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Hypergene alterations of succinite and its vulnerability under various environmental conditions

Uliana Naumenko, Victor Matsui,
Oleksandr Aleksandrov, Oleksii Naumenko

Institute of Geological Sciences, NAS of Ukraine (Kyiv, Ukraine)

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correspondence to
Uliana Naumenko; Institute of Geological Sciences of NAS of Ukraine, 55b Oles Honchar Street, Kyiv, 01054 Ukraine;
Email: uznaum@gmail.com;
orcid: 0000-0001-9420-4044

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abstract

The article describes the alteration of succinite under conditions of hypergenesis as a result of oxidation, including a change in colour, contraction of the outer surface and the appearance of microcracks, changes in the construction of the outer framework of macromolecules and chemical composition and conditions of complete destruction. The main factors influencing the resistance of succinite under hypergene conditions are described — the influence of acid potential, air, light, fluctuations in temperature and humidity, geological affiliation to certain sediments. The problem of changing the quality of succinite is determined. It is shown that its destruction is associated with disturbed bedding conditions, changes in groundwater regime, as well as sea surf activity in the littoral (coastal) zone. Succinite that was found in stratigraphic sections not uncovered by erosion in the primary bedding, mostly retains its composition, properties, and structure formed in the past stages of plant resin fossilization. Placers of succinite that were formed as a result of erosion and re-deposition of primary Eocene–Oligocene placers are different due to the dimensions of succinite grains, their degree of grain rounding and overall presence of more resistant to weathering ones. The process of amber destruction is quite lengthy. Succinite, like other minerals more resistant to weathering, undergoes various stages of change in nature. An illustrative example of succinite destruction is succinite found during archaeological excavations. It was found that succinite is practically not preserved in deposits of loess and red carbonate formations. Due to its organic origin, amber ranks last in the group of placer-forming minerals and is characterized by the lowest constant hypergene resistance, which is determined by its low density (1.07) and minimum hardness (2.3). The conclusions and recommendations given in the article on the transformation and preservation of succinite that are brought to the surface are based on the analysis of extensive scientific literature, as well as many years of research on this gem in the Polissia and Dnieper region. Recommendations are given for long-term preservation of succinite under surface conditions, as well as museum samples, amber products in everyday life and during transportation.

Гіпергенні зміни сукциніту та його вразливість в різних природних умовах

Уляна Науменко, Віктор Мацуй, Олександр Александров, Олексій Науменко

Резюме. В статті охарактеризовано перетворення сукциніту в умовах гіпергенезу в результаті окислення, що включає зміну окрасу, контракцію зовнішньої поверхні і появу мікротріщин, зміну побудови зовнішнього каркасу макромолекул та хімічного складу, умови повного руйнування. Описані основні фактори впливу на стійкість сукциніту в гіпергенних умовах — вплив кислотного потенціалу, повітря, світла, коливань температури і вологості, геологічної приналежності до тих чи інших відкладів. Визначено проблему зміни якості сукциніту. Показано, що його руйнування пов'язане з порушеними умовами залягання, зміною режиму ґрунтових вод, а також морської хвилеприбійної діяльності в літоральній (прибережно-морській) зоні. В стратиграфічних розрізах, не розкритих ерозією, тобто в первинному заляганні пласта, сукциніт в цілому зберігає свій склад, властивості і структуру, набуті у минулі етапи фосилізації рослинних смол. Розсипи сукциніту, сформовані у результаті розмиву та перевідкладення первинних (корінних) еоцен-олігоценових розсипів значно відрізняються, перш за все розмірністю зернового сукциніту, ступенем окатаності і в цілому наявністю більш стійких до вивітрювання екземплярів. Процес руйнації бурштину досить тривалий. Сукциніт, як і інші, більш стійкі до вивітрювання мінерали, проходить різні стадії змін в природі. Наглядним прикладом руйнації сукциніту слугує сукциніт з археологічних розкопок. Встановлено, що у відкладах лесової і червоноколірної карбонатної формації сукциніт практично не зберігається. В силу свого органічного походження бурштин займає останнє місце в групі розсипоутворюючих мінералів і характеризується найнижчою константною гіпергенною стійкістю, що визначається його низькою густиною (1,07) і мінімальною твердістю (2,3). Наведені в статті висновки та рекомендації про перетворення і збереження сукциніту, виведеного на денну поверхню, базуються на аналізі обширної наукової літератури, а також багаторічних досліджень самоцвіту в бурштиновому Поліссі і Придніпров'ї. Надано рекомендації щодо довготривалого збереження сукциніту, виведеного на денну поверхню, а також музейних зразків, виробів з бурштину в побуті та при транспортуванні.

Ключові слова: рослинні смоли, викопні смоли, сукциніт, фосилізація, гіпергенне перетворення, окислення, гіпергенна стійкість, зона аерації, археологічний сукциніт.

Адреса для зв'язку: У. Науменко; Інститут геологічних наук НАН України; вул. Олеся Гончара, 55б, Київ, 01054 Україна; e-mail: uznaum@gmail.com; orcid: 0000-0001-9420-4044

Introduction

The idea of publishing an article about the destruction of succinite was developed due to the predatory illegal extraction of succinite by heavy earthmoving equipment, hydraulic pumps, etc., which affects not only the Ukrainian Polissia, but also the gem itself, brought to the aeration zone, where it loses its hypergene resistance. The tendency of the gem to destruction is confirmed by archaeological finds of products made from it, which were extracted and processed several hundred years ago. At present, under conditions of anthropogenic air pollution, the destruction of succinite is rapidly accelerating. Its state is also adversely affected by ultraviolet radiation, which causes ionization in organic compounds.

To protect some conifers and other plants from various physical injuries and diseases in the process of evolution, a complex system of accumulation and secretion of resin has been developed. Products of post mortem changes of resin secretions (trunk, underbark, interbark) and other fillings of resin pockets, various accumulations in the soil are considered as fossil resins. The latter acquire the characteristic features of fossilized mineral species of fossil resins only in the process of long-term (millions of years) and gradual replacement of organic material by mineral ones in the process of physical and geochemical transformations.

Succinite (Fig. 1) is the mineralogical name of the predominant (more than 90 %) and the most valuable type of fossil resins (FR). It is distributed exclusively in the Baltic–Dnipro amber province, which stretches from the British Isles and southern Sweden to the shores of the Black and Azov seas.

Fig. 1. Ukrainian succinite. A specimen found in sediments on the right bank of the Dnipro River. Novi Petrivtsi. Photo by I. Naumenko.

Рис. 1. Український сукциніт. Зразок, знайдений в відкладах на правому березі річки Дніпро. Нові Петрівці. Фото І. Науменка.

At the stage of land-bog transformation of plant resins, the soft resin under the influence of various environmental conditions and the manifestation of physicochemical processes turned into a solid state. Depending on the conditions, the processes took place in two ways:

1) Fossilization, which took place in a marine environment with the formation of amber-like resin with a large amount of succinic acid;
2) Fossilization, which took place in an acidic environment in parallel with the processes of carbonization of plant residues that led to the formation of brown coals and lignites with the inclusion of various mineral types of fossil resins.

The age of succinite is determined by the time of segregation (separation) of soft resin from the parent vegetation and the accumulation of primary biogenic-sedimentary deposits of proto-amber in the understorey of amber forests and at some distance from them in the lower part of the Middle Eocene (Buchak time, Lutetian) (Savkevich 1970, 1983). The formation of succinite as a unique mineral and its primary placers in the littoral zone and remote parts of the shelf took place at the end of the Middle–Late Eocene to the Early Oligocene (Trofimov 1974, 1978; Serebrodolskyi 1984; Bogdasarov 2010). The authors also share this point of view (Matsui 2011, 2016; Matsui & Naumenko 2019).

Succinite is the most ancient ornamental and precious gem that the local residents of Ukraine mined in the Middle Dnipro Region and Ukrainian Polissia in the late Palaeolithic (18–16 thousand years ago). The surviving remains of archaeological succinite from Ukrainian burials are the oldest in Europe.

From the preserved fragments of succinate specimens collected by the hands of ancient people for 15 thousand years, we can analyse the state of preservation of succinite under extreme hypergene conditions.

The processes of physicochemical transformations of succinite in the upper part of the Earth's crust due to exogenous factors (weathering, oxidation, mechanical destruction, etc.) play an important role in changing the material composition of its chemical compounds and properties. In the process of oxidation, it becomes more brittle and fractured, its optical properties change, detachments appear, products from it lose their lustre and beautiful appearance. Long-term storage of succinite specimens and products made from it—the latter often having historical value—is a serious problem.

The issue of preserving the integrity of succinite and its processed products is extremely important. Succinite has unique properties and is widely used in industry, agriculture and medicine.

Weathering of succinite is accompanied by the removal of carbon, sulfur and a relative increase in oxygen with the formation of an oxidation crust of increased hardness. Amber corrosion is not associated with electrochemical reactions, but with chemical or physicochemical processes. Succinite is easily scratched, damaged by micro-impacts, and quickly oxidized. Being porous, it is easily permeable to liquid and gaseous substances. Also, solvents such as alcohols, benzene, chloroform, solutions of ammonia, acids or alkalis cause amber to swell or dissolve, leading to irreparable damage.

Due to the intensive development of succinite deposits in Ukraine, Belarus, and the Baltic countries, a sharp increase in demand for amber and its processed products, the expansion of trade and the great popularity of the gem, there is a need to study the problems of its preservation. First, it is the clarification of the storage conditions for raw succinite, amber products and collection samples on museum shelves, in private collections, conservation and restoration of succinite during archaeological excavations, etc. (Berezanska & Shumova 2002; Matsui & Solianik 2004; Popova 2008; Shumova 2011). This scope of problems is also covered in earlier works (Frakey 1990; Kosmovska-Tseranovich 2007; Pastorelli 2009; Pastorelli *et al.* 2013).

The aim of the research was to clarify the influence of hypergene alterations on the properties of amber, to determine the conditions of succinite alterations in natural environments, museum storage, and archaeological excavations.

The research object is Ukrainian succinite of Eocene–Oligocene age, whereas the research subject is the conditions of preservation of the natural state of succinite, its physical and chemical characteristics.

Analysis of recent studies

Departments of natural science, museum workers, and design artists have been studying this problem. In Ukraine, it is the Institute of Geological Sciences of NAS of Ukraine (Matsui & Solianik 2004, etc.), the State Gemmological Centre of Ukraine (Lysenko *et al.* 2016, etc.), the Institute of Archaeology of NAS of Ukraine (Berezanska & Shumova 2002; Shumova 2011, etc.), Taras Shevchenko National University of Kyiv (Nesterovskiy & Volkonskaya 2019, etc.); in Poland—the Polish Academy of Sciences Museum of Earth (Warsaw) (Maliszewski *et al.* 2013, etc.), in Belarus—Brest State A.S. Pushkin University (Bogdasarov 2010, etc.), in Russia—Kaliningrad Regional Amber Museum (Boikina & Manukian 2017; Problems... 2015, etc.).

Among the modern studies for assessing the structure and state of amber preservation, the methods of electromagnetic spectroscopy in the UV, visible, and infrared ranges, FTIR spectrometry are used (Kosmowska-Ceranowicz 2013).

Scientific conferences on amber mining, conservation and research are also held regularly. In 2015, an international conference 'Problems of restoration and conservation of amber' was held (Kaliningrad, Russian Federation), where basically all reports were devoted to the preservation and restoration of amber. But, unfortunately, publications on the preservation of raw amber and products from it often lack experimental data on the influence of hypergene processes on the alteration of succinite from the primary bedding.

Results and Discussion

Succinite represents amorphous high molecular compounds of organic substances. It is a fossil resin, which has lost most of its volatile components during the fossilization process. The approximate chemical formula is $C_{10}H_{16}O$ (Matsui & Naumenko 2019). Characteristic features are the high content of free succinic acid ($C_4H_6O_4$) in the amount of 3.5–5% to 8%, viscosity (low brittleness), quite noticeable solubility, refractoriness, ease of processing and a high degree of decorativeness. The chemical composition is variable. The content of Carbon is up to 80%, Hydrogen is about 10.5%, Oxygen is 10.5–11%, and Sulphur is 0.4% and more (Frakey 1990). The hardness on the Mohs scale is 2.0–2.5 (Matsui 2011). The hardness index is unstable (even in one piece it fluctuates within ±5%). Hardness increases with increasing impurities, in particular of iron sulphides. Wet samples of succinate just taken from the bedrock have a lower hardness than the pieces that have been exposed to open air. The colour is generally yellow with various shades—from almost colourless to yellow-brown, white, and even black. Moreover, varied colouration can be observed in a single specimen of succinite. This feature provides individual, unique colour images of the specimens. The degree of transparency is also quite variable—from perfectly transparent to opaque dark brown and black. Transparency depends on colour intensity and the presence of gas-liquid inclusions and voids in the gem. When heated, the opaque succinite becomes light golden in colour.

Succinite has a very low thermal and electrical conductivity (dielectric constant is 2.863). When heated to 140–200°C, it becomes elastic, at a temperature of 250–300°C it starts to boil, and at a temperature of 350–380°C it melts. According to the degree of transparency, it can be transparent, translucent, and opaque. The density is 0.9–1.10 g/cm^3, which roughly corresponds to the density of sea water. That is, succinite floats in salty water and sinks in freshwater, but due to the large number of air bubbles and other 'light' inclusions, it does not settle on the bottom and is freely transported over long distances in both sea and bodies of freshwater. This explains the amazing buoyancy of succinite in the surf zone and its resistance to repeated washing, transfer and reburial over tens of millions of years.

Succinite contains a variety of inclusions of remnants of the Eocene fauna and flora, which were captured during the separation of the soft resin and are preserved to this day (Fig. 2), as well as inorganic rock fragments, gas-liquid inclusions, air and water bubbles. Succinite occurs in the form of rounded pieces and inflows weighting from fractions of a gram to ten or more kilograms. Under ultraviolet light, it glows dimly, has shades from bluish white to yellowish green. When polishing is examined, areas of increased fluorescence appear in it.

The peculiarities of the material composition of succinite noted by the authors, which increase the resistance to adverse natural phenomena, were formed during the long-term fossilization (petrification) of plant resins. The fossilization of resins was influenced by hypergene processes that took place on the Earth's surface, in areas of active groundwater penetration and within the surf coastal (littoral) zone.

Among placer-forming minerals characterized by high hypergene resistance, succinite (a mineral of organic origin) ranks last (Table 1) (Shilo 2000). A number of authors (Savkevich 1970; Trofimov 1978; Serebrodolskyi 1984; Bogdasarov 2010; Matsui 2016, etc.) established and described hypergene changes in plant resins at all stages of the soft resin transition, from its outflow to the day surface to the completion of formation of the main features of the known to science FR mineral species, including succinite, the final formation of which took place in the littoral zone of the sea basin.

Fig. 2. A specimen of amber with inclusions; from the collection of the 'Ukrainian Amber World' association.

Рис. 2. Зразок бурштину з включеннями; з колекції Асоціації «Український бурштиновий світ».

Table 1. **Physical properties and constants of hypergene resistance of minerals according to Shilo 2000**

Mineral	Density, ρ, g/cm³	Hardness, H	Degree of hypergene resistance, pH	Constants of hypergene resistance, $C_{hr} \approx lg(pH)$
Platinum	21.5	4	86	1.93
Gold	16.9	2.7	45.63	1.65
Cassiterite	7	6.5	45.5	1.65
Zircon	4.7	7.5	35.25	1.54
Diamond	3.5	10	35	1.54
Topaz	3.6	8	28.8	1.44
Ilmenite	4.7	5.5	25.85	1.14
Quartz	2.6	7	18.20	1.26
Amber	1.07	2.3	2.46	0.39

In the primary bedding formed by succinite placers (fossil resins), the geochemical activity of minerals almost fades. The mineral is isolated from the external landscape environment. Thus, in stratigraphic sections not exposed by erosion, succinite as a whole retains its composition, properties and structure acquired in the previous stages of fossilization of plant resins.

However, being on the surface, amber is very vulnerable to aggressive factors, especially to oxygen and low humidity. Deterioration of the properties of amber is manifested in the destruction and loss of cohesion of the outer layers, as well as significant changes in texture and optical properties. Oxidation and atmospheric degradation are considered to be the main factors of negative alterations in amber. These processes affect compounds that have double bonds (C=C), which are oxidized to hydroxide, peroxide and carboxyl groups. As amber ages, the content of Carbon, Hydrogen and Sulphur decreases, and the amount of Oxygen increases. The mass of amber can increase, but also can decrease due to evaporation of low molecular weight oxidation products. The number of volatile terpenes may also decrease. The famous English gemmologist Helen R. Frakey (Frakey 1990) noted the disappearance of fluorescence in the collection specimens of simetite (Sicilian amber) of the 19th century without explaining the cause of this phenomenon.

Variable humidity and temperature conditions are also the factors that accelerate weathering of amber. In modern conditions, succinate—moved in some cases by natural processes or human economic activity on the day surface—is oxidized under the influence of oxygen, light (especially intense in ultraviolet rays), and elevated temperatures. Under the influence of oxidation, it changes colour and internal structure, an oxidative crust and sugar texture gradually appears, and then the amber is crushed into fine powder and dust. In the aeration zone, the polished surface of succinite specimens under the influence of oxidation changes noticeably only after 10–15 years (Savkevich 1970).

Thus, in the process of hypergene oxidation, in addition to the change in colour and the formation of an oxidation surface crust (protective shell against mechanical damage), the material composition changes significantly, Oxygen content increases, Carbon, Hydrogen and Sulphur contents decrease, succinite properties change. Succinite placers formed as a result of erosion and re-deposition of primary (bedrock) Eocene–Oligocene placers differ significantly, first of all, in the size of succinite grains, the degree of roundness and, in general, the presence of specimens more resistant to weathering. The features of hypergene destruction of succinite in deposits of loess and red-carbonate formations are described in our previous works (Matsui & Solianik 2004; Matsui 2011, 2016).

The amber from Neogene and Quaternary deposits of the Baltic, including glacial ones, has a stronger brown or red oxidation (weathering) crust and a slightly greater hardness, often represented by discoloured pieces (Trofimov 1974). The oxidizing crust was formed at the earliest stages of fossilization of resin secretions during resin leakage and in the soil of 'amber' forests, as well as in the form of thin secondary encrustations (0.2–0.3 mm), which covered the erased convexities of succinite pieces in the coastal-marine placers, indicating the existence of weak oxidation processes in the littoral zone.

The process of destruction of amber is rather long. A clear example of its destruction is succinite from archaeological excavations (Fig. 3).

Fig. 3. A specimen of destroyed amber from the Late Palaeolithic site of Mezhirich, Kaniv Raion, Cherkasy Oblast collected by V. Matsui. Photo by U. Naumenko.

Рис. 3. Зразок зруйнованого бурштину з Межирицької пізньопалеолітичної стоянки. С. Межиріч Канівського р-ну Черкаської обл. Зразок В. Мацуя. Фото У. Науменко.

Fig. 4. Amber from the ancient site of Mezhirich; specimen and reconstruction from the National Museum of Natural History of the National Academy of Sciences of Ukraine.

Рис. 4. Бурштин зі стародавнього городища Межиріч; зразок та реконструкція з Національного науково-природничого музею НАН України.

Late Palaeolithic human settlements of the Stone Age are located in the Dnipro Basin: Mezyn (Desna River), Dobranychivka (Supii River), Mezhyrich (Ros and Rosava rivers), Kyiv (Kyrylivska Street), Semenivka (Trubizh River), and Rivne (Horyn River), where the succinite was delivered by Stone Age hunters from local deposits of the Prypiat Amber-bearing Basin (Shovkoplyas 1965; Pidoplichko 1978; Nuzhniy 1997; Matsui 2002). The settlements with traces of unique residential buildings made of mammoth bones are buried under the loess rocks of the Upper Pleistocene, which in age correspond to the final stage of the Valdai glaciation. Specimens of mostly untreated succinite from the village of Mezhyrich are unevenly and deeply cut by unsystematically oriented cracks into cellular, irregularly shaped detachments (Fig. 4).

The finds contain small carbonate nodules, tightly 'soldered' into pieces of succinite, occasionally there are separate rounded nodules of destroyed succinite on carbonate cement. Often there are 'nests' of destroyed succinite, the outer shell of which consists of dusty and fine-grained fractions of a crushed oxidation crust. The central part of the specimen has a transparent red colour. These facts indicate the destruction of archaeological succinite in the burial, which occurred in the direction from the upper peripheral surface of the specimen to its central part.

Similar remains of succinite destroyed in loess rocks were found in the late Palaeolithic settlement of Barmaky (Rivne), the absolute age of which, based on radiocarbon dating of remains of mammoth bones, is 14 300±220 years, according to M. M. Kovaliukh of the Institute of Environmental Geochemistry NAS of Ukraine (Nuzhniy 1997).

The most numerous collections of archaeological succinite from all collections known in Europe collected in one place is established in the Hordiivka burial ground (14th–11th centuries BC) (Berezanska & Shumova 2002; Shumova 2011). A unique burial mound is located on the left bank of the Southern Bug River in the interfluve of its tributaries Bytiuh and Trostianchyk, near the village of Hordiivka, Trostianets Raion, Vinnytsia Oblast. There are three groups according to the state of preservation of products made of succinite. The first group (about 50%) of the products were broken into deep and small chaotically located cracks and scattered into fully oxidized amorphous small particles (except for a small number of necklaces, which retained a more or less dense structure) (Berezanska & Shumova 2002). The second group (about 30%) of the products had a more oxidized state, they still retained their shape, but with a little pressure the necklace turned into powder. The third group (about 20%) of raw materials retained a relatively dense base covered with an oxidative film that is subjected to chemical and mechanical cleaning. Shumova (2011) notes that already during the excavations, archaeologists observed an increase in the oxidation of succinite in the air, which led to a loss of transparency and colour, and with rapid drying, the products were covered with patina. The destruction of the unique archaeological collection was delayed due to restoration and conservation measures using a vacuum fixing method and by washing and impregnating each item with a solution of amber varnish, followed by fixing the surface with organosilicon composition.

Succinite from the scientific collection of the National Museum of Natural History NAS of Ukraine collected after World War II by I. G. Pidoplichko in the riverine cliffs of the Dnipro and ravine-beam network near the villages of Novi Petrivtsi and Stari Petrivtsi is coloured mainly in red and reddish-brown, which indicates its high oxidation state in the open air. Laboratory studies of the material composition of samples of the succinite collection were carried out.

Workers of natural history museums are well aware of the problem of oxidation (destruction) of succinite and, if necessary, apply storage rules in everyday practice, which limits the negative effects of light, air, temperature and humidity fluctuations in places of storage and demonstration of collection material. Popova (2008: 63) notes: 'Unfortunately, over time, amber is very strongly oxidized, so the amber plates made in the 17th century cracked, dimmed and lost their ability to transmit light. The images on them are barely readable.' According to Kosmowska-Ceranowicz (2013: 59): 'The collection of Baltic amber varieties, as it turned out, requires constant conservation. It is practically impossible to preserve a specific sample of amber in the same condition in which it was described on the day of purchase after twenty years, not to mention a period of 50 years or more. Museum workers are well aware of this, as they have to grind stones every time before exhibiting them. This is also known by amber masters who create works of art in which the contrast of colour and transparency of amber is essential' (the authors' translation).

Conclusions

The formation of succinite and its preservation in nature is entirely related to the specifics of the geochemical environment of host and overlying rocks. Hypergene alterations of succinite under surface and underground conditions occur from the end of the Early Oligocene, Neogene and Quaternary environments to geological modern times.

Amber succinite from native Eocene–Oligocene placers is often re-deposited in essentially carbonate rocks of the Neogene and Quaternary, which is associated with its destruction and even complete destruction.

Amber succinite placers, which are overlain by marine and continental sediments, generally continue to retain their basic properties acquired earlier. In cases of amber deposits exposure or their significant subsidence and other changes caused by the manifestations of geological, geochemical processes, including due to human economic activity, succinite undergoes significant transformations. During the oxidation of amber-succinite, the internal structure and colour change, the content of Oxygen, Carbon, Hydrogen, Sulphur, microhardness, luminescence parameters, etc. increase.

Archaeological succinite was formed in the Late Pleistocene–Holocene at the stage of intensive erosion of red-carbonate and loess formations of the Pleistocene, and its burial is confined to essentially loess rocks. Archaeological finds of amber during their stay in the cultural layers undergo physical and chemical changes, so their condition is very different from modern amber products. In addition, when removing the amber find, it is oxidized, which leads to cracking of the object, and under the influence of ultraviolet rays the material gradually loses transparency. That is, after extraction, the object from the archaeological amber continues to slowly deteriorate, which requires immediate conservation.

Illegal amber mining causes irreparable damage to the preservation of raw amber in natural conditions due to the disturbance of the geological layers with the inclusion of amber and its leaching to the surface, which leads to the natural destruction of the mineral.

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