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Monitoring of the peculiar fluorescence behavior of oil-in-water emulsions prepared in an aromatics-saturated water phase

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It is well-known that produced water (PW) consists of a water phase rich with polar components – mainly aromatic hydrocarbons – and filled with non-polar, oil droplets. Before discharge to the sea, the PW is submitted to deoiling processes that can only remove dispersed oil, leaving behind an “appropriate-to-discharge” water yet rich with aromatics and other dissolved components that cannot be detected by the reference analytical method recommended by the OSPAR Convention. Fluorescence spectroscopy arises then as an alternative technique for monitoring the total hydrocarbon content in PW because it has, among other advantages, the ability to detect fluorophores in both dissolved and dispersed phases. However, since crude oil is a complex matrix, understanding the emission spectra of PW and crude oil-in-water emulsions can be very challenging as they result from several different compounds interacting in addition to inner filter effects and fluorescence resonance energy transfer. Thus, we addressed the peculiar fluorescence behavior of oil-in-water emulsions with the aim to investigate the spectral changes according to the increase in known concentrations in the oil phase, given a constant concentration in the water phase. A mixture of crude oil in ultrapure water (2:1 v/v) was prepared and, after 72 h, the water phase was collected and centrifuged at 10,000 rpm for 15 min to remove oil droplets and keep only dissolved, hydrophilic molecules. A 5.0 g L⁻¹ dispersion of the same crude oil was prepared using the saturated water phase as a dispersing medium to prevent hydrophilic components to migrate from the oil droplets to the surrounding continuous phase. 1.5 mL of the saturated water phase – herein referred to as “0 mg L⁻¹ sample” or “blank” – in a 10 mm pathlength quartz cuvette was taken as the starting point of the measurements, to which aliquots of the 5.0 g L⁻¹ dispersion were sequentially added and shaken vigorously. The spiked oil concentrations ranged from 161 to 1250 mg L⁻¹, and 3D fluorescence landscapes (excitation from 300 to 350 nm, emission from 320 to 580 nm) were recorded at each concentration, using both right-angle (RA) and front-face (FF) cuvette geometries. Increasing concentrations led to redshift and a decrease in the emission intensities in RA spectra and an increase in FF intensities, which might be associated with different optical pathlengths for RA and FF geometries. While the light travels through a longer path across the sample in RA geometry, it hits more fluorophore molecules along the way resulting in a gradual increase in inner filtering. On the other hand, the FF geometry leads to shorter path lengths and therefore is less affected by inner filter effects associated with higher oil concentrations. Further data treatment by trilinear decomposition of the 3D fluorescence landscapes indicated that the saturated water phase (blank) has a high concentration of some chemical components, which tends to decrease with additions of oil by RA and is almost constant by FF. Those components have an emission peak at shorter wavelengths compared to the bulk oil inside the droplets, which indicates that the addition of oil droplets triggers the migration of only some of the dissolved components to inside droplets, driven by different partitioning coefficients. While RA data shows signals from both dissolved and dispersed fluorophores in oil-in-water emulsions, apparently FF geometry captures mainly the signal from dissolved species. Therefore, the analysis by both geometries allows for identifying which part of the fluorescence signal is due to dissolved or dispersed phases.

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