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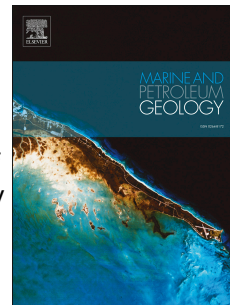
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Development of new method for D/H ratio measurements for volatile hydrocarbons of crude oils using solid phase micro-extraction (SPME) coupled to gas chromatography isotope ratio mass spectrometry (GC-IRMS)

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Highlights

- **The hydrogen isotope fractionation was not observed**
- **The D/H analytical errors range from 0.8‰ to 4.2‰**
- **SPME-GC/IRMS is efficient for δD analysis of volatile hydrocarbons**

Abstract: In compound-specific isotope analysis (CSIA), one major challenge is the effective sample treatment and the isolation of sufficient amount of analytes for accurate measurements for isotope ratios. The hydrogen isotope ratio (D/H) of normal level hydrocarbons in crude oils has been successfully used as an isotopic tracer in oil-oil and oil-gas correlations, but little progress so far has been made for small size hydrocarbons (C₆-C₁₅). In this paper, we presented a novel method for sample treatment and achieved the D/H ratio analysis for these particular compounds. The method can be characterized as an efficient approach featured by SPME (solid phase micro-extraction) coupled to GC-IRMS (gas chromatography-isotope ratio mass spectrometry). In this investigation, the CAR/DVB/PDMS fiber was evaluated and used as the absorptive material, and the operating conditions, including the extraction time (0.1 min ~150 min) and the extraction temperature (-30 °C ~ 140 °C), which may affect the adsorption amount, were carefully evaluated, and it was found that the adsorption yields varied considerably not only with the operating conditions but also with the molecular size. In addition, the possible hydrogen isotopic fractionation, which may exert strong influence on D/H ratios, were also discussed, it was found that the hydrogen isotopic fractionation were not observed in the extraction process, and the D/H ratios obtained via SPME-GC/IRMS were in good agreement with the

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