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ELECTRON SPIN RESONANCE DATING OF FOSSIL TOOTH ENAMEL.

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Abstract: In this work the ESR method has been applied to determine the age of tooth enamel found in Mingachevir archeological site. ESR signal intensity of paramagnetic center identified in fossil tooth shows linear dependency with the adsorbed dose. The mean age of the sample was determined as 275.82 ± $10.39x10^3$ years.

Key words: ESR dating, tooth enamel, ROSY software, archaeology

1. Introduction

Electron spin resonance (ESR) analysis is one of the alternative methods on dating of ancient artifacts and enables to extend the time limit beyond the conventional radiocarbon dating. Due to long mean life of some paramagnetic centers (approximately $1x10^8$ years), it makes it possible to identify them in materials as old as $1x10^6$ years which can be applied to a variety of problems in geology, archaeology and paleoanthropology [1]. At the same time ESR method can be used as an alternative method on verification and/or crosschecking the results obtained by radiocarbon dating method. ESR dating is based on the fact that natural ionizing irradiation produces paramagnetic centers in some materials, particularly in tooth enamel with the long mean life. Those centers are stable at the temperatures below 100ºC and might be considered as a measure of the total irradiation dose to which a particular sample has been exposed. This effect has been used with tooth enamel for determining the age of archeological sites [2,3]. In this work the same method has been applied to determine the age of tooth enamel found in Mingachevir archeological site using ESR method.

1. Materials and methods

The investigated object was fossil tooth of elephant (*Palaeoloxodon antiquus*[1]) found in Mingachevir district of Azerbaijan in 2010 with well-preserved teeth. Sample preparation and ESR measurement procedures followed standard techniques [5].The enamel was initially removed from teeth using a dental drill and water cooling. The 2 mm mean thickness enamel was then placed in a 30% NaOH solution for one day to disinfect it and separate any remaining dentine.

[1] The **straight-tusked elephant** (*Palaeoloxodon antiquus*) is an extinct species of elephant that inhabited Europe during the Middle and Late Pleistocene (781,000–50,000 years before present). It was formerly thought to be closely related to the living Asian elephant; however, in 2016, DNA sequence analysis showed that its closest extant relative is actually the African forest elephant, *Loxodonta cyclotis*. It is closer to *L. cyclotis* than *L. cyclotis* is to the African bush elephant, *L. africana*, thus invalidating the genus *Loxodonta* as currently recognized. (*Callaway, E. (2016-09-16). "Elephant history rewritten by ancient genomes". Nature.* doi:10.1038/nature.2016.20622.)

A dental drill was used to strip around 50±5 μm from inside and outside of the enamel surface to ensure that alpha radiation had no effect. In total 1.2 gr. enamel was collected and it was air-dried at room temperature for three days. Enamel powder was divided into six equal parts and each aliquot was placed inside glass tubes (Suprasil) for irradiation. The samples were irradiated using ^{60}Co source. Dose rate of the ^{60}Co source has been determined by MagnettechMiniscopeMS400 EPR Spectrometer using individually wrapped, bar code labeled BioMax Alanine Dosimeter Films (developed by Eastman Kodak Company). ESR signal for the sample found at Chukhur-Gabala site of the Gabala district Azerbaijan was measured with a Bruker EMXplus(X-band) spectrometer. The spectrometer parameters used were: 3491 G central field, 100 G scan range, 3,2 G amplitude modulation, 100kHz modulation frequency, 163.840ms time constant and 2.232 mW microwave power. The samples were then irradiated with additional doses and ESR signals were measured at the same conditions.

In order to estimate the natural dose rate soil samples were collected from the site and U, Th, and K content analysis by gamma spectrometry Canberra GR4520 which has a low-level gamma spectrometry system with 15 cm lead shielding and high-resolution GeHP hyper pure germanium detector, having 43.5% resolution efficiency for 661.6 keV.

2. Results and Discussions

Figure 1 shows the ESR spectra for the enamel sample from the tooth found Mingachevir district of Azerbaijan. Six equal aliquots of enamel sample have been irradiated at the ^{60}Co source with the dose ranging from 44.7 to 223.5 Gy. The intensity of the ESR signal measured according to conventional peak to peak height method which shows a linear dependency (Fig.2) from the absorbed dose. We also established that the dose rate does not influence the intensity of the ESR signal. The archaeological dose (D_a) obtained by the extrapolation back to zero ordinate was 515±45 Gy. (Fig.2)

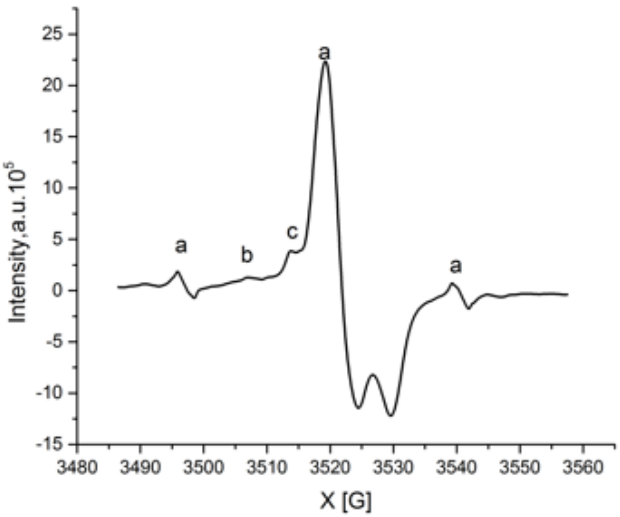

Fig. 1. EPR spectra of elephant fossil tooth enamel powder. Punctuation and attribution of EPR signals have been borrowed from [6]: (i) Signal labeled "a"- a septet centered on the main CO^-_2 signal at g=2.0032 (only three lines are visible on that magnetic field range) formed by a free dimethyl radical; (ii) isotropic line (marked "b") at g=2.0115 might be attributed to CO_3^-; and the isotropic line at g= 2.0056 (marked "c") is usually attributed to a free radical, probably SO_2^-

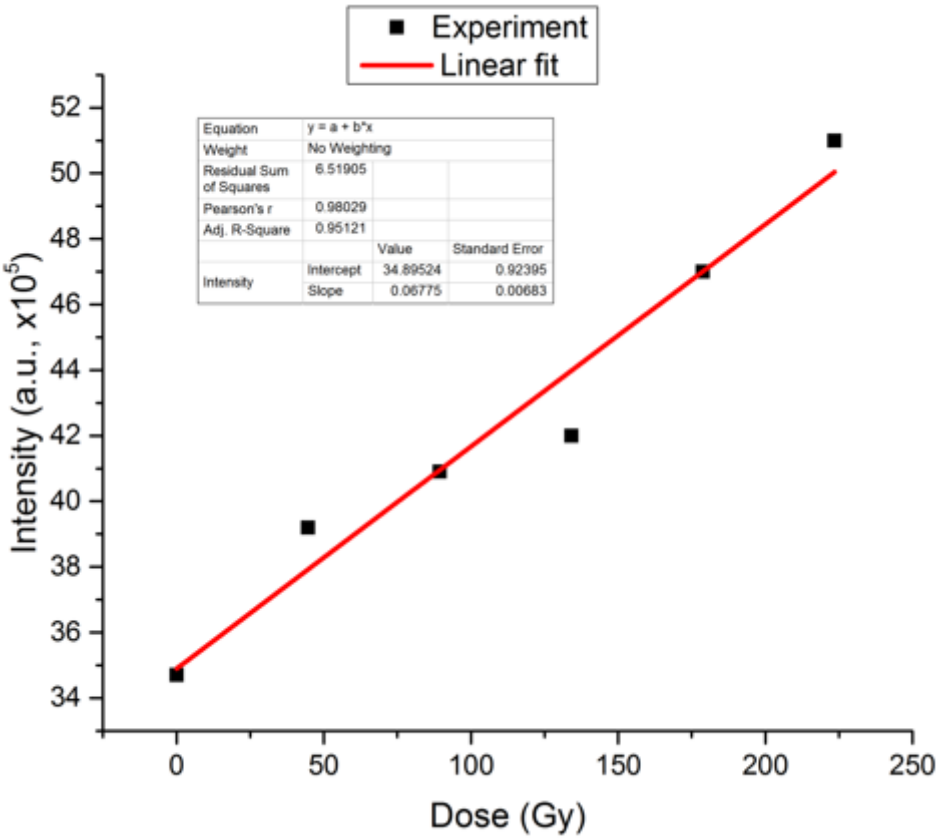

Fig. 2. Dose-response curve of fossil tooth enamel EPR spectra intensity. The intensity of EPR signal calculated conventional peak to peak height method. The curves were fitted with OriginPro 2018b.

ROSY software [4] was used for calculating the enamel sample's age A by comparing accumulated archaeological dose (D_a) to the average dose rate (D) over such period:

$$A = \frac{Da}{D}$$

Calculation of D was based on estimated cosmic dose rate as well as U, Th and K content obtained from soil sample analysis which were 3,51 ppm for U, 6.71 for Th and 1.99% for K. Uranium content obtained for enamel was 0.71 ppm. The cosmic dose rate determined to be 0.293×10^{-3}Gy/year. The average moisture content of the sediment was taken as 25% on the bases of measurement at the site. Total estimated annual dose rate was equal to 2.93×10^{-3}Gy/year.

3. Conclusions

ESR signal intensity of paramagnetic center identified in fossil tooth shows linear dependency with the adsorbed dose. The intensity of this peak increases with the amount of γ-irradiation absorbed by the tooth enamel. Additive dose method makes it possible to determine the accumulated archaeological dose and enables the estimation of the fossil tooth age.

The mean age of the sample found at Mingachevir district Azerbaijan was determined as $275.82 \pm 10.39 \times 10^3$ years.

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ARXEOLOJİ QAZINTI ZAMANI TAPILMIŞ DİŞ EMALININ EPR METODU İLƏ TARİXLƏNDİRİLMƏSİ

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Xülasə: Arxeoloj qazıntı zamanı Mingəçevir ərazisindən tapılmış fil dişinin yaşı EPR metodu ilə təyin edilmişdir. Diş emalında müşahidə olunan EPR siqnalın intensivliyi udulmuş dozadan asılı olaraq xətti dəyişir. Bu xassədən istifadə edərək tapılmış nümunənin yaşı 275.82 ± 10.39x10^3olmuşdur

Açar sözlər: EPR yaş təyini, diş emalı, arxeologiya

ОПРЕДЕЛИЯ ВОЗРАТА ЗУБНОЙ ЕМАЛИ МЕТОДОМ ЭПР

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Резюме: Методом ЭПР определен возраст зубной эмали найденной во время археологических раскопок в Мингечауре. Интенсивность ЭПР сигнала линейно возрастает с увеличением поглощенной дозы. Используя этого свойства, нами определен возраст этого зуба раной к 275.82 ± 10.39x10^3 годам.

Ключевые слова: ЭПР датирование, зубная эмаль, археология