



Problem of soot aggregates separation and purification for Carbon isotopic composition analyses – burning experiment and real black layers from speleothems examples

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Burning products are often used as an indicator of fire or prehistoric men activities. When it consists of macroscopically visible black layer it may be studied by different methods. When it is dispersed within sediment it is necessary to apply method for burning product separation. Soot aggregates as a result of incomplete combustion of organic materials are most reliable indication of burning. Size of soot particles is too small to observe by optical microscopy.

There are two main advantages of application of transmission electron microscopy (TEM) for investigations of samples formed as a result of organic materials (like wood) combustion. First, it makes possible to investigate not only morphology but also its interior structure. The carbon layers arrangement is characteristic for particles obtained from combustion processes, and it directly confirm that these particles were formed that way. And second, analysis of chemical composition using of EDS spectroscopy in transmission microscope are precise and its spatial resolution is about a few nanometers.

Burning chamber for wood burning experiments was constructed. It allows wood burning with controlling of burning temperature, carbon isotopic composition in carbon dioxide of burning atmosphere and carbon dioxide originated during burning. Burning products are collected on the plates with controlling of plates material, temperature and distance from flame.

Two types of samples were studied. The first type of samples consisted the products of recent wood burning. The second type of samples consisted of black layers collected from speleothems.

Soot aggregates were chemically separated from other burning products collected on plates. Process of chemical separation and purity of soot material were tested by TEM observations. Isotopic carbon composition at each step of soot separation as well as original wood fragments was analysed at the Isotopic Laboratory for Dating and Palaeoenvironment Studies, Polish Academy of Sciences in Warsaw. Measurements were performed using a Finnigan MAT 253 Mass Spectrometer coupled with a Flash 1112HT Elemental Analyser in continuous flow mode. Three international isotope standards were used to calculate the results: USGS 40, USGS 41 and IAEA 600. Significant differences of carbon isotopic composition in wood and different burning products were found. Carbon isotopic composition of wood fragments, “charcoal” and soot aggregates pre-treated (HCl and HF) before isotopic composition analysis was between -26 to -29‰. Black layer material with no pre-treatment had a $\delta^{13}C$ at the level -22‰.