FTIR Micro-Spectroscopy Study of Petroleum Rod Wax Deposits Formed in an Oil-Producing Well

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The deposition of organic solids, such as waxes and asphaltenes, in oil well equipment is a major problem occurring in various petroleum-producing regions worldwide and leads to decreased efficiency and a higher cost of produced oil. The solid deposits represent very complex mixtures, usually dominated by long-chain hydrocarbons (mainly n-alkanes but also iso-, cyclo-alkanes, and aromatic compounds) with various proportions of resins/asphaltenes, trapped oil, water, and inorganic material. Many questions regarding the liquid-solid phase transitions, mechanisms of nucleation, aggregation, and deposition of these complex mixtures under the changing pressure and temperature conditions of production are still not well understood, which presents a major problem for the development of efficient preventive and remediation technologies.

The present study is a part of a series of studies we performed at the X19A and X26A beamlines, using the advantages of synchrotron radiation techniques, to characterize the same sample set of naturally-formed wax deposits collected from the sucker pump rods of an oil-producing well. Samples were taken in order of increasing depth and temperature and encompassed depth and temperature ranges of ca. 6000 ft. and 30°C, respectively. Fourier transform infrared (FTIR) micro-spectroscopy is a nondestructive technique for the sample and provides both imaging and compositional information. These advantages allowed us to target characterization of aggregates with sizes from 20 μm × 20 μm to 150 μm × 150 μm present in the same wax deposit sample.

The preliminary interpretation of the results of this study allowed us to identify two major types of aggregates present in the same wax deposit – predominantly non-polar and predominantly polar ones (Figure 1). The FTIR spectra of the first type aggregates demonstrate strong characteristic bands of CH2 and CH3 groups that could be related to long-chain normal and iso-alkanes identified by our previous high-temperature gas chromatography studies. FTIR mapping of a single aggregate further revealed a heterogeneous distribution of the hydrocarbons and presence of carboxylic acids with identical to the hydrocarbons distribution. The carboxylic acids, most likely long-chain acids, are identified by characteristic bands in 2800-2100 cm⁻¹ region and are in much lower concentration compared to the hydrocarbons. The occurrence and distribution of carboxylic acids is a new finding and could be related to the processes associated with oil-water interfacial phenomena and interactions in micro-emulsions. The second type of aggregates identified in our FTIR study also have the characteristic bands of CH2 and CH3 groups but with relatively lower intensity compared to the first type. In addition, bands characteristic for aromatic, sulfide, sulfoxide, and nitrogen-containing compounds are present together with a well-defined hydrogen bonding region associated with NH and/or OH groups. Moreover, it was found that characteristic bands for a number of inorganic compounds are specifically associated with this type aggregates, mainly carbonates of Ca, Fe, Mn, Ni, Zn, and Pb. This latter finding complements and provides an independent line of evidence about the likely chemical state of trace metals in these deposits obtained by our XRF study at the X26A beamline.

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Figure 1. FTIR point spectra of different solid aggregates from the same wax deposit (#7) illustrating the compositional characteristics of discussed two types of aggregates.