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MICROWAVE ASSISTED EXTRACTION OF EXTRACTABLE ORGANIC MATTER IN ROCK SAMPLES

The characterization of organic matter in the potential source rocks has always been of interest for petroleum geochemists and exploration teams in the oil and gas industries. Microwave assisted solvent extraction is a well-established sample preparation technique applied in several official methods. Milestone's ETHOS X equipped with fastEX-24 eT rotor was used in this study to prove its efficacy in the extraction of extractable organic matter in rock samples

INTRODUCTION

Petroleum source rocks consist of sedimentary organic and inorganic matter components. The former encompasses both terrestrial and marine (plant and animal) materials accumulated and preserved in sediments and sedimentary rocks such as shales and carbonates. The characterization of organic matter (quantity, quality, generating potential and maturity) in the potential petroleum source rocks has always been of interest for petroleum geochemists and exploration teams in the oil and gas industries during source rock studies (or evaluation) and hydrocarbon exploration respectively.[1]

An important parameter to be evaluated is the amount of extractable organic matter (EOM) commonly performed with ultrasonic extraction working with a solvent mixture of dichloromethane and methanol.

Despite its good mechanical effect on the rock, sonication methods require high solvent volume and a long extraction time, often requiring multiple extractions of the same aliquot.

Thanks to the selective heating of the materials, Microwave-Assisted Solvent Extraction (MASE), a well-established sample preparation technique, is a reliable and efficient alternative to ultrasonic methods, enabling extractions with reduced solvent volume and time.

This application report aims to compare the extraction efficiency of microwave, working with Milestone ETHOS X equipped with fastEX-24 eT, to the sonication protocol. It represents a guideline for EOM analysis on petroleum source rock samples.

EXPERIMENTAL

EQUIPMENT

- Milestone's ETHOS X.
- fastEX-24 eT rotor[2]
- 100-mL disposable glass vials.
- SFS-24 (Simultaneous Filtration System).
- Analytical balance

Figure1- ETHOS X and fastEX 24 eT

SFS-24

STANDARD AND REAGENTS

Standards, surrogates and internal standard were purchased by Sigma Aldrich. Pesticide grade solvents must be used in these tests. Sodium sulfate anhydrous, silica gel (activated for at least 16 h at 130°C) and glass wool or paper filter were used in the work up procedure.

SAMPLES

Five rock samples were used to compare sonication and microwave extraction. Three samples out of five with low organic content while two samples with high organic content.

SAMPLE PREPARATION

Conventional ultrasonic extraction was performed using a mixture of dichloromethane (CH_2Cl_2) and methanol (CH_3OH) solvents in ratio of 93:7. Each sample requires three extraction cycles.
The same solvent mixture was used to compare the performance of microwave extraction using Ethos X with fastEX-24 eT.

Samples, dried and ground, were weighed directly into the 100-mL extraction disposable glass vials. 30 ml of solvent mixture was used and the glass vials were closed (automatic capping tool available).

EXTRACTION PROCESS AND CLEAN UP

The proper built-in method was chosen. The MW program used for this work was the following:

Step	Time (min)	Power (W)	Temperature (°C)
1	15	up to 1600	110
2	10	up to 1600	110

Table 1 - Microwave Program

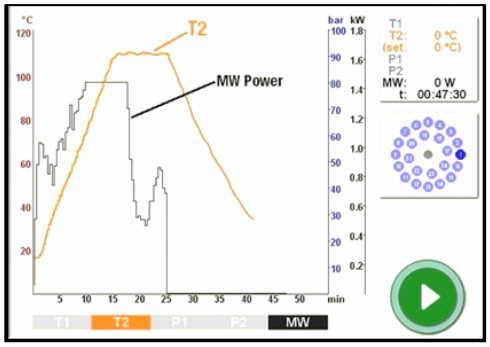

Figure 2 – Microwave run profile

After the extraction, samples were filtered with milestone SFS-24 simultaneous filtration system using sodium sulfate anhydrous. The vials were rinsed with additional solvent aliquots. SFS-24 allows to filter 24 samples simultaneously with different types of filters available. Extracts and rinse solution were collected together. The extract was subsequently dried with nitrogen flow for the gravimetric evaluation.
To avoid interference of sulphur with target molecules, sulphur removal from the extract might be necessary. For this purpose, place activated copper turnings in vials and leave them overnight.

APPLICATION REPORT

ETHOS X – EOM – Extractable organic matter

ANALITICAL STEP

The mass of EOM was reported in mg/g of extract (or percentage) and it is obtained by drying the solvent and weighing the residue. EOM values are used to compare the extraction performance in this study.

RESULTS AND DISCUSSION

Results of the comparison between fastEX-24 T and sonication are showed in the tables 2.

Sample	EOM (mg/kg)	
	Sonication	Microwave (*ETHOS X*)
1	273	322
2	125	201
3	315	466
4	35672	41569
5	29873	38760

Table 2 – EOM extraction results

The study was performed comparing the extraction efficiency of ETHOS X *vs* sonication extraction. As reported in table 2, ETHOS X promoted a more efficient extraction of EOM working with reduced solvent volume and enabling a much faster extraction process. Moreover, ETHOS X extraction did not require multiple extractions on the same aliquot, but a simple 40 min process was enough to achieve high extraction efficiency (cooling step included).

After the extraction with dichloromethane/methanol, the samples are usually fractionated on a chromatography column using silica gel (or alumina) as solid phase, and three different solvents – hexane, dichloromethane, and methanol as mobile phase. This chromatography separation allows to divide three different fractions, aliphatic hydrocarbons, aromatic hydrocarbons and polar fraction, for further evaluations.

CONCLUSION

The results demonstrated the efficiency of the ETHOS X with fastEX-24 eT rotor for the EOM extraction. For all the samples the extraction yield achieved with Ethos X was higher compare to the ultrasonic extraction.

The fastEX-24 eT rotor enables simultaneous solvent extraction of up to 24 samples in only 40 minutes.

Contamination, memory effects, and cleaning are completely eliminated thanks to the use of disposable glass vials. The use of contactless temperature control ensures high reproducibility of the extraction process.

ETHOS X provides extracts with the lowest solvent usage and significant time compared to all the other extraction techniques.

REFERENCES

1- Waheed Gbenga Akande, A review of experimental procedures of gas chromatography-mass spectrometry (gc-ms) and possible sources of analytical errors, Earth Science, 2012; 1(1):1-9

2- ETHOS X and fastEX-24 eT https://www.milestonesrl.com/products/microwave-extraction/ethos-x-for-environmental

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MILESTONE Srl - Via Fatebenefratelli, 1/5 - 24010 Sorisole (BG) - Italy
Tel: +39 035 573857 - Fax: +39 035 575498
www.milestonesrl.com email: analytical@milestonesrl.com