Crude Oil Asphaltenes Studied by Terahertz Spectroscopy
Mohamed M. Matoug and Reuven Gordon*

Department of Electrical and Computer Engineering, University of Victoria, P.O. Box 1700, STN CSC, Victoria B.C. V8W 2Y2, Canada

ABSTRACT: Terahertz time-domain spectroscopy (THz-TDS) was used to study the asphaltenes in different crude oils. THz-TDS has a feature of measuring the amplitude and time delay and consequently the refractive index and absorption coefficient spectra simultaneously. Our approach was based on measuring the THz signal from neat crude oil samples and comparing it with the THz signal after removing the asphaltene from the oil samples (maltene). The results show that the differences in the time delay and the peak amplitude between the neat oil and the maltene have a linear relation with the asphaltene content. The refractive index spectra of the asphaltene show variation in the low THz frequencies and comparable spectra in the higher frequencies. The absorption of the asphaltene was mild, and no distinctive absorption feature was observed except for some narrow absorption peaks that we attributed to water molecules adsorbed on the asphaltene.

INTRODUCTION
Over the past 3 decades, crude oil has remained the largest source of energy in the globe, with approximately 33% of the energy in 2016. Operations in the oil and gas industry are full of complexity, and the presence of asphaltenes is critical to different phases of the oil industry. Asphaltenes are the heaviest, densest, and most polar components in crude oil. They are a complex mixture of thousands of chemical components that have a deleterious effect on the production, transportation, and refining processes. Because of their chemical and structural complexity, asphaltenes are defined by their high solubility in aromatic solvents (e.g., toluene) and low solubility in n-alkanes solvents (e.g., n-heptane). Asphaltenes' tendency toward precipitation and deposition often causes serious problems for the wellbores and pipelines such as clogging and corrosion, which reduce the oil flow and increase the nonproduction time due to maintenance. To address these asphaltene-related issues, researchers have investigated spectroscopic and analytical techniques for quantification and basic understanding.

The measurement of the asphaltene content is an important parameter to determine the quality and properties of oil. Therefore, an accurate and efficient method to determine the asphaltene content is crucial. There are few standard methods reported in the past to measure asphaltenes. They are gravimetric-based methods that work by adding n-alkane to precipitate the solid particles, followed by separating them by either filtration or centrifugation, and finally weighing the asphaltene using a balance scale. Even though this method is simple, it is time-consuming and requires a lot of solvent and a laboratory environment. The main disadvantage of this approach is the poor reproducibility that can show variations up to 20%, particularly from oil with low asphaltene content.

There have been numerous reports to address the gravimetric method issues using spectroscopic techniques. Part of these works aimed to measure the asphaltenes directly from the oil without extractions using techniques such as fluorescence spectroscopy, Fourier transform infrared, near-infrared (NIR), mid-infrared, all along with chemometric methods. On the other hand, another work has used a combination of separation and optical absorption efficiently to measure the asphaltene. The latter approach proved to be more accurate, as it removes the uncertainty that comes from the analogous and interfering molecules to asphaltene such as resin. Complementary to the asphaltene content measurement, measuring the asphaltene precipitation onset point is important for understanding the asphaltene phase behavior, and it is often measured by refractive index-based techniques. Since optical absorption and refractive index have been important techniques to probe different properties of the asphaltene, it would be beneficial to find a technique that provides both simultaneously. Terahertz time-domain spectroscopy (THz-TDS) has this advantage intrinsically, but there is a lack of literature on this topic.

Terahertz technologies are being applied in a wide range of areas such as imaging, security, medical communications, and spectroscopy. The THz-TDS technique gives information about both amplitude and phase of the sample under test, which enables us to calculate the real and imaginary parts of the refractive index simultaneously, with negligible
harmful effect to the sample due to the low photon energy of the waves.\textsuperscript{16,22} Recently, this spectroscopic technique has found its way to petroleum applications in different reports. It has been proven that the THz technique can be used to qualitatively identify the source of crude oil among different samples based on absorption and the refractive index spectrum.\textsuperscript{26,27} Other works include detecting the disaggregation of the crude oil particles in the presence of a magnetic field,\textsuperscript{28} characterizing wax crystals in waxy crude oil,\textsuperscript{29} measuring the water content in crude oil,\textsuperscript{30} measuring the properties of some crude oil products,\textsuperscript{31,32} natural gas,\textsuperscript{33} and coal,\textsuperscript{34} and all of these have only been presented over the past few years.

In this work, we study asphaltenes using THz-TDS in crude oil samples that have different asphaltene contents. Our approach was to investigate the effect of the asphaltene fraction on the THz signals of the crude oil. To do that, we have compared both the THz time-domain signal and frequency domain spectrum between the neat crude oil and the deasphalted crude oil samples (maltene) of different oils. We found that the difference in the time delay of the peak of the THz time-domain signal correlates well with the asphaltene content of the crude oil. On the other hand, the absorption of the time-domain peak signal seems to correlate with the asphaltene content but with less sensitivity. We have also noticed that the crude oil frequency domain spectrum has absorption lines that match the lines of the water. These absorption lines disappear when the asphaltenes have been removed from the oil.

\section*{RESULTS AND DISCUSSION}

The interesting feature of THz waves to propagate through an opaque oil sample\textsuperscript{28} made it convenient for us to study asphaltene in crude oil. Figure 1 shows THz time-domain signals of a crude oil sample that were taken at two thicknesses; 10 and 20 mm. It is clear that the oil absorption is relatively low. In fact, when considering the Beer–Lambert law for this oil sample, we can find that THz waves can be detected easily even at thicknesses larger than 100 mm. This will make THz a promising technology for in situ, noninvasive crude oil characterization.

Figure 2a shows the time-domain waveform of the reference scan (empty sample holder), neat oil, and the deasphalted oil (maltene) from oil 1 in the 10 mm thick side of the sample holder. There are few observations that can be made about the peak time and the peak amplitude. First, we can see that there is a negative shift in the time delay between the neat oil peak time and the maltene peak time. Second, there is change in the peak amplitude between the neat oil and the maltene, where the peak amplitude became a little stronger after removing the asphaltene. The measurement was done for oil samples 2 and 3 as well, and it was found that the time shift and the change in the peak-to-peak amplitude, between the neat oil and maltene, have a linear relation with the asphaltene concentration as shown in Figure 2b.

The oil samples 4, 5, and 6 which have less asphaltene content (<2%) have been measured by the 20 mm side of the sample holder. Figure 2c shows the time-domain waveform of the reference, neat oil, and the maltene of oil 4. Increasing the sample thickness will result in longer travel time for the THz pulse and larger time/amplitude shift between the oil and maltene, therefore, an increase in the resolution. Figure 2d shows the relation between the asphaltene content and both the shift in time delay and the change in peak-to-peak amplitude, and the same linear trend can be seen here too. The only sample where THz transmission was not achieved was oil 7. This is not consistent with the fact that oils and hydrocarbons have mild absorption, which has been shown here and in the literature.\textsuperscript{28–30} Therefore, we think that this might be due to impurities in the fluid, sand, for example, that have high absorption in the THz range.

To have a clearer linear fit, we have combined the data in Figure 2b,d in one plot as shown in Figure 3. This is done by normalizing the time delay data of oil (4–6) by the ratio of the sample’s thickness (20 mm/10 mm) and by expressing the peak-to-peak change in terms of optical density (OD) change (ΔOD = OD_{maltene} − OD_{oil}). We chose to use OD rather than the absolute amplitude because the measurements were done at different times and the reference THz signal amplitude cannot be always the same. The linear trend was improved by combining both measurements with linearity of $R^2 = 0.98$ in the peak time versus the asphaltene content plot. As these oil samples were taken from different geographic locations around the world, the results shown in Figure 3 indicate that this method has the potential to be used in measuring the asphaltene content of any oil sample in a similar procedure to what has been reported before using optical spectroscopy.\textsuperscript{15} In that way, the measurement can be done within approximately 2–3 h and consume significantly less solvent and oil samples with an accuracy that could outperform the traditional gravimetric methods.

On the basis of the sensitivity of this result which is 0.079 (ps/10 mm) per 1% asphaltene content and the standard deviation of the measurement which is 0.012 ps, the asphaltene limit of detection (LOD) was calculated to be 0.46%. This was done by taking the LOD to be 3 times the standard deviation. The crude oil absorption in the THz frequencies is very moderate, and so this LOD can be simply boosted by just increasing the oil sample thickness. For example, by increasing the sample thickness from 10 to 50 mm, which will still result in a detectable signal, the LOD will decrease to 0.09%.

The principle behind the negative peak shift that we see after removing the asphaltene can be explained by the Lorentz–Lorenz relation (eq 1).\textsuperscript{17} This equation describes the relation between the refractive indices of a multicomponent mixture. Asphaltene has the highest refractive index in the crude oil and when the asphaltene is removed, the effective refractive index of the oil is reduced. As a result, the THz pulse travels faster.
through oil without asphaltene compared with oil with asphaltene, resulting in what we see as a negative shift in the peak time between the neat oil and the maltene.

The change in amplitude due to asphaltene removal can be explained by the Beer–Lambert law. The Beer–Lambert law is often used in THz spectroscopy as a tool to determine the absorbance and concentration of a particular component in a multicomponent mixture.

According to Beer’s law, if a solution consists of two components, the total absorption can be regarded as a linear combination of both components by

$$A = e_1 c_1 l + e_2 c_2 l,$$

where $e$ is the molar absorptivity and $c$ is the concentration. Confirming the validity of Beer’s law is often done by measuring the absorption of one component in a solution at different concentrations. This was challenging to verify here because asphaltene is only soluble in toluene at low concentration (<1%) before they aggregate and deposit and because the absorption of toluene is higher than that of asphaltene. It was not possible to observe a clear change in the absorption as result of varying the asphaltene concentration. However, the result shown in Figure 3 suggests that this relation is valid to a high degree.

$$\frac{n_{o1}^2 - 1}{n_{o1}^2 + 2} = \frac{\phi_{asp}}{\phi_{asp}} \frac{n_{asp}^2 - 1}{n_{asp}^2 + 2} \frac{n_{mal}^2 - 1}{n_{mal}^2 + 2} \frac{n_{mal}^2 + 2}{\phi_{mal}}$$  (1)

The refractive index of the oil and maltene can be calculated directly from the measured time-domain signal, and subsequently, the asphaltene refractive index can be estimated from these two spectra. The refractive index is calculated by $1 + \frac{\phi c}{\omega d}$, where $\phi$ is the fast Fourier transform (FFT) phase difference between the sample and the empty sample holder signals, $\omega$ is the angular frequency, $c$ is the speed of light in vacuum, and $d$ is the sample thickness. As shown in eq 1, the oil can be considered as a mixture of two components, maltene and asphaltene, where $n_{asp}$, $n_{mal}$, and $n_{asp}$ are the refractive index spectra of the neat oil, maltene, and asphaltene, respectively. $\phi_{asp}$ is the asphaltene fraction of the crude oil, which can be calculated by measuring the asphaltene mass and considering its density to be 1.2 g/mL, and $\phi_{mal}$ is the maltene fraction of the crude oil ($\phi_{mal} = 1 - \phi_{asp}$). The $n_{asp}$ can be extracted from this equation because all other variables can be either measured or calculated.

Figure 4 shows the calculated asphaltene refractive index spectra from oil samples 1, 2, and 3 in the range of (0.3−2.2) THz. The asphaltene from oil 1 and oil 3 have exponential decay-like spectra, whereas the asphaltene from oil 2 did not exhibit the same feature but instead it shows an almost constant refractive index in the reported range. All asphaltene refractive
index spectra seem to stabilize within the values of \((1.75−1.85)\) in the spectral range of \((1.5−2.2)\) THz. The refractive index relates to the polarity of the material and because the asphaltene polarity is linked to the heteroatom and metal content,\(^{41}\) we may infer that the variation in the refractive index is due to the variation of the heteroatoms of the asphaltene. In fact, some polar fluids such as ethanol and water have this feature at lower THz frequencies,\(^{43}\) which gives us another reason to think that this is due to asphaltene polarity. The accuracy of the reported refractive index is subject to error because of variations in the asphaltene density or because of variations of the asphaltene measurement. When the asphaltene content is low, its measurement has higher uncertainty; therefore, we limited the calculation to oils with high asphaltene content. This measurement gives us an idea of the refractive index of asphaltene where no other data are available in the literature.

The absorption spectra of the asphaltene can be estimated by calculating both maltene and oil absorption coefficients and taking the difference between them. After applying the FFT to the time-domain signals, the THz frequency spectra of the reference, neat oil, and the maltene are calculated. The absorption coefficient \(\alpha\) can be calculated by \(-\left(2/d\right)\ln\left(T(n + 1)^{2}/4\pi\right)\), where \(T\) is the FFT magnitude ratio between sample and reference signals, \(d\) is the sample thickness, and \(n\) is the refractive index.\(^{27}\)

Figure 5a shows the THz frequency spectra of the reference, neat oil, and the maltene of oil 1, whereas Figure 5b shows the corresponding calculated absorption coefficient of the neat oil, the maltene, and the difference which we attributed to asphaltene. The reference signal has a bandwidth of roughly 4 THz, but because the oil has stronger absorption in the high frequencies, the effective spectra became less than 2.5 THz (the signal approaches the noise floor beyond this frequency). Similarly, the asphaltene absorption spectra of the other oils have been calculated and plotted together in Figure 6. The figure shows that asphaltene has mild absorption. Only the asphaltene from oil 2, which has the highest asphaltene content, has clear absorption across all of the frequency range. Because the asphaltenes are the most polar components in the crude oil with the dipole moment larger than water,\(^{31}\) the asphaltene absorption was less than what we anticipated, especially in the asphaltene-rich oil samples.

The asphaltene absorption of some of the samples was unnoticeable (close to zero), and in some frequencies, it was negative. Negative absorption is not real in this case, as it is not consistent with the time-domain signal shown in Figures 2 and 3. The time-domain signal measurement is more rigorous, as it is directly measured from the detector without processing or assumptions, whereas the frequency domain spectra is a result of the Fourier transform algorithm, which is more susceptible to variation when not all the conditions to apply the algorithm are met. The purpose of calculating the asphaltene absorption spectra was not to use it as a quantitative measure but mainly to detect any absorption peaks because molecules of this size usually vibrate at the THz range. These peaks can only be determined by applying FFT to the time signal, but the result did not show any clear peaks. It is also worth mentioning that

![Figure 4. Calculated refractive index of asphaltene from oil 1, oil 2, and oil 3.](image)

![Figure 5. (a) THz frequency domain spectrum for the reference, oil, and maltene of oil 1. (b) Absorption coefficient of oil, maltene, and asphaltene of oil 1.](image)

![Figure 6. Comparison of the asphaltene absorption from 6 oils.](image)
we are not considering the complex Fresnel transmission coefficients at the sample holder oil interface and the oil sample holder interface. The change in the refractive index because of the asphaltenes’ removal will change this coefficient slightly and consequently will add some uncertainty to the results.

In addition, Figure 6 shows that there are some narrow absorption lines in the asphaltene absorption spectra, especially in the oils that have larger asphaltene content. Those lines match well with the water absorption lines. The vertical lines in Figure 6, which represent the typical water absorption lines, overlap with all of the clear absorption lines in the asphaltene spectra. However, not all expected water lines are seen in the spectra. The water absorption lines do not appear when probing water molecules as bulk water but instead they appear when measuring water in the vapor phase or when adsorbed on a material at very low quantity to the level that it does not form bulk water. There have been many reports in the literature that confirm the interactions between the asphaltenes and water, though these interaction mechanisms are not well understood. What is known is that the asphaltenes absorb moisture, and the asphaltene content have a linear relation with the water content of the crude oil.

On the basis of the measurement data and the literature, it may be inferred that these lines come from scattered water molecules in the oil that was adsorbed on the asphaltenes and that the water was removed during the asphaltene separation process. In addition, the fact that there is a reasonably linear trend, as shown in Figure 7, between the summation of all absorption peaks of the water lines and the asphaltene content make us lean more toward this conclusion.

The high OD of crude oils and asphaltenes made it challenging to measure their refractive index and absorption in the visible and NIR range without excessive dilution. On the other hand, the mild absorption of the crude oil and asphaltene in the THz frequencies made it possible to measure thick samples in the transmission configuration. The result of this work shows the potential of the THz-TDS to be used in the future in various application such as measuring asphaltene content, asphaltene precipitation onset, and asphaltene yield curve.

**CONCLUSION**

In this work, THz-TDS was used to study asphaltenes in different crude oils. The difference in the time delay and the peak amplitude between the neat crude oil and the maltene correlated linearly with the asphaltene content. Variation was found in the refractive index spectra of the asphaltene from different oils in the low THz frequencies and comparable spectra in the higher frequencies. The absorption coefficient spectra show a mild absorption because of asphaltene with no clear feature except for some narrow absorption lines that we attributed to water molecules adsorbed on the asphaltene. The water molecules may provide a more specific (albeit proxy) spectral feature for asphaltene in oil but do not show as good linearity as the refractive index and absorption variations.

**MATERIALS AND METHODS**

**Materials.** Seven crude oil samples, produced from various oil fields around the world, were purchased from ONTA, Inc. Toronto, Canada, with (14.7-37.2) American Petroleum Institute (API) span and (0.34-8.63)% asphaltene content. Table 1 summarizes the properties of these crude oils.

| source | density (g/mL) | API | asphaltene (%) |
|--------|---------------|-----|----------------|
| oil 1  | 0.898         | 25.9| 6.82           |
| oil 2  | 0.968         | 14.7| 8.63           |
| oil 3  | 0.877         | 29.7| 2.58           |
| oil 4  | 0.856         | 33.7| 1.09           |
| oil 5  | 0.838         | 37.2| 1.43           |
| oil 6  | 0.845         | 35.9| 0.34           |
| oil 7  | 0.939         | 19.1| 0.49           |

Solvents used in this work, purchased from Sigma-Aldrich, Oakville, Canada, are HPLC-grade toluene (CAS 108-88-3) and HPLC-grade n-heptane (CAS 142-82-5).

**THz-TDS Measurements.** Figure 8a shows the THz-TDS setup (TeTechs, Inc. Waterloo, Canada) that was used in this work. The measurement was done in transmission configuration in free space spectroscopy. A 1550 nm 80 femtosecond laser (Calmar) was used as pump and probe beams to generate and detect the THz signal. To get a higher THz bandwidth, we have used a photoconductive antenna (PCA) reported previously. The length of the probe beam path was varied with the delay-line to change the spectral position of the (THz-TDS) waveform. To get higher resolution frequency spectra, the scanning length was set to 30 mm which corresponds to 200 ps. The transmitter PCA was biased with 80 V and the pump beam was chopped with 10 kHz. The signal from the receiver PCA was sent to a lock-in amplifier and then
to a computer for processing. We used a sample holder made of Teflon (poly(tetrafluoroethylene)), as this material has a little absorption in the THz range. The dimensions of the holder are 10 × 20 mm and hold a sample of approximately 2.5 mL. Figure 8b shows the THz time-domain waveform under ambient condition and after it has been purged with nitrogen. The whole setup was enclosed in a glass box and purged with nitrogen until the relative humidity was less than 3%. Under these conditions, no absorption lines were observed in the frequency domain spectrum as shown in Figure 8c.

**Extractions and Measurement of the Asphaltene Content.** The asphaltene content was measured following the procedure reported in ref 10, which can be described in three steps. First, 10 g of the oil was mixed with n-heptane at a volume ratio of 1:40 and left for two days in the dark to reach equilibrium. The mixture was filtered using 0.22 μm membrane filter paper a few consecutive times. The filter cake was washed with hot n-heptane until the effluent was almost colorless. Second, the recovered asphaltenes were dissolved again in toluene at approximately 1 g/100 mL concentration and the mixture was filtered. The reason for this step is to remove any nonasphaltene solids, such as sand, and dissolve the maltenes molecules adsorbed on asphaltene. Finally, the asphaltene in the (asphaltene/toluene) mixture needs to be precipitated and measured again. This is done by mixing this mixture with heptane at 1:40 ratio. The size of the (asphaltene/toluene) mixture is large and would need a huge amount of heptane to precipitate the asphaltene; therefore, the size of the (asphaltene/toluene) mixture was reduced to about 10 mL by evaporating the toluene. Then mixture was mixed with heptane at 1:40 ratio and filtered. The recovered asphaltene was left to dry and then it was weighed. This method is time-consuming and needs up to 1 L of solvents for a single oil sample. The measured percentages of asphaltene in the oils used in this work are listed in Table 1.

To get the maltenie fluid for each oil (oil w/o asphaltene), the filtered mixture from the first step was vacuum-evaporated to remove the heptane until there was no notable change in the weight with continuing evaporation.

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