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Combining molecular fingerprints with multidimensional scaling analyses to identify the source of spilled oil from highly similar suspected oils

Peiyu Zhou [a], Changshu Chen [a], Jianjun Ye [a], Wenjie Shen [c,d], Xiaofei Xiong [a], Ping Hu [a], Hongda Fang [a], Chuguang Huang [a], Yongge Sun [b,*]

[a] South China Sea Environmental Monitoring Center, State Oceanic Administration, Guangzhou 510300, PR China
[b] Department of Earth Science, Zhejiang University, Hangzhou 310027, PR China
[c] School of Earth Science and Geological Engineering, Sun Yat-sen University, Guangzhou 510275, PR China
[d] Guangdong Provincial Key Laboratory of Mineral Resources & Geological Processes, Guangzhou 510275, PR China

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ABSTRACT

Oil fingerprints have been a powerful tool widely used for determining the source of spilled oil. In most cases, this tool works well. However, it is usually difficult to identify the source if the oil spill accident occurs during offshore petroleum exploration due to the highly similar physiochemical characteristics of suspected oils from the same drilling platform. In this report, a case study from the waters of the South China Sea is presented, and multidimensional scaling analysis (MDS) is introduced to demonstrate how oil fingerprints can be combined with mathematical methods to identify the source of spilled oil from highly similar suspected sources. The results suggest that the MDS calculation based on oil fingerprints and subsequently integrated with specific biomarkers in spilled oils is the most effective method with a great potential for determining the source in terms of highly similar suspected oils.

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1. Introduction

During offshore petroleum exploration, exploitation and transportation, oil spill accidents may occur for a variety of reasons (*e.g.,* over-pressuring, mechanical failure, corrosion of pipeline, ship collision, etc.). Accidents bring about severe economic losses and can also cause potential long-term damage to the marine ecosystem. The Deepwater Horizon oil spill in the Gulf of Mexico in 2010 and oil leaks at the Penglai 19–3 oil field in the Bohai Bay, China, in 2011 are two recent major disasters (Hayworth et al., 2011; Mulabagal et al., 2013; Sammarco et al., 2013; Wang et al., 1999, 2013). The first issue in ocean management is to determine the ownership of responsibility, which requires technical support to identify the source of spilled oil quickly with strong and accurate evidence (Munoz et al., 1997; Wang and Stout, 2007).

Identification of the source of oil spills is one of the important aspects in the fields of environmental forensics due to the practical requirements accompanying the development of petroleum industry, which covers chemistry, environmental science, law, geochemistry and other related subjects (Philp, 2007; Wang and Stout, 2007; Wang et al., 2011). Traditionally, gas chromatography (GC), gas chromatography/mass spectrometry (GC/MS), and high performance liquid chromatography (HPLC) have been used to extract oil fingerprints for *n*-alkanes, isoprenoids, steroids, terpenoids, and polycyclic aromatic hydrocarbons. The compounds of interest are usually considered to be stable after accidents in terms of their chemical properties, and they can be tracers to track the source of spilled oil (Barakat et al., 1999; Daling et al., 2002; Malmquist et al., 2007; Salas et al., 2006; Stout et al., 2001; Wang et al., 1994, 2006; Wang et al., 2011; Wang and Fingas, 2003; Yim et al., 2011). Undoubtedly, oil fingerprints have been a powerful tool with great successes in determining the source of spilled oil. In most cases, it is easy to identify the source of spilled oil by the oil composition characteristics of organic molecules, even directly by an overview by gas chromatography when the oil fingerprinting characteristics of the suspected oils vary from each other greatly. However, the situation will be different because oil fingerprints usually can not work well if the oil spill occurs in the sea area where an oilfield group is developing. As such, the suspected sources are possibly complicated by crude oils derived from same source strata with high similar oil fingerprinting characteristics despite being from different hydrocarbon reservoirs and experiencing different migration fractionation and/or secondary

* Corresponding author.
E-mail address: ygsun@zju.edu.cn (Y. Sun).

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in-reservoir alterations. Furthermore, the surrounding oil facilities of the incident such as platforms, pipelines and storage facilities may be involved in the accident. Therefore, two main factors will play important roles with respect to source identification. One is the inherited "genetic relationship" among suspected oils from the oilfield group. Another is the field processing technologies applied during offshore oil production such as oil multi-blending, and oil–gas–water phase separation, resulting in difficulties in tracking the source of the oil spill.

To address the difficulties in identifying the source of spilled oil from highly similar suspected oils using traditional GC and GC/MS techniques, here we introduce the multivariate statistical method, integrated with traditional oil fingerprints analyses, for investigating an oil spill accident that occurred in a developing oil and gas field in a petroliferous basin of the South China Sea. The aim of this study is to show how oil fingerprints combined with mathematical methods can identify the source of spilled oil from highly similar suspected sources. The results could have great potential for application in tracing the source of spilled oil in complex situations.

2. Samples and experiment

2.1. Sampling and sample preparation

A mysterious oil spill occurred in the waters of the South China Sea, and there is one oilfield group with several development platforms outside the incident area (Fig. 1). Crude oil is pumped to the platform through pipelines followed by oil–gas–water separation, then transported to an island terminal by pipelines for further processing and storage, and finally sold to oil refineries.

Based on the background of the oil spill accident, there are several possibilities for the source of spilled oil, including from an oil tanker, platform, submarine pipeline and others. Nevertheless, the spilled oil might drift away from the initial site afterwards. Therefore, samples were selected from platforms, from the island terminal adjacent to the incident area, and from commodity inspection oils prepared for sale. The detailed backgrounds of the spilled oil and suspected oils are listed in Table 1.

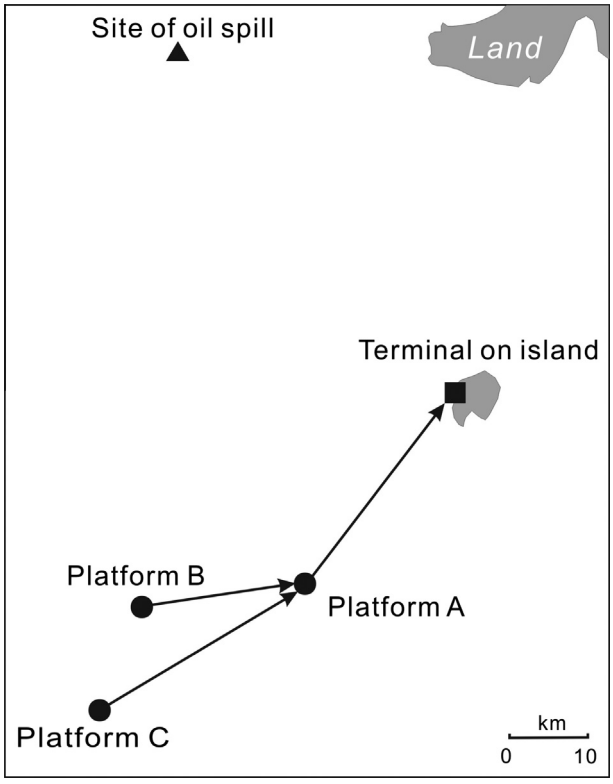

Fig. 1. Map showing the location of spilled oil and its distance to the oil platform and the island terminal.

Weighed samples of spilled oil and suspected oils (approximately 0.4 g) were dissolved in 8 ml of chromatographically pure hexane with anhydrous sodium sulphate for removing water, followed by ultrasonic oscillation for 20 min to fully dissolve the samples. Asphaltene, a non-volatile macromolecular organic matter unsuitable for GC analysis, was deposited and removed from the hexane dissolved oils using high speed centrifugation at the rate of 8000 rpm. 400 μL of supernatant was dissolved in 0.6 ml of chromatographically pure hexane for GC/MS measurements. Due to the demands for a quick response to the oil spill, compound-grouped fractions (*e.g.*, aliphatic, aromatic and polar (NSO) fractions) were not prepared here.

2.2. Gas chromatography (GC) and gas chromatography–mass spectrometry (GC/MS)

The aliphatics were determined on a Shimadzu GC2010 GC equipped with a flame-ionization detector (FID) and DB-5 column (30 m × 0.25 mm × 0.25 μm), and the oven temperature was started at 60 °C (2 min) and then increased to 300 °C at 6 °C/min (23 min). GC/MS analyses of spilled oil and suspected oils were performed on an Agilent 5975C mass spectrometer coupled with an Agilent 7890A gas chromatograph. Chromatographic separation was achieved with a 30 m × 0.25 mm ID fused silica capillary column coated with a 0.25 μm film of DB-5 ms. The oven temperature was started at 60 °C (2 min) and then increased to 300 °C at 6 °C/min (18 min). Helium was used as the carrier gas with a flow rate of 1.0 ml/min. The transfer line temperature was 280 °C, and the ion source temperature was 230 °C. The ion source was operated in electron impact (EI) mode at 70 eV. The selected ion monitoring (SIM) mode was used with *n*-alkanes of 85, naphthalenes of 128, 142, 156, 170 and 184, phenanthrenes of 178, 192, 206, 220 and 234, dibenzothiophenes of 184, 198, 212 and 226, fluorenes of 166, 180, 194 and 208, chrysenes of 228, 242, 256 and 270, steranes of 217 and 218, and terpanes of 191.

Table 1
Sample backgrounds.[a]

Sample No.	Locations	Types
Y1070	The accident waters	Spilled oil
Y308	The output of pipeline from platform B (to platform A)	Suspected oil
Y302	The output of pipeline from platform C (to platform A)	Suspected oil
Y301	The output of pipeline from integrated platform A (produced by platform A only)	Suspected oil
Y376	The output of pipeline from integrated platform A (produced by platforms A, B and C, and followed by oil–gas–water separator)	Suspected oil
Y393	Mixed oil after de-water and heat exchanger	Suspected oil
LCY1	Commodity inspection sample from the island terminal (7 days before oil spill was found)	Suspected oil
LCY2	Commodity inspection sample from the island terminal (2 days before oil spill was found)	Suspected oil

[a] The development platform usually has two functions: pumping crude oil from underground reservoirs and preliminary countermeasures. The mixed oil of platform A, platform B and platform C was transported to the island terminal after the three-phase separation of oil, gas and water. There were two oil sales from the island terminal (two and seven days before oil spill was found).

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