CDS olutions

APPLICATIONS INFORMATION USING ADVANCED SAMPLE HANDLING TECHNOLOGY

Analysis of Algae using Pyrolysis and THM

Analytical pyrolysis provides a means to analyze complex materials, even biological samples, using GCMS. Microbes, tobacco, plant fibers and foods have all been studied using this technique. Bio-fuel sources, including vegetable oils, wood, lignin and grasses have also been analyzed using Py-GC/MS, which uses thermal energy to convert large molecules like polysaccharides and proteins into smaller, volatile molecules suitable for GCMS.

Algae samples may be analyzed using a variety of thermal techniques. Figure 1 shows a sample of dried algae pyrolyzed at 600°C directly to the GC. At this temperature, the biopolymers are degraded and the pyrogram consists of characteristic fragments, including aromatics, nitriles and aliphatics in addition to compounds desorbed intact. Since they are a potential source of fuel, algae are frequently analyzed for their oil content. By pyrolysis, the fatty acids are generally fragmented, producing the normal alkanes and alkenes seen in the pyrogram.

Another approach is thermally-assisted hydrolysis/methylation (THM) in which a reagent like tetramethyl ammonium hydroxide (TMAH) is added to the sample, which is then warmed to produce the methyl esters of the fatty acids. Figure 2 shows a sample of algae heated to 400°C in a quartz tube to which 2 µL of TMAH solution (25% in methanol) was added. The sample was allowed to stand for 5 minutes after the TMAH was added, prior to analysis. Under these conditions, the biopolymers are not degraded, so the resulting chromatogram is simpler, consisting almost exclusively of the fatty acid methyl esters released from the algae.

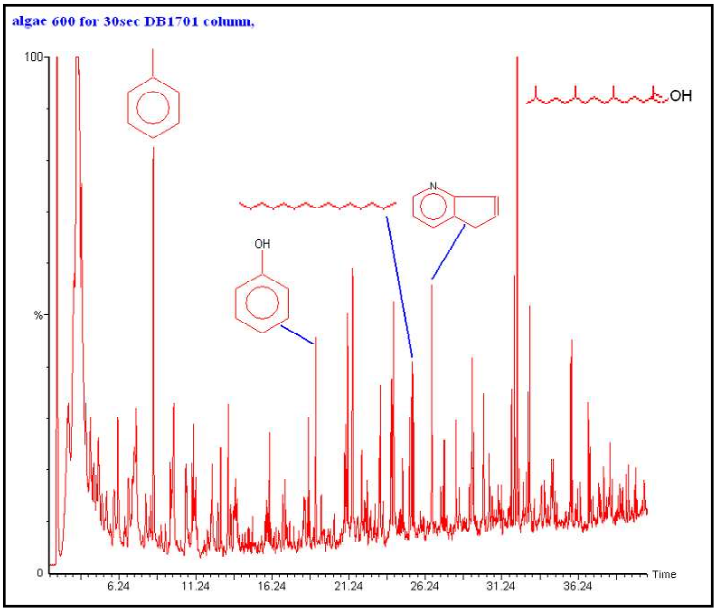

Figure 1. Dried algae pyrolyzed at 600°C.

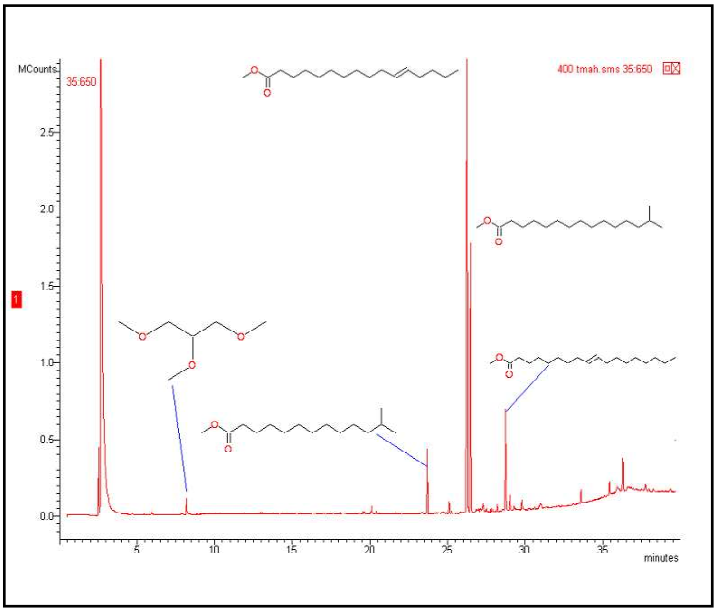

Figure 2. THM analysis of algae using TMAH.

Equipment

Samples were analyzed using a Pyroprobe 5250 Autosampler interfaced to a GC/MS. Samples were placed onto the top of a quartz spacer rod, then topped with a small plug of quartz wool to contain the TMAH solution.

Pyrolysis

CDS Analytical Pyroprobe 5250 Autosampler

Pyrolysis:	600°C for 15 seconds
THM:	400°C for 20 seconds
Valve oven:	300°C
Transfer line:	300°C

GCMS

Carrier gas:	Helium
Split:	50:1
Column:	25 m x 0.25 mm 5% phenyl
Oven	
Initial:	40°C for 2 minutes
Ramp:	10°C/minute
Final:	300°C for 5 minutes
Range:	35 to 550

FOR MORE INFORMATION CONCERNING THIS APPLICATION, WE RECOMMEND THE FOLLOWING READING:

Challinor, J. M., Review: The development and application of thermally assisted hydrolysis and methylation reactions, J. Anal. Appl. Pyrolysis, 61 (2001) 3-34.

Additional literature on this and related applications may be obtained by contacting your local CDS Analytical representative, or directly from CDS at the address below.

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