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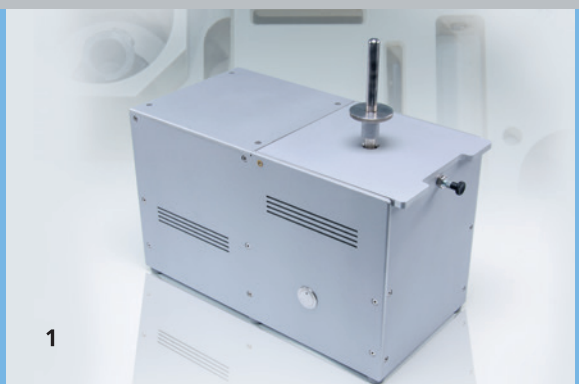
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1 *Operating instrument*
2 *Sampler*

MICROFLUIDIC-BASED SYSTEM FOR THE DETERMINATION OF ASPHALTENES IN CRUDE OIL

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Introduction

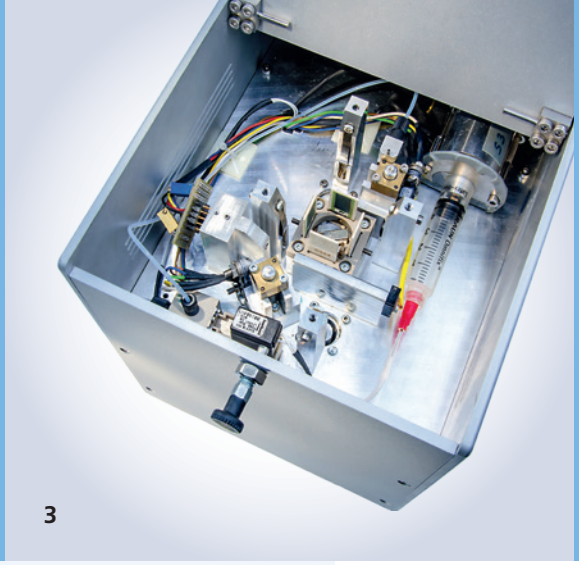
Crude oils are composed of diverse substances. Among them asphaltenes, defined as the n-heptane-insoluble, toluene-soluble components of carbonaceous material, constitute the largest aromatic fraction in petroleum. They show a variable concentration in crude oils within individual reservoirs and create a myriad of production problems in both exploration and crude oil production. Therefore, it is of utmost interest to determine the asphaltene content of crude oil to deliver it to the appropriate refinery.

Competences

A portable stand-alone demonstrator based on a microfluidic chip for asphaltene determination in crude oil in the concentration range of 0.1 to 15 % by weight (10 % relative repeatability) has been developed, which follows the standard protocol IP143 (Fig. 1 & 3). Instead of weighing the precipitated and dried asphaltenes, an optical setup to measure the asphaltene concentration via absorption was established. A special sampling device allows for metering of 50 µl and 100 µl of oil, respectively (Fig. 2). Using several crude oils with varying asphaltene contents a calibration curve was established.

Setup

The microfluidic chip (Fig. 4) is made of PEEK to withstand solvents and oil samples, and can be produced by injection molding when higher numbers are needed. It consists of a mixing reservoir (with a filter and a magnetic stirrer), two solvent reservoirs for n-heptane and dichloromethane (DCM), respectively, a waste reservoir, two turning valves with connected channels, and glass rods for optical detection. The reader instrument contains all mechanical, electronic and



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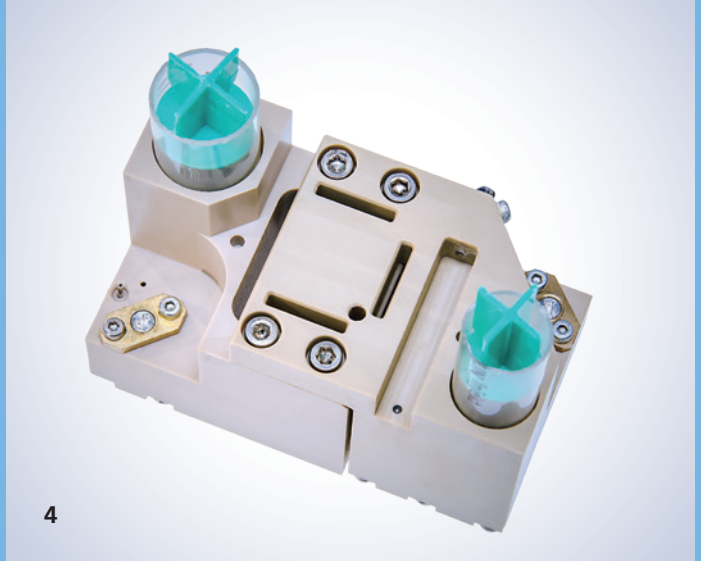

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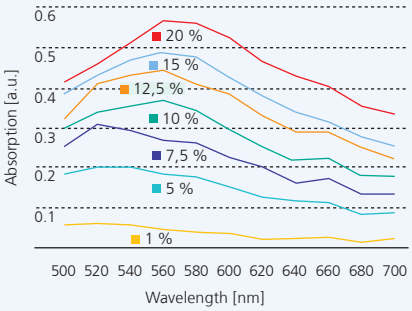
Fig. 5: Optical detection of asphaltenes dissolved in DCM on the chip.

5.1 Absorption curves for different asphaltene concentrations in % solved in DCM



5.2 Change in absorption for different wavelengths as a function of asphaltene concentration

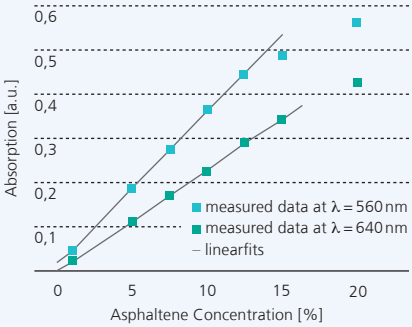
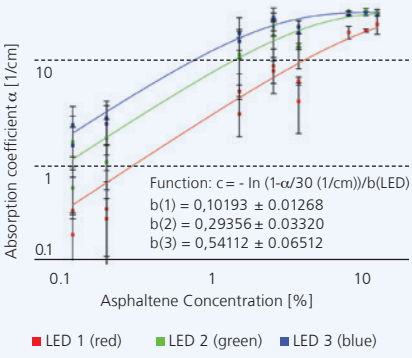


Fig. 6: Calibration curves using several crude oils with varying asphaltene contents.



components necessary for chip operation and is controlled by a PC using LabView software.

To meter crude oil, a sampler (Fig. 2) was developed, which works independently of viscosity. The sampler is dipped into the crude oil and a defined volume of oil is entrapped in a special cavity. It is inserted into the chip and oil is released into the solvent for mixing. Meters of 50 µl or 100 µl sample volume are available with a standard volume deviation of 3 % in order to cover the full asphaltene concentration range of 0.1 to 15 % by weight from a fluidic point of view.

Within the chip all processing steps are controlled by the reader. The crude oil sample is mixed using a magnetic stirrer with a 20-fold excess of preheated n-heptane (40 °C) to precipitate the asphaltenes. The precipitate is filtered, washed with further hot n-heptane to remove any entrained oil, redissolved in DCM, and optically analyzed (Fig. 5). The calibration curve is based on the values of several oils with varying asphaltene content using three different wavelengths (470 nm, 530 nm, 615 nm) (Fig. 6). It was shown that the full concentration range can be detected. However, the variability of the results suggests further improvement so that the device can comply with the target for the repetitivity and reproducibility – not only for high and medium but also for low concentrations.

Summary

The optical analysis of isolated asphaltenes is carried out by a combination of different wavelengths implemented in the analysis system to address both requirements:
a) high absolute concentration resolution for small concentration values (0.1-3 % by weight) and b) covering the whole concentration range from 0.1 % up to values of 15 % by weight.
By analyzing several crude oil samples with varying asphalthene content a calibration curve can be established. Asphaltene contents were determined by the standard protocol IP143. (This method already revealed a deviation of about 10 % by weight). The limiting value for the concentration resolution is the signal noise. The typical standard deviation was determined to be <0.001-0.02 absorption units.

Concentration range	Achievable concentration resolution	Relative error in weight
0.1 %–3 % by weight	0.004 % (absolute concentration) at 500 nm	4 %
0.1 %–15 % by weight	0.185 % (absolute concentration) at 600 nm	<1.2 %

References

WO 2009/001096 A1/EP 2009 423 A: Sample Plate (BP Oil International Limited).

Project Partner: BP Oil International Limited

3 *Operating instrument: housing opened to insert the microfluidic chip*

4 *Disposable microfluidic chip*