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Miocene Dipterocarpaceae leaf from Sumatra and amber geochemical analyses

Una hoja de Dipterocarpaceae del Mioceno de Sumatra y análisis geoquímicos en ámbar

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ABSTRACT

A large leaf was discovered on a piece of fossilized resin from Jambi Province, Sumatra. Based on its location, mid-Miocene age, and structural similarity to modern leaves of members of the family Dipterocarpaceae, it is tentatively assigned to the genus ?*Dipterocarpus*. This action is supported by the presence of Group B resinites as determined by the use of carbon-13 NMR Spectroscopy. The fossil leaf was surrounded by a hard sub-bituminous coal that had been subjected to high temperatures and pressures that destroyed most of the resinite. The presence of a light brown covering surrounding the fossil suggests that there was erosion of silicified sandstone. The leaf would fall within the Malesian floristic Region together with some 25,000 other species of flowering plants. The Dipterocarpaceae predominately inhabit tropical regions of the Old World. Conditions that occurred in Sumatra when the leaf was fossilized probably greatly resembled those in the state coal mine in Wonthaggi. These Australian lignitic coals still contain associated fossil resin in the form of plant material such as leaves; however, acids destroyed most animal remains. The Sumatran resin that contained the fossil leaf under study was likely subjected to intense volcanic activity, as revealed by XRD studies. Trace amounts of tonstein minerals *i.e.* quartz, illite, and mica (muscovite) are related to volcanic ash falls. In fact, volcanic activity and its relationship to the increased production of resin during the Miocene may have continued since the middle of the twentieth century.

Keywords: coal mines, amber, Miocene, leaf, Indonesia.

RESUMEN

Una hoja grande fue descubierta en una pieza de resina fosilizada de la Provincia de Jambi, Sumatra. Con base en su ubicación, una edad del Mioceno medio y una estructura similar a las hojas modernas de miembros de la familia Dipterocarpaceae, se asigna tentativamente al género ?Dipterocarpus. Esto es apoyado por la presencia de resinitas del Grupo B, determinado con el uso de Espectroscopía de carbon-13 NMR. La hoja fósil estaba rodeada por carbón duro sub-bituminoso, que fue sujeto a altas temperaturas y presiones que destruyeron la mayor parte de la resinita. La presencia de una cubierta café claro alrededor del fósil sugiere que existió erosión de la arenisca silicificada. La hoja pertenecería a la Región florística Malesiana, que comprende otras 25,000 especies of plantas con flor. Las Dipterocarpaceae habitan predominantemente regiones tropicales del Viejo Mundo. Las condiciones que ocurrieron en Sumatra cuando la hoja se fosilizó, se asemejan probablemente a las encontradas en la mina estatal de carbón en Wonthaggi. Los carbones lignícticos australianos aún contienen resina fósil asociada en forma de material de plantas tal como hojas, sin embargo, los ácidos destruyeron la mayor parte de los restos de animales. La resina de Sumatra que contiene la hoja fósil bajo estudio fue probablemente sujeta a una intensa actividad volcánica, como lo revelan estudios por XRD. Cantidades traza de minerales tipo tungsteno i.e. cuarzo, ilita y mica (muscovita) se relacionan con caída de ceniza volcánica. De hecho, actividad volcánica y su relación con el incremento en la producción de resina durante el Mioceno pudo haber continuado hasta mediados del siglo veinte.

Palabras clave: minas de carbón, ámbar, Mioceno, hoja, Indonesia.

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1. Introduction

Indonesia is one of the world's largest producers and the biggest exporters of coal. According to information presented by Indonesia's Ministry of Energy and Mineral Resources, Indonesian coal reserves are estimated to last around 83 years if the current rate of production is maintained (Septiawan *et al.*, 2021).

Indonesia is located in the geographical region called Malesia. It is an area that includes Peninsular Malaysia, Borneo, Sumatra, Java, Sulawesi, Mollucas, Philippines, Lesser Sundas, and New Guinea (Figure 1). The area is geologically dynamic and volcanically active and is assembled from multiple terranes. It straddles zones of subducting Pacific and Indian Ocean seafloor and it currently includes more than 20,000 islands that reflect the most recent cycle of submergence and reconnection caused by eustatic sea-level variation. Tectonic plate collisions have caused the rise of mountains and the formation of marine basin making the region one of the most geologically complex on Earth (Kooyman *et al.*, 2019).

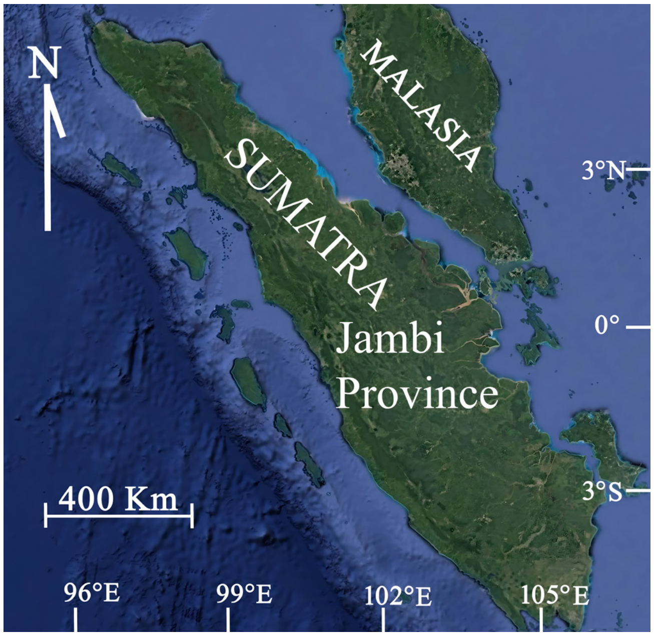

Figure 1 Location map of the Jambi Province in Sumatra, Indonesia.

One of the areas in Indonesia that is rich in coal is Jambi Province. Coal is an important source of revenue for the state, and Jambi province is one of the largest producers (Septiawan *et al.*, 2021). The primary source of the intensively mined coals of Southeast Asia was terrestrial plant matter. The coals were formed during periods of ever-wet (perhumid) climate (Ziegler *et al.*, 2003). Jambi Province is located in the middle of the east coast of Sumatra Island. The Jambi area is rich in natural resources originating from agriculture, plantation, and mining exploration. The piece of amber under study was found in an open-pit coal mine in this province (Figure 2).

The material under study is a large piece of Sumatran amber which features a magnificent nearly complete big leaf impression. The objective of our study is the taxonomic description of this leaf, the elucidation of the chemistry of the fossil resin and its paleoenvironment at the time of deposition. On the flora of the area, it is known that Dipterocarpaceae is the most common tree family encountered in Indonesia's tropical rainforest and plays an important role in the forest ecosystem function. The trees are found to grow mainly in the lowland forest however, they are also found in various other habitats. Dipterocarps are large trees with some recorded to be able to reach up to 70-80 m, thus making this family important because of its economic and ecological significance (production of timber, camphor, and resins). The Dipterocarpaceae encompasses three subfamilies having intercontinental distribution such as 1) Dipterocarpoideae distributed in Southeast Asia, 2) Pakaraimoideae endemic to South America, and 3) Monotoideae restricted to Africa, Madagascar, and South America (Kusumadewi *et al.*, 2023).

Dipterocarpoideae exhibit greater species diversity compared to the other two subfamilies. The family is disseminated across continents following a strict disjunct distribution pattern which raises a question of its place and time of origin. The Dipterocarpoideae clade dominates in Southeast Asia and almost 80% of its diversity has been

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found to occur in wet forests of Southeast Asia, particularly in Western Malaysia (Borneo) hence Southeast Asia was considered to be its center of origin. There are two plausible hypotheses proposed for the evolutionary history and biogeography of the family Dipetocarpaceae. One hypothesis supports a SE Asian origin (Into India hypothesis). The other suggests a Gondwanan origin (Out of India Hypothesis; Kundu, 2008).

Dipterocarps are an important source of resins. The resins (damar) from certain species of *Dipterocarpus*, Hopea, and Shorea are used for caulking *e.g.* boats among other uses. The resin is secreted in cavities and normally oozes out through the bark. The resins are of two kinds. The first is a liquid resin that contains resinous material and essential oils (oleoresins) remains liquid in nature and has a distinct aroma. It is often referred to as oleoresin in the literature. Commercial production is often through artificial wounding. The second is a hard resin which is called dammar when obtained from dipterocarps. This is the solid or brittle resin, which results from the hardening of the exudate following evaporation of the small content of essential oils. However, the classification of resins is very chaotic, and in the trade 'dammar' is also used occasionally to refer to an oleoresin. The hard resin (dammar) is found as natural exudations on living trees, in lumps on the ground beneath the trees, near dead stumps, or even found buried in the ground. These dammars are usually collected by aborigines. Natural exudation also occurs from trees that are unhealthy or damaged by the heartwood borer (Simmathiri and Turnbull, 1998).

Figure 2 Open coal mine where the amber piece was found.

The dipterocarp forests in the perhumid zone of Asia form the cradle for a considerable proportion of life forms found on Earth. Arguably, the only effective way to preserve a sizable portion of this biodiversity will be through affective management, including the production of timber and other valuable products (Simmathiri and Turnbull, 1998).

Historically, ancient records of dipterocarps report that the birthplace of Buddha was Lumbini, situated on the bank of the River Rohini where there were groves of Shorea robusta (sal), called 'Mangala Salvana'. Marco Polo's chronicles of 1299 mention the trade of camphor (from Dryobalanops aromatica) by Arabs since the 6th century (Simmathiri and Turnbull, 1998).

Nowadays both government and private organizations are actively involved in the research and management of Dipterocarpaceae such as reforestation and ex situ conservation (Kusumadewi *et al.*, 2023).

2. Geological setting

The specimen under study was found in an open-pit coal mine in the village of Tanjung Belit, located in Jujuhan Subdistrict, Bungo Regency, Jambi Province in Sumatra, Indonesia (Figure 1). The coal mine belongs to a private company (Pt. Sinar

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Mas, specifically, Pt. Sinamarinda Lintas Nusantara). Coal mining is an important industry in the development of Jambi Province (Daulay, 2020). Coal is one of the main commodities of energy resources in Indonesia, especially in the Sumatra region. South Sumatra basin hosted the largest and most economically significant coal deposits of the island, with rank mostly consisting of low-rank coal, such as lignite with two sub-bituminous coals (Tobing *et al.*, 2014). According to the coal mine workers, the amber piece was found at a depth of 80 meters, however, its exact stratigraphic position is unknown. The general geological setting of the Jambi Province includes sandstones, claystones with iron oxides, and intercalated coals, deposited in environments from marine at the top (Talang Akar Formation) to fluvio-deltaic near the bottom, or formed in the coastal shelf (Baturaja Formation). The age of the resin-bearing layers is Lower Miocene-Middle Miocene (23-13 Ma; Stach *et al.*, 2019).

3. Materials and methods

The Indonesian amber piece under study presents an irregular ovoid shape measuring in length 35 cm, width 20 cm, height 18 cm, and weight 4.43 kg (Figure 3).

The fossil under study appears as a tumbled freeform piece and has a slightly waxy feel to it. It has a megafossil plant inclusion resting on it, a magnificent leaf impression with petiole, and a much smaller partial leaf that appears to disappear into the resin. The color of the whole of fossilized resin is dark brown in the crust and tan opaque color inside. It presents countless millimeter-size laminations. It is believed that it was formed as a rolling 'ball' of amber by accretion.

Anderson and LePage (1995) reported the presence of resinite 'balls' (although much smaller in diameter) in some middle Eocene localities on Axel Heiberg Island in the Canadian Arctic. These peculiar amber 'balls' have also been found in amber deposits in the Dominican Republic measuring about 5 cm in diameter as observed by one of us (VF). Visual evaluation of the specimen under long-wave UV light (LWUV) shows a strong violet fluorescence of the crust and fossil leaf and greenish fluorescence of the tan-creamy laminations that form the bulk of the specimen.

The repository of the material under study is the Friedman-Constantino Amber Collection where it is kept under the inventory number FC-RC-430.

3.1. NUCLEAR MAGNETIC RESONANCE

Examination of the sample by nuclear magnetic resonance (NMR) spectroscopy was carried out on both carbon (^{13}C) and hydrogen (^{1}H, also called proton) nuclei. With carbon, the experiment operates on the powdered bulk so that the analysis is assured to characterize the entire sample.

Figure 3 Detail of nearly complete ?*Dipterocarpus* leaf.

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Examination of the hydrogen nuclei is carried out in solution, with deuterated chloroform (CDCl3) as the solvent. The solution phase may entail some loss of material due to partial insolubility. Normally, a sample of ca. 100 mg is powdered in a mortar to fine grains for the ^{13}C experiments and then dissolved in chloroform for the ^{1}H experiments. The two methods provide complementary, independent, phenomenological profiles of the materials.

In contrast to CG/MS, with which efforts are made to identify individual molecules out of a host of constituents, NMR spectroscopy provides molecular fingerprints either of the bulk or of the soluble portion (Lambert et al, 2013). Solution ^{1}H spectra were obtained at 500 MHz on a Varian Inova-500 spectrometer at room temperature without spinning. Spectra were referenced in CDCl3 to tetramethylsilane (TMS) at δ 0.0. Parameters for standard 1D spectra were as follows: spectral width 12,000 Hz, pulse width 60°, delay time 1.0 s, acquisition time 1.0 s, and scan number 4. Solid state ^{13}C data were recorded on a 400 MHz Varian NMR System with a 5 mm T3 PENCIL probe or on a 400 MHz Bruker Avance III HD NMR Spectrometer with a 4 mm HX probe. The magic angle spinning rate was set to 5000 Hz. The cross-polarization (CP) pulse sequence was used for normal proton decoupling on both spectrometers.

For interrupted decoupling (dipolar dephasing), a 50 μs (Varian) or a 48 μs (Bruker) delay was applied in the ^{1}H channel just before the 180° pulse in the ^{13}C channel. We used adamantane (Varian) or glycine (Bruker) to adjust the Hartmann-Hahn matching condition for normal CP experiments and to adjust the observation pulse and the delay time for dipolar dephasing. A typical parameter set was as follows: spectrum frequency 100.544 MHz (Varian) or 100.524 MHz (Bruker), spectral width 296 ppm, pulse width 3.4 μs for the 90° pulse for both ^{1}H and ^{13}C (Varian) or 2.5 μs for ^{1}H and 4.0 μs for ^{13}C (Bruker), pre-delay time 5 s, contact time 5 ms, acquisition time 50 ms, scan number 256, carrier frequency 110 ppm, and a ramped pulse with 83 Watts used in the ^{1}H channel during contact time. Solid state ^{13}C spectra were referenced to an external adamantane peak at δ 38.3 (Varian) or to an external glycine methylene peak at δ 43.4 (Bruker) and were converted to a scale with TMS at δ 0.0.

3.2. XRD AND ELEMENTAL ANALYSIS

X-ray diffraction is a non-destructive technique that observes and evaluates the properties of crystalline materials such as inorganic compounds and minerals. XRD exposes the material under study to monochromatic X-ray beams, which scatter through the sample's atoms, generating a unique diffraction pattern. This pattern is then compared to the ICDD's database (International Centre for Diffraction Data) to determine the exact compounds found within the sample. The types of minerals as well as their elemental composition contained in the fossilized resin can be identified by XRD analysis. XRD analysis was carried out on a sample of the fossilized resin under study.

Instrumentation used: Rigaku Ultima III x-ray diffractometer (performed at MES, Inc., Richardson, TX). The elemental analysis of the same sample was determined by Energy Dispersive X-Ray Spectroscopy (EDS), using a Thermo Scientific UltraDry EDS Detector. EDS was performed by the same consulting firm as above.

The analysis was conducted on the tan area of the fossil resin to perform XRD analysis and to determine chemical composition. The sample was sent to another consulting firm due to inconclusive results by the first firm (MES, Inc., Richardson, TX) by the XRD analysis. The second consulting firm was K/T GeoServices, Inc. in Gunnison, CO. The sample was sent to further evaluate the clay mineralogy (Clay X-ray diffraction analysis) of the same opaque tan area of the resin under study. Instrumentation used: Siemens D500 automated powder diffractometer equipped with a copper X-ray source (40kV, 30mA) and a scintillation X-ray detector.

4. Results

This is the first report of a foliar structure of a member of the family Dipterocarpaceae in amber. Members of this family of small to lofty trees that dominate the lowlands of Asia consist of at least 16 genera and 695 species. These trees supply most of the hardwood used in the world today (Heywood, 1985; Langenheim, 2003). The fossil leaf was discovered during the process of surface mining for coal. This mostly involved open pit mining and mountaintop removal.

The fossil was found at a depth of 80 meters in an open-pit coal mine. Based on its age, location and physical features cited below, we are describing this fossil as a new species of the genus Dipterocarpus. All parts of plants of this family possess resin canals that secrete an oily aromatic resin called dammar when wounded. The resin in contact with the fossil leaf was determined to be composed of Group B resins (polycadinenes) based on ^{1}H and ^{13}C Nuclear Magnetic Resonance analysis reported in the present study.

4.1. SYSTEMATICS

This fossil is composed of two foliar structures. The main structure is a nearly complete leaf while the accessory structure is a small portion of an attached second leaf. The description below refers to the main leaf, which is nearly complete except for the right tip which is replaced by a dark impression (Figure 3).

Order Malvales
Family Dipterocarpaceae
Genus *?Dipterocarpus* Gaertn.

Locality. Jambi (Djambi) Province, Sumatra, Indonesia.
Age. Early Miocene
Description. Leaf simple, oval-ovate in shape, 40 mm in length and 35 mm in greatest width; triangular and wedge-shaped (cuneate) at the base with a distinct petiole and stipules; midrib single; 9 linear veins margin slightly wavy, with a distinct drip tip, indument (hair covering) present, blade

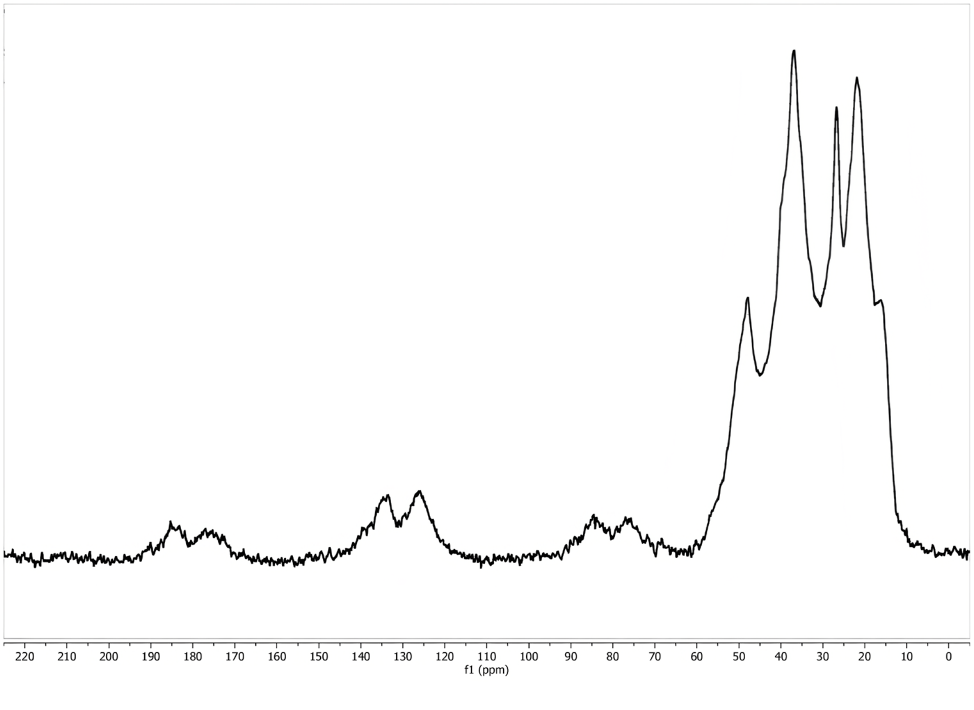

Figure 4 400 MHz ^{1}H spectrum of sample 2148. The peak at δ 0.0 is from the standard tetramethylsilane, that at δ 2.1 is from an impurity acetone, and that at δ 7.3 is from the solvent chloroform.

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margins entire, stipules leaving a persistent scar.

Comments. Some portions of the main leaf possessed superficial structures. These could represent insect remains (Lepidoptera) but not enough detail is present to be certain of their origin. However, Lepidoptera larvae are known to develop on dipterocarp leaves (Longatang *et al.*, 2021).

4.2. NMR STUDIES

^{1}H and ^{13}C Nuclear Magnetic Resonance studies of the Sumatran fossil resin under study (sample labeled 2148) from the Jambi deposit were assigned to Group B fossil resins in the NMR classification, whose chemical structure is based on polymers of sesquiterpenoid hydrocarbons, specifically polycadinenes (Lambert *et al.*, 2008; Figure 4 ^{1}H spectrum and Figure 5 ^{13}C spectrum).

4.3. X-RAY DIFFRACTION ANALYSIS

The mineralogical composition of the sample as reported from a consulting firm in Richardson, Texas, USA, proved to be inconclusive. The lack of sharp distinct peaks is indicative of an amorphous, most probably organic, non-crystalline material. Analysis of the produced diffraction pattern failed to yield any relevant mineral components. The same sample was further analyzed for Clay X-ray diffraction (XRD) by a firm in Colorado to evaluate its Clay mineralogy.

The results show the presence of quartz (0.1 weight percent), illite and mica (muscovite) 2.3% and amorphous (amber) 97.6%. The following minerals were not detected: smectite, kaolinite, chlorite, potassium of plagioclase feldspars, and amphibole.

4.4. ENERGY DISPERSIVE X-RAY SPECTROSCOPY

EDS spectrum results, run by the same consulting firm in Richardson, Texas showed high levels of carbon (87.81%), lower levels of oxygen (8.57%), traces of aluminum (0.13%), silicon (0.17%), phosphorus (0.03%), sulphur (0.07%), iron (1.95%) and nickel (1.28%).

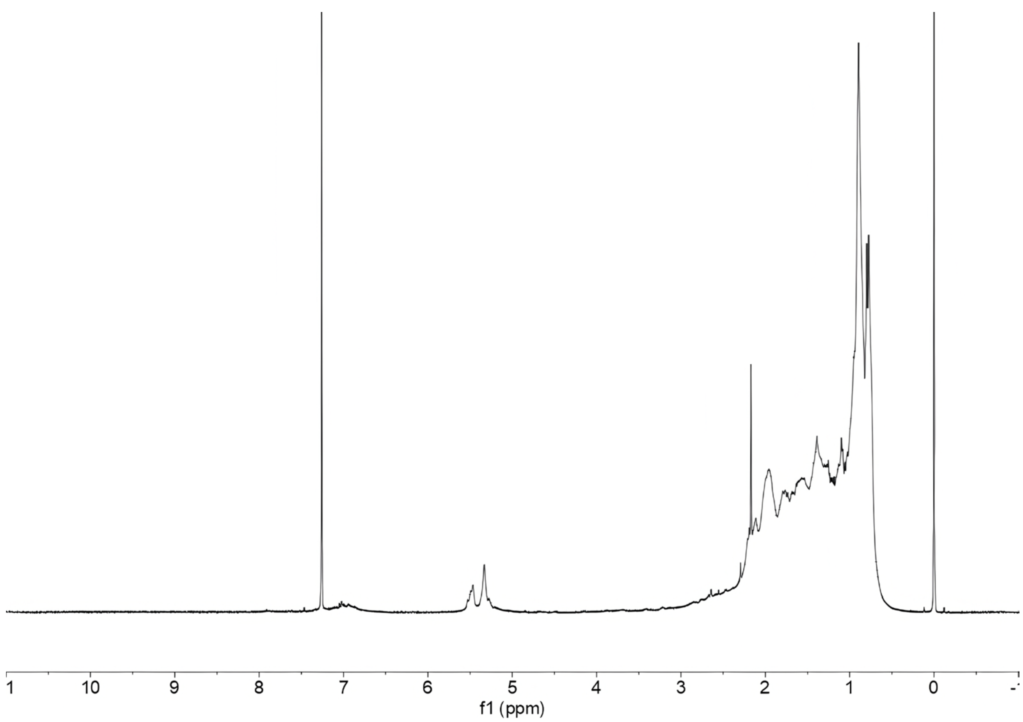

Figure 5 100 MHz ^{13}C spectrum of sample 2148, recorded with cross-polarization and magic angle spinning. The peaks at δ in the regions 70-90 and 170-190 are spinning sidebands.

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5. Discussion and conclusions

Indonesian amber is found in Neogene coal layers (Miocene, about 13 Million years ago) of the central Sumatra Basin on the island of Sumatra (Durham 1956; Leelawatansuk *et al.*, 2013). The amber is found mainly in a primary deposit (in the original location as opposed to alluvial or secondary deposits) between three district coal layers in open strip mines. The amber is a byproduct of coal mining but is not mined specifically since it is not economically feasible, occurring in deep layers under 30-80 m of sediments and rocks. Fossil resins from Jambi Province occur in immature coal layers (lignite beds). The paleoenvironment of Sumatra during the Tertiary period was warm and humid, being a part of southern Sundaland located about 10 degrees from the equator, with a paleoclimate favorable for peat formation (Friederich *et al.*, 2016).

Mineralogically, Sumatra amber is a Glessite resin that is formed from the resin of the deciduous tree family Dipterocarpaceae (Narudeesombat *et al.*, 2014). Glessite is a resin established by Helm (1881), based on the Latin word glessum, used by Tacitus in referring to amber used by the Est tribe who inhabited the beaches of the Baltic region where it was originally found. The first infra-red spectra comparing a modern dipterocarp resin with a Sumatran amber was reported by Langenheim and Beck (1965), in relation to the botanical origin of amber. Later the dipterocarp fossil resins were classified as Class II (polycadinenes) based on their chemical structure (Anderson *et al.*, 1992).

More detailed studies on 'Indonesian ambers' from lignite mines (Borneo, Sumatra, Sulawesi, Papua New Guinea) using Nuclear Magnetic Resonance revealed the widespread dipterocarp origin of these resins in Southeast Asia (Lambert et al, 2013). The Indonesian resins were assigned to Group B of the NMR classification, based also on the presence of polycadinenes in their chemical structure. Indonesian fossilized resins show other physical properties such as refractive index and specific gravity values that are consistent with data gathered on ambers in general (Leelawtnasuk *et al.*, 2013). The values obtained by EDS on the elemental composition of the resin under study (see Results section) are also consistent with those reported in the literature for ambers.

In 2017, Kosmowska-Ceranowicz and coworkers associated enhanced resin production with volcanic activity based on investigations on ambers from Sumatra and Borneo. These authors reported the presence of numerous mineral and rock inclusions in their structures *e.g.* pyrogenic quartz, fragments of tonsteins, volcanic glass, and various minerals related to volcanic phenomena. Indonesian amber presents a wealth of resin varieties, with varying degrees of transparency and color intensity (yellow, red to brown, light beige color, and transparent to translucent to opaque). These resin varieties were formed from different tree exudate outflows at different times and probably in different parts of the tree (Naglik *et al.*, 2018).

The creamy inclusions present in many samples of Indonesian ambers appear to be fragments of clayey rock captured by the plastic mass of fossil resin. According to Kosmowska-Ceranowitz *et al.* (2017), their SEM-EDS investigations indicate the presence of kaolinite, quartz, and chlorite and confirm the identification of the creamy light-gray inclusions as a tonstein. These authors further suggested that the region's significant volcanic activity in the Miocene is confirmed by the accumulations of volcanic tuff transformed into tonstein (Kosmowska-Ceranowitz *et al.*, 2015; 2017). Tonstein is a rock developed through the transformation of the pyroclastic material ejected by volcanoes and formed through subaerial weathering in a palustrine (swampy) environment. They are most commonly found in coal seams (Bohor and Triplehorn, 1993). Tonsteins are generally kaolinite or, less commonly, other clay minerals such as montmorillonite and illite.

The non-kaolinite component of the clay-mineral fraction represents an incomplete alteration of the volcanic ash to kaolinite, probably due to incomplete flushing of ions because of restricted

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water movement through swamps (Bohor and Pillmore, 1976). Based on our XRD data it is possible to infer that the sample under study occurred in an environment of stagnant and alkaline waters where illite was the final alteration product. This, alongside mica (muscovite) and quartz, are the only traces that suggest volcanic activity during the Miocene at the time of resin production and deposition. Volcanic activity in Sumatra during Paleogene-Neogene to Quaternary has been reported to have occurred in three semi-continuous cycles: late Oligocene-mid-Miocene, mid-Miocene to early Quaternary and late Quaternary (Van Bemmelen, 1949). This activity influenced paleomire and coal deposition all across Sumatra and may have resulted in tonstein layers (Triplehorn, 1990).

Concerning the fluorescence of the Sumatran resin under study, it is documented that the occurrence of aromatic hydrocarbons in fossil resins from swamp depositional systems is very likely (Naglik *et al.*, 2018). The reducing conditions are favorable for the aromatization of plant exudate compounds, however, the presence of aromatic hydrocarbons may be responsible for luminescence phenomena in fossil resins (Stach *et al.*, 2018). Drzewicz *et al.* (2020) stated that the source of fluorescence is not from a single compound but due to a large number of compounds embedded within the fossil resins. The presence of fluorescent unsaturated and aromatic compounds in fossil resins from different worldwide localities has been often reported in the literature (Bellani *et al.*, 2005). However, further studies are needed.

The amber specimen under study presents a strong fluorescence under LWUV light. The fluorescence is violet-colored on the leaf blade and the surface of the entire specimen. A greenish fluorescence is observed in the tan-gray portion of the specimen. The emitted fluorescence is likely affected by the chemical structure of the resin. The most intense fluorescence is found in resins from volcanically and tectonically active areas, as is the case for this Sumatran resin sample (Stach *et al.*, 2019). These events initiated significant environmental changes caused for example, by forest fires, atmospheric pollution, climate changes, etc. Fluorescent compounds may form during fires and might be transferred into resins from the surrounding environment.

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Handling editor

Nombre Apellido

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