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ESR AND THERMOLUMINESCENCE (TL) DATING OF ARCHEOLOGICAL MONUMENTS IN AZERBAIJAN

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Abstract: This study sought to enhance the precision of ESR and TL dating of archaeological monuments in Azerbaijan. Teeth and pottery samples were collected from the following archaeological sites: Jalilabad-Polutepe, Fuzuli-Leletepe, Ismayilbeytepe, Agstafa-Hesensu, Berde-Emirli-3, Jalilabad-Pashatepe, Gobustan-Anazaga, Gobustan-Boyukdash, Gobustan-Kichikdash, Jalilabad-Khudutepe, Chukur-Gebele, Mingechevir, Jalilabad-Elikomektepe, Gebele-Galayeri, and Yardimli-Arvana. The ESR and TL methods were employed to ascertain the ages of the samples obtained from the aforementioned sites.

Keywords: dating, ESR, TL, Azerbaijan, archeology.

1. Introduction

Thermoluminescence (TL) dating and Electron Spin Resonance (ESR) dating are both methods used to date minerals and artifacts based on their exposure to radiation. While they share some similarities, they operate on different principles and are suited to different types of materials and dating ranges. Here's a comparison of both methods. TL dating is useful for artifacts and sites where materials have been subjected to heat or sunlight, while ESR dating excels in dating fossil teeth and marine deposits over much longer time spans. Both methods (Table 1) contribute significantly to archaeology, geology, and our understanding of human evolution [1]. Table 1 shows the comparison of TL and ESR dating methods.

Table 1

Comparison of TL and ESR Dating

Aspect	Thermoluminescence (TL) Dating	Electron Spin Resonance (ESR) Dating
Principle	Measures light emitted during heating to release trapped electrons	Measures trapped electrons based on their magnetic resonance
Materials	Ceramics, heated rocks, sediments	Tooth enamel, shells, quartz, corals
Age Range	Up to ~500,000 years	100,000 to 5 million years
Clock Reset	Last heating or exposure to sunlight	Initial formation or last exposure to sunlight
Accuracy	Can be less precise than ESR for older samples	Suitable for dating much older samples, including fossils
Applications	Archaeology, ancient pottery, and sediments	Paleoanthropology, dating human fossils and ancient teeth

The objective of this study was to enhance the precision of ESR and TL dating of archaeological monuments in Azerbaijan [2–10]. The artifacts were gathered from a range of archaeological locations, and their chronological ages were established. This process provides valuable insights into the sequence of events at different locations, contributing to a broader understanding of historical or prehistoric activities across various sites.

2. Materials and Methods

An alternative approach to dating ancient artifacts is electron spin resonance (ESR) analysis, which permits the imposition of a longer time limit than is possible with traditional radiocarbon dating. The ability to identify paramagnetic centers in materials as ancient as 1×10^6 years, due to their extended mean lifespan (approximately 1×10^8 years), offers significant potential for addressing a number of issues in geology, archaeology, and paleoanthropology [1]. Furthermore, the ESR method can be employed as a supplementary technique for cross-referencing and/or verifying the results of the radiocarbon dating method. The foundation of ESR dating lies in the understanding that certain materials, particularly dental enamel with its extended lifespan, generate paramagnetic centers when subjected to natural ionizing radiation. The sample preparation and ESR measurement procedures were conducted in accordance with established standard techniques (see Fig. 1). As illustrated in the accompanying illustration, the enamel was initially removed from the teeth using a dental drill and water cooling.

The enamel, with a mean thickness of 2–3 mm, was then placed in a 30% NaOH solution for one day to disinfect it and separate any remaining dentine [2–10]. A dental drill was utilized to strip around 55 ± 5 μm from both inside and outside of the enamel surface to ensure that alpha radiation had no effect. In total 1.2 grams of enamel was collected and air-dried at room temperature for three days.

Fig. 1. Tooth sample preparation for ESR dating.

The enamel powder was divided into six equal parts and each aliquot was placed inside glass tubes (Suprasil) for irradiation [11–16]. This area is designated for sample preparation, which is conducted under controlled lighting conditions. The physical preparation area is equipped with the requisite materials for optimal sample treatment prior to chemical preparation, including saws, dental drills, agate mortars, sieves, mills, hot plates, scales, and a magnetic separator, among other items [17–21]. The chemical preparation area is fitted with fume hoods and all other laboratory materials required for acid attacks or the separation of minerals through dense liquids.

Conversely, the Quartz inclusion method is utilized for TL dating. The Harshaw TLD 3500 Manual Reader is employed to measure the TL characteristics of the samples. The dose rate of the ^{60}Co source was determined by the Magnettech Miniscope MS400 EPR Spectrometer using individually wrapped, barcode-labeled BioMax Alanine Dosimeter Films (developed by Eastman Kodak Company) [22–26]. The irradiated samples were weighed to 5 ± 0.5 mg and read out after

one day in an N_2 atmosphere in a Harshaw 3500 manual reader, using a linear heating rate of 5 °C/s.

To ascertain the natural dose rate, soil samples were collected in close proximity to the pottery sample. Uranium, Thorium and Potassium concentrations in soil were measured using gamma spectrometry Canberra GR4520, which features a low-level gamma spectrometry [27–32] system with 15 cm lead shielding and high-resolution GeHP hyper-pure germanium detector [33–35], achieving a resolution efficiency of 43.5% for 661.6 keV. Dose rates and age calculations were conducted using the online DRAC version 1.2.

3. Results and Conclusion

The results of the dose rates and age calculations from TL and ESR measurements are illustrated (Table 2) as follow:

Table 2 shows the results of the TL and ESR dating measurements of the monuments in Azerbaijan.

Table 2

TL and ESR Ages of various monuments in Azerbaijan.

Sample name	**TL age (BP) ± error**	**Material**
Jalilabad-Polutepe	6400 ± 530	Ceramic
Fuzuli-Leletepe	7919 ± 455	Ceramic
Ismayilbeytepe	6908 ± 117	Ceramic
Agstafa-Hesensu	7913 ± 324	Ceramic
Berde-Emirli-3	8189 ± 850	Ceramic
Jalilabad-Pashatepe	7826 ± 513	Ceramic
Gobustan-Anazaga	15013 ± 1324	Fired soil
Gobustan-Boyukdash	6835 ± 383	Ceramic
Gobustan-Kichikdash	5655 ± 233	Ceramic
Jalilabad-Khudutepe	7116 ± 343	Ceramic
Chukur-Gebele	2230 ± 510	Ceramic water pipe
Sample name	**ESR age (BP) ± error**	**Material**
Jalilabad-Polutepe (sample 1)	7421 ± 130	Tooth
Jalilabad-Polutepe (sample 2)	7774 ± 129	Tooth
Jalilabad-Polutepe (sample 3)	7960 ± 146	Tooth
Agstafa-Hesensu	8432 ± 416	Tooth
Berde-Emirli-3	7937 ± 258	Tooth
Gobustan-Boyukdash	1894 ± 97	Tooth
Gobustan-Boyukdash	14402 ± 267	Mollusc shell
Gobustan-Kichikdash	2620 ± 304	Tooth
Gobustan-Kichikdash	4233 ± 364	Deer antler
Jalilabad-Khudutepe	8225 ± 123	Tooth
Chukur-Gebele	2550 ± 200	Tooth
Mingechevir	275800 ± 10390	Tooth
Jalilabad-Elikomektepe (sample 1)	5959 + 122	Tooth
Jalilabad-Elikomektepe (sample 2)	5782 + 118	Tooth
Gebele-Galayeri	5514 ± 124	Tooth
Yardimli-Komani	4775 ± 664	Tooth
Yardimli-Saribulag	5356 ± 199	Tooth

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ДАТИРОВАНИЕ АРХЕОЛОГИЧЕСКИХ ПАМЯТНИКОВ АЗЕРБАЙДЖАНА С ПОМОЩЬЮ ЭСР И ТЕРМОЛЮМИНЕСЦЕНЦИИ

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Резюме: Целью данного исследования было повышение точности датирования археологических памятников Азербайджана методами ЭСР и ТЛ. Образцы зубов и керамики были собраны со следующих археологических памятников: Джалилабад-Полутепе, Физули-Лелетепе, Исмаилбейтепе, Агстафа-Хесенсу, Берде-Эмирли-3, Джалилабад-Пашатепе, Гобустан-Аназага, Гобустан-Бёюкдаш, Гобустан-Кичикдаш, Джалилабад-Худутепе, Чукур-Гебеле, Мингечевир, Джалилабад-Эликомектепе, Гебеле-Галаери и Ярдымлы-Арвана. Методы ЭСР и ТЛ были использованы для определения возраста образцов, полученных с вышеупомянутых памятников.

Ключевые слова: датирование, ЭСР, ТЛ, Азербайджан, археология.

AZƏRBAYCAN ƏRAZİSİNDƏ YERLƏŞƏN ARXEOLOJİ ABİDƏLƏRİN EPR VƏ TL METODLARLA TARİXLƏNDİRİLMƏSİ

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Xülasə: Bu tədqiqatda Azərbaycanda arxeoloji abidələrin ESR və TL tarixləndirmə üsulları ilə mütləq yaşları tədqiq olunmuşdur. Diş və saxsı nümunələri aşağıdakı arxeoloji ərazilərdən toplanmışdır: Cəlilabad-Polutəpə, Füzuli-Lələtəpə, İsmayılbəytəpə, Ağstafa-Hesensu, Bərdə-Əmirli-3, Cəlilabad-Paşatəpə, Qobustan-Anazağa, Qobustan-Böyükdaş, Qobustan-Kiçikdaş- Xudutəpə, Çukur-Gəbələ, Mingəçevir, Cəlilabad-Elikoməktəpə, Qəbələ-Qalayeri və Yardımlı-Arvana. Yuxarıda qeyd olunan yerlərdən alınan nümunələrin yaşını təyin etmək üçün ESR və TL metodlarından istifadə edilmişdir.

Açar sözlər: tarixləndirmə, EPR, TL, Azərbaycan, arxeologiya.