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Chemical Characterization of Brazilian Miocene Amber Samples by Comprehensive Two-Dimensional Gas Chromatography Coupled to Time-of-Flight Mass Spectrometry (GCxGC-TOFMS)

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Amber is a fossil resin originated from polymerization of angiosperms or gymnosperms resins constituted mainly by terpenoids. The knowledge concerning amber molecular composition provides important information related to its botanical affinities. The objective of this study was the individual identification of terpenoids in Brazilian amber extracts, being the first application of GCxGC-TOFMS to acquire data concerning amber molecular composition and botanical affinities determination.

Three amber samples of Miocene age from Solimões Formation (Acre Basin, Brazil) were extracted (CH₂Cl₂:CH₃OH, 1:1, v:v) and analyzed in a GCxGC-TOFMS System Pegasus 4D (Leco). The column set used was a DB-5MS (30 m, 1D) and a BPX-50 (1.3 m, 2D). The compounds were tentatively identified by the use of ChromaTOF 4.22 Software, Nist Mass Spectral Database and elution order.

The total ion chromatograms acquired are analogous for all samples. Mono-, sesqui- and diterpenoids, decalines and aromatic compounds were identified. Concerning sesqui- and diterpenoids, aromadendrane, humulane, germacrane, caryophyllane, bisabolane, labdane, abietane and kaurane were some structural classes identified in all extracts. Triterpenoids were not detected. A careful analysis of the chromatograms revealed three distinct regions, associated to monoterpenoids, hydrocarbonic sesquiterpenoids and decalines, and polar sesquiterpenoids and diterpenoids. The same pattern was observed by GC-MS data; however, several coelutions were solved by GCxGC allowing the identification of important diagnostic structures to amber botanical origin. The molecular composition of the samples points to Fabaceae family. Such angiosperm group has resins based on sesquiterpenes that most often occur as hydrocarbons and diterpenes of labdane class, both were identified in the analyzed samples. The use of GCxGC-TOFMS in analysis of amber demonstrated the applicability of this technique in paleontological studies and its potential of distinguishing amber samples from different basins, ages and origin by the structured chromatogram.