

RAPID CHARACTERISATION AND INVESTIGATION OF OIL SHALES BY THERMAL DESORPTION-PYROLYSIS-GC/MS USING MULTI-FUNCTIONAL PYROLYZER

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Industry burn oil shale directly as a low-grade fuel for power generation and heating purposes and can use it as a raw material in chemical and construction-materials processing. Oil shale has been gained attention as a one of the future energy resources as the price of conventional sources of petroleum has risen and as a way for some areas to secure independence from external suppliers of energy. However, oil-shale mining and processing involve a number of environmental issues, such as land use, waste disposal, water use, waste-water management, especially greenhouse-gas emissions and air pollution. The conventional analytical methods of oil shales has been used with Fischer Assay and thermogravimetric analysis but these are not enough for the fully evaluation of complex composition of oil-shales. In order to minimize these issues, rapid, reliable and precise analytical methods have been requested for long time.

The analytical system using a vertical type of micro-furnace pyrolyzer combined with capillary column GC/MS has been demonstrated itself as one of powerful tool for rapid characterization of various artificial and natural polymers. [1, 2]. In this work, the multi-functional pyrolyzer (Frontier Laboratories; Japan) was introduced with oil shale sample for the first time to estimate its potential in investigation of the oil shale organic matter (kerogen) generally known as a natural complex biogeopolymer and ancient precursor of natural petroleum in Earth's crust. The analytical system consists of pyrolyzer with GC/MS used for testing of the two representative Estonian Kukersite and Dictyonema oil shales is described as follows.

The system enables both flash pyrolysis and stepwise programmed thermal desorption/pyrolysis using deactivated stainless steel capillary column (Ultra ALLOY column; Frontier Laboratories Ltd. UA1-30M-1.0F), and evolved gas analysis of the oil shales in widely varied temperature region as from 100 to 800 °C using simultaneous detection mode of GC/MS and pulse flame photometric detectors (PFPD). As a specific feature for the Kukersite straight chain aliphatic saturated and unsaturated hydrocarbons with carbon number up to C₁₇ as well as rare long alkyl chain homologous 5-alkylresorcinols have been displayed in rapidly generated summary chromatograms. Both flash and programmed pyrolysis express the same regularities in pyrolysate compound composition while the concentrations of similar compounds differ. Also, one can see in the chromatograms abundance of aromatic compounds as alkyl derivatives of benzene, toluene, naphthalene, anthracene and phenanthrene typical not only for Kukersite but for many other oil shales as well. Furthermore, the trace amount of variety of sulfur compounds were also identified using PFPD and mass spectrum.

The potential of such multi-functional Py-GC/MS system includes thorough investigation the compounds formed resulting from Kukersite and Dictyonema flash and programmed pyrolysis, studying the regularities of formation different compounds depending on pyrolysis temperature creating the fundamentals of advanced technologies for oil shale more effective liquefaction in obtaining liquid fuel and chemicals. With this aim the Estonian Kukersite and Dictyonema oil shales are investigated in the frames of international collaboration between Frontier Laboratories Ltd. and Laboratory of Oil Shale and Renewables Research at Tallinn Technical University (Estonia).

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