neolithic attribution; but they are statistically indistinguishable within ± 2 σ confidence level.

However, if we consider the weighted mean, the data appear to be better grouped, and the error noticeably reduced. These results are also reported in Fig. 7 which gives a visual representation of the distribution and precision of data.

Table 3. Mean age, standard error, weighted mean age and error of the mean for all the Mesolithic and all the Neolithic samples obtained with all the techniques (MAAD TL, SAR TL and SAR OSL) compared with those calculated separately for each technique.

	#	Mean age	St. dev.	Weighted mean age	Weighted calendar age	Uncertainty (1σ)	Uncertainty (%)
Mesolithic							
MAAD	4	8130	1050	7900	5890	360	4.6
SAR TL	5	9230	1770	9150	7140	240	2.6
SAR OSL	5	8450	1180	8050	6040	200	2.5
Neolithic							
MAAD	5	7710	1720	7350	5340	620	8.2
SAR TL	5	7280	1740	6550	4540	160	2.4
SAR OSL	4	5950	560	5750	3740	160	2.8

As a conclusion, it appeared that luminescence techniques correctly dated our set of prehistoric samples. Conventional fine-grain technique, with additive protocol for palaeodose evaluation, gave the less precise and more scattered results, while the Single Aliquot approach was systematically more precise and accurate.

In particular for Mesolithic ceramics the ages obtained with SAR TL and SAR OSL are in agreement with the radiocarbon ages, while for the Neolithic ones the most similar was SAR TL. Quite surprisingly, the better results were obtained using the blue stimulated OSL emission of polymineral fine-grain samples, without any mineralogical selection. The richness in quartz of the ceramic pastes is the most probable reason of these results.

In order to verify the capability of Electron Paramagnetic Resonance as dating techniques for pottery, the CW-EPR spectra were recorded for all the samples. A typical CW-EPR spectrum of pottery is shown in Fig. 8a where only a large

line due to Fe (III) at g~2 can be recognized. This signal is not dose dependent and thus not useful for dating purpose. The Pulsed EPR method was then applied to verify the presence of species with long relaxation times as radiation induced defects. Since transition metal ions have short relaxation times, they cannot be recorded.
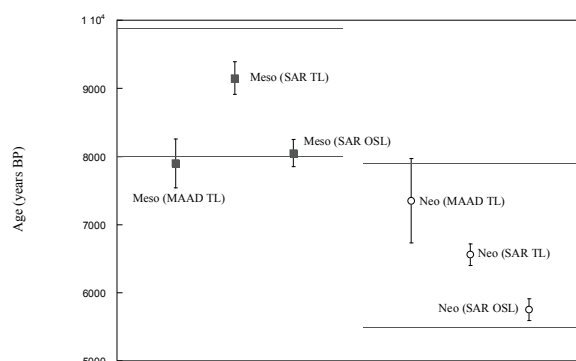

Figure 7. Weighted mean of the ages and error of the mean for all the Mesolithic (15) and all the Neolithic (16) data obtained with all the techniques (MAAD TL, SAR TL and SAR OSL) compared with those calculated separately for each technique.
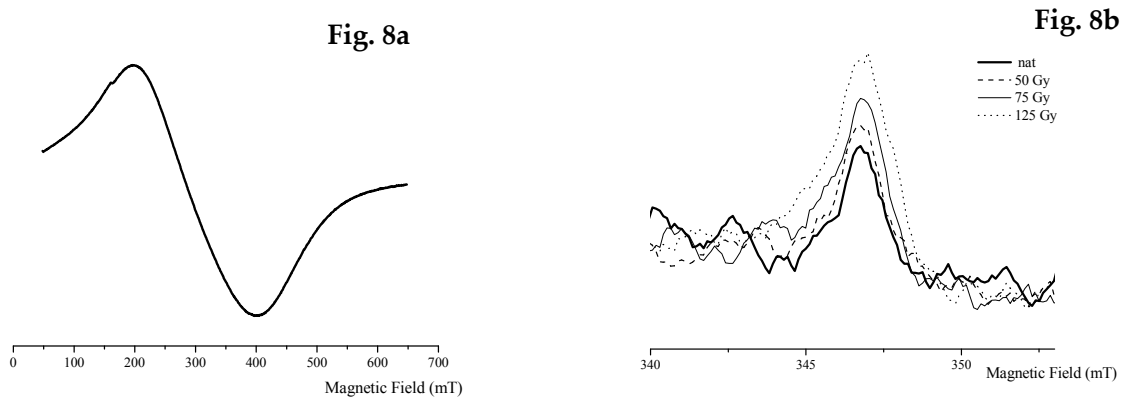
Fig. 8a

Fig. 8b

Figure 8. a) CW-EPR spectrum obtained for sample Neo3. b) EDEPR spectra for the same sample. There are shown natural and irradiated signals.

For six samples, in such pulsed conditions a dose dependent signal was recorded, the natural EPR emission being well distinguishable from background (Fig. 8b).

The aim of these measurements was to verify if there were signals potentially suitable for dating and to identify parameters influencing the procedures. An artificial added beta dose confirmed the growth of the observed natural signal with incremental irradiation (Fig. 8b). Irradiation was carried out by the same ^{90}Sr-^{90}Y beta source used for TL analysis and measurements were performed following the MAAD protocol without preheating the aliquots.

A chemical etching was performed to verify if such signal could be generated by the crushing during the sample preparation procedure and a thermal annealing test at 450°C was conducted in order to verify the zeroing of the EPR signal by heating and to exclude the presence of a paramagnetic background. The signal seemed not to be induced by crushing and not affected by residual background after the heating treatment. The signal is not directly referable to a specific paramagnetic center but it is supposed that it could be generated by the superimposition of different radiation induced defects. For each aliquot the intensity of the signal was calculated as the integral under the spectrum after baseline correction. The intensity increases linearly with the dose as shown in the growth of Pulsed EPR signal vs. dose reported in Fig. 9.

The growth of Pulsed EPR signal vs. dose is reported in Fig. 9. Even if the signals are dose dependent, an overestimation of the absorbed dose was observed. Further investigations are in progress to find the causes of the overestimation, taking into account the irradiation sources and the mineralogical-petrographic compositions of the potsherds. Moreover the characterization of the defects responsible for the signal will be necessary.
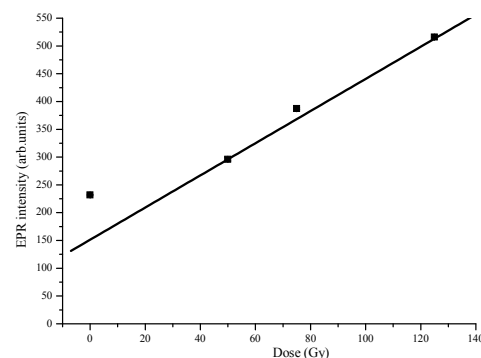

Figure 9. EDEPR growth curve with dose obtained from sample Neo3.

CONCLUSION

Luminescence techniques allowed to correctly date two groups of Sudanese ceramics, sampled from a Mesolithic - Neolithic sequence of archaeological layers. The samples were prepared following the fine-grain technique, without mineralogical separation, just selecting the 4-11 μm polymineral fraction.

The main advantage of this technique is the low amount of material needed for sample

preparation, which is often the main constraint when dealing with prehistoric samples.

Poor precision and accuracy characterized the ages measured with additive procedure, which is definitely not recommended for such prehistoric samples. Much better results were instead obtained with SAR protocol using both TL and blue-stimulated OSL emission.

The preliminary results of EPR dating are encouraging, but the identification of a natural radiation induced signal is not an easy task. Pulsed EPR appeared to be a valid tool to spot radiation induced defects in pottery, even without any chemical treatment of the sample and with the possibility of performing several measurements on the same aliquot of the material.

Moreover, in order to obtain more reliable and consistent results, EPR measurements on quartz extracted from ceramics should be performed.

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