

Summary

Many different and inconsistent results have been published about occurrence of fullerenes in natural samples. Simultaneously, reproducibility of positive results is often a difficult task. One of the possible reasons of contradictory and negative results is supposed to be inefficient extraction of fullerenes from geological matrices.

The goal of this work was to determine optimal conditions of ultrasound extraction, when applying this method to extraction of fullerenes from geological materials. Various parameters of ultrasound extraction were tested by sonication of bituminous coal in toluene with synthetic C_{60} added to powdered sample. High-performance liquid chromatography (HPLC) was used to determine fullerene content in the obtained toluene extracts. Results obtained by sonication in Tesla ultrasound bath showed, that higher concentrations of C_{60} in final extract can be achieved, when optimal sonication time is selected – the highest yield was observed after 30 min of sonication. Another important parameter appears to be the temperature of the sample during sonication. After 3 h of sonication in temperature range 280 – 285 K, concentration of C_{60} in extract was about 45 % higher than yields obtained by sonication in temperature range 290 – 310 K. The difference was less pronounced, when shorter sonication time (30 min) was chosen. A little increase of C_{60} concentration in toluene extract was noted, when preextraction of coal by pentane and pentane + toluene by Soxhlet extraction (Soxtec system) was involved before C_{60} was added to the sample. Filtration method may have a little effect on detected concentration of C_{60} too. Multiple extractions of samples already extracted carried only < 0,4 % of further C_{60} . Presence of nitrogen atmosphere above the sample or bubbling of N_2 into the suspension during sonication did not lead to increase of C_{60} content in the final extract, moreover, a strong decrease was observed, especially in the second case. Demineralization of sample after C_{60} was added led to strong decrease of yields of C_{60} as well. Considerably lower yields were observed, when Bandelin Sonorex ultrasound bath was employed.

By testing stability of C_{60} in toluene solution without presence of solid phase, no direct influence of increased temperature or ultrasonic irradiation on fullerene decomposition was observed. After sonication of C_{60} (Bandelin Sonorex) for 3 hours in toluene solution together with fraction of saturated hydrocarbons (separated from pentane extract of coal), a moderate decrease of C_{60} was detected. Further slight decrease was observed, when C_{60} was sonicated together with toluene extract of coal. Final concentration of C_{60} after 3 h of sonication was about 17 % lower, compared to the initial C_{60} content. It seems, that there are mechanisms of fullerene reduction in solution based on reaction with hydrocarbons during fullerene extraction. However, the decrease in fullerene concentration is not so expressive, compared to results of experiments, when solid phase was involved.