MILESTONE
HELPING CHEMISTS

MICROWAVE SAMPLE PREPARATION OF ASPHALT SAMPLES FOR R&D AND Q&C LABORATORIES

CUSTOMER

The research project, led by Willem Suitela at TNO Mobility & Built Environment in Delft, the Netherlands, focuses on building materials such as cement and concrete, road construction materials such as asphalt concrete and aggregates, and asphalt in particular. The aim is to make asphalt either more durable or more recyclable.

CHALLENGE

To accurately determine the composition of the elements in asphalt, it is essential that the sample be broken down into the smallest possible particles and completely digested with concentrated acid during sample preparation. Most microwave instruments had difficulty completely digesting these samples.

SOLUTION

Willem Suitela tested microwave digestion with ETHOS UP. In conjunction with ICP-AES, the system worked well for his elemental analyses. It was the only microwave oven that had completely digested the reference sample.

BACKGROUND

The “average” life of a right lane on a highway is about 11 years. The other lanes are much more durable, lasting about 6 years longer. Ideally, either the asphalt would have a longer lifespan or it would be optimally recyclable. Enter Willem Suitela, senior researcher at TNO Mobility & Built Environment in Delft, the Netherlands. Suitela has spent 32 years researching building materials such as cement and concrete, road construction materials such as bituminous concrete and aggregates, and asphalt in particular. He retired in April 2024 after a distinguished career. In the last week of his working life, he talked about his research and the need to pass it on to the next generation of researchers.

UNIQUE RESEARCH INTO ASPHALT

Asphalt “rejuvenation” is just one of Suitela’s many research projects, but it is quite unique. “There’s very little research being done on asphalt; it’s our niche,” he explains. “There are also no standard methods, so there’s a lot of trial and error. But you live and learn.” When he says “you,” he means himself and his colleague Marla Cambil, who joined him in March 2023. She earned her bachelor’s degree in chemistry from the Rotterdam University of Applied Sciences and, with her previous expertise in powders, was well-positioned to take over from Suitela. Suitela was able to hand over his job with peace of mind and focus on making plans for his retirement. “First, I’m going to travel, and then I’m going to take up my old hobby, fishing.”

LAB PROFILE
ETHOS UP | ASPHALT

RHEOLOGY, FT-IR, AND ELEMENTAL ANALYSIS

In addition to rheology and FT-IR spectroscopy, TNO also performs elemental analysis in its Delft laboratory. “You can use the composition of the elements in the asphalt (such as lead, zinc, vanadium, iron, molybdenum, potassium, calcium) as a fingerprint,” explains Cambil, who joined the conversation. To perform this analysis accurately, it is essential that the sample be broken down into the smallest possible particles and completely digested with concentrated acid during sample preparation.

ANALYZING ASPHALT WITH AN ICP-AES

At first, Suitela used a different method of digestion, he recalls. “You just added acid and hoped for the best.” He recently switched to microwave digestion using the Milestone ETHOS UP microwave digestion system from Salm en Kipp. In conjunction with ICP-AES, this system worked well for his elemental analyses. “I had been looking at microwave technology for asphalt digestion for a couple of years and started experimenting with a used model. I discovered that the combination of hydrochloric acid and nitric acid worked well for digestion, but that sometimes you need to add hydrogen peroxide or hydrofluoric acid, for example, when dealing with blast furnace slag. My second-hand microwave couldn’t handle it and wasn’t powerful enough.

Preparing the vials for digestion in the ETHOS Up high-performance microwave digester. (Photo by Marco Vellinga)

TESTING WITH REFERENCE SAMPLES

As a test, he sent building material samples and an unspecified “TNO reference sample” to several microwave oven suppliers for analysis. “Most of the microwave systems had trouble completely digesting the powders”, he says. But the ETHOS UP, with its 2 x 950 watt magnetrons and rotating diffuser, did the job. It was the only one that completely digested the reference sample. “I thought it was an advantage that **Milestone specializes in microwave instruments**. They know what they’re doing.”

What is asphalt?
Asphalt - also known as bitumen - is a mixture of hydrocarbons left over after distillation of the lighter fractions of crude oil. This highly viscous liquid is used to bind aggregates and minerals and gives asphalt concrete its black color. Approximately 85% of all asphalt is used to make asphalt concrete.

From pavement to asphalt
Before asphalt can be analyzed, it must first be cleaned from a lump of asphalt pavement. The lump is first crushed, and the aggregates are washed and filtered out in a sort of “asphalt washer” and centrifuge. Next, the solvents are distilled from the mixture. The viscous residue (asphalt) is then ready for analysis.

NON-QC APPLICATION

Microwave technology has become quite common for sample preparation in many QC laboratories. However, its use in research, as in the TNO laboratory, is less common. The ETHOS UP system came with many pre-installed methods - from makeup to soil - that can be easily selected from a menu on the touchscreen display. But asphalt is not one of them. “We have to develop those methods ourselves,” says Cambil. “We have a basic setup, but we want to be able to digest and analyze larger amounts of material.”

MICROWAVE DIGESTION

Although digestion with ETHOS UP is not necessarily faster than the “old” method, because sample preparation still involves a lot of manual work, **microwave digestion has proven to be highly effective and reliable**, explains Cambil. “We can process 15 samples at a time and the run time is 1 hour. A big advantage is that we use much less sample material than before, only 50 mg and 10 ml

“A big advantage is that we use much less sample material than before and achieve 100% digestion for asphalt.” - Marla Cambil, researcher at TNO Mobility & Built Environment

After digestion, the asphalt or construction material is analyzed in this ICP-AES. (Photo by Marco Vellinga)

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of reagent per vial. **We achieve complete digestion for asphalt.** By combining the contents of three vials, we can achieve a reliable measurement in the ICP. "We are now experimenting with increasing the amount of material in the ETHOS vials to see if we can still get complete digestion with a different solvent mix," adds Suitela. "If we can get this to work, we will be able to transfer the samples in their entirety to the ICP-AES without the need for an intermediate step."

"Microwave digestion is not necessarily faster, but it is very effective and reliable." - Willem Suitela, Senior Researcher, TNO Mobility & Built Environment

RESEARCH INTO MORE SUSTAINABLE ASPHALT

TNO will continue its research into asphalt, as there are many aspects of the material that need to be explored. One of them, sustainability, is a hot topic. Questions such as 'What is the quality of the asphalt and how does it affect the life of the pavement' and 'How do additives such as fibers and polymers affect the strength and flexibility of the pavement' provide good angles for research. Another important question is 'What is the best way to recycle old asphalt?' "Recycling is also very important for construction materials," says Suitela. "We have already started to analyze the powders in concrete. We want to know their exact composition and whether or not they are suitable for reuse. We also want to study the effect of particle size and pore size on the strength of the concrete. There are so many interesting topics for my successor to work on in the coming years," says Suitela, "and a reliable microwave digestion is crucial to all of them."

THE BENEFITS OF THE ETHOS UP MICROWAVE FOR TNO

- Optimal sample preparation for ICP, ICP-AES, AAS, etc.
- Accurate and safe control of the digestion process through easyTEMP contactless temperature control in all vessels, and automatic pressure control through a safety spring release mechanism.
- The high-pressure capacity of the SK-15 eT rotor allows reactive samples to be safely digested.
- Reusable Teflon vessels suitable for all applicable acids such as HCl, HNO_3, H_2SO_4, and HF, etc.
- Teflon-coated stainless-steel door with innovative opening and self-resealing mechanism.
- Very powerful magnetrons (2x 950 Watts) for fast and complete digestion.
- Advanced reaction sensors for comprehensive quality monitoring.
- Compact control terminal with easy-to-use touchscreen display.
- Ergonomic advantage: no need to work in a fume hood.
- Consistent and reproducible results.
- Safe and reliable.

ABOUT MILESTONE

With over 50 patents and more than 20,000 instruments installed in laboratories around the world, Milestone has been widely recognized as the global leader in metals prep technology for the past 30 years. Committed to providing safe, reliable and flexible platforms to enhance your lab's productivity, customers worldwide look to Milestone for their metals digestion, organic extractions, mercury analysis and clean chemistry processing needs.

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