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Characterization of some crude oil samples from Niger delta area of Nigeria using infrared absorption spectrometric technique

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ABSTRACT

Crude oil obtained from different locations in the Niger Delta area of Nigeria was analyzed by Infrared Absorption Spectrometric technique using Nicolet IS5 Fourier Transform spectrometer to identify the functional groups and compounds in the samples. Results obtained revealed that the amount of surface active components in the crude was in following trend, sample E > sample A > sample C > sample D > sample B, while the level of biodegradability follows the trend; sample E > sample B > sample A > sample C > sample D. Results show that Sample E has the highest amount of surface active components as well as the highest level of biodegradability. Sample B has the least amount of surface active components, while sample D has the least level of biodegradability. The presence of functional groups such as amines, sulfates, isocyanates, hydroxyl, halo compounds, thiols and nitro compounds in the crude increases the surface active properties of the crude due to their polarity and hydrophilicity, which influences the interfacial tension of the crude and the oil recovery efficiency. The level of crude biodegradability is dependent on the amount of aliphatic saturates in the crude, the concentration of acidic components as well as sulphur and nitrogen compounds in the crude. Infrared spectroscopy identifies the functional group in crude samples, this is necessary in knowing the amount of the surface active components and the level of biodegradability of crude oil.

Capsule Summary: Characterization of crude oil using infrared absorption spectrometric technique is very essential in determining the level of biodegradability, recovery efficiency as well as product quality after exploration.

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INTRODUCTION

Infrared (IR) spectroscopy has to do with the relationship between infrared radiation and matter. Infrared radiation is an electromagnetic radiation with longer wavelengths than those of visible lights (Demirdöven et al., 2004). When a compound is placed along the path of an infrared radiation, some of the light frequencies are absorbed while others are transmitted as indicated by the detector placed on the other side of the compound. The infrared spectrum is a plot that shows how the percentage of light transmitted through the compound varies with the frequency of the infrared measured in cm^{-1} and also wave number (Kohli et al., 2006). The energy of the frequency of an infrared radiation is transferred to a compound once radiation is absorbed by the compound and this leads to bond vibration. There are different types of bond vibrations depending on the frequency of the infrared radiation and the type of bond involved (Akaegbobi, 1999). The intensity of the peaks or bands eluting from the bond vibrations could either be strong, weak or medium depending on the functional group present in the compound. The shape of the peak as well as the wavelength at which the peaks elute are also critical in functional group identification, the peaks could be broad or sharp (Villar et al., 2012). The infrared portion of the electromagnetic spectrum is divided into three regions namely the near, mid and far- infrared, based on their relationship to the visible spectrum. The mid infrared spectroscopy which corresponds to 4000 – 400 cm^{-1} is used to study the fundamental vibrations which gives essential information about the sample composition in terms of chemical groups present and its purity (Hargate, 2006). The basic function of infrared spectroscopy is in functional group identification, it therefore does not have the ability to determine the whole structure of a molecule neither does it give the percentage composition of each of the components in the molecule (Demirdöven et al., 2004).

Infrared (IR) spectroscopy is one of the important techniques employed in crude oil assay. Crude oil assay refers to the chemical evaluation of crude oil feed stocks to determine their molecular and chemical characteristics. The results of crude oil assay testing gives an elaborate detailed hydrocarbon analysis data for refiners, oil marketers and producers (Shcherbakov et al., 2012). Data obtained by crude assay assist refineries determine if a crude oil feedstock is compatible for a particular petroleum refinery or if the crude oil could cause yield, quality, production, environmental and other problems (Villar et al., 2012).

Though Infrared spectroscopy is not a comprehensive crude assay however comparing the corresponding spectra of the samples obtained from the different locations will reflect the molecular composition of the samples which can be used in further studies to trace the source and impact of spilled hydrocarbons as well as monitoring their fate on the environment by pattern recognition technique (Christy et al., 1989).

The aim of this study is to characterize crude oil samples obtained from different locations in the Niger Delta area of Nigeria using infrared spectroscopy. The Niger Delta area of Nigeria is the hub of oil and gas exploration and production in Nigeria. The functional groups present in crude oil which are identified from the infrared spectrum of the crude give vital information about the crude. Results obtained from infrared absorption spectroscopy are useful in determining crude oil parameters such as API (American Petroleum Institute) gravity, density, interfacial tension, wettability, emulsion stability, biodegradability etc.

MATERIAL AND METHODS

Description of study area

The sampling sites were at four locations in the Niger Delta area of Nigeria namely Usan, Ogbele, Abo and kokori oil fields. Condensate samples were obtained at Ogbele gas plant. Usan field is located approximately 62 miles (100 kilometers) off the coast of the Niger Delta in water depths of about 2,400 feet (750 meters). The project utilizes a floating production, storage and offloading (FPSO) vessel with a storage capacity of two million barrels of oil and it produces a maximum of 180,000 barrels of crude oil daily. The FPSO measures 320m in length, 61m in width, 32m in depth, and weighs 114,000 metric tons. Associated gas is being re-injected in the reservoir. Usan crude is a medium gravity, low sulfur crude oil produced in deep water off the coast of Nigeria. Figure 1 shows a map indicating the location of Usan Field. The wells represent the sources of the sampling points (Arinze et al., 2016). The Ogbele field is located in the onshore eastern delta 45 km northwest of Port Harcourt. Ogbele is within Nigeria and is south of Rumuachara, Omoviri and Odaga. Ogbele has an elevation of 8 metres, at latitude 4° 55' 11.3" (4.9198°), north Longitude of 6° 40' 30.2" (6.6751°) east. Figure 2 shows a map indicating the location of Ogbele field. Ogbele Field is a subsidiary of Chevron's oil mining lease (OML) 54 and in 1996 it was the first marginal field awarded to be negotiated with an indigenous company that is the Niger Delta Petroleum Resources (NDPR). The operators (NDPR) commenced oil production in October 2005 with an estimated production of five million barrels which was later upgraded to twenty million barrels. The field facilities include a 20 000 b/d flow station and a 100 mmcfd gas plant. In 2012 NDPR became the first third party supplier to Nigeria Liquified Natural Gas (LNG) exporting 15 to 30 mmcfd. A 1 000 b/d/ diesel topping plant is also located on site, refining takes place based on the dictates of market forces. Condensates are processed at the Ogbele gas plant (NDPR, 2011).

Abo field was discovered in 1997, it is a deepwater oil field located at oil mining lease (OML) 125, some 55 kms off the Nigerian coast. The field lies in water depths of around 450 to 750 metres and is operated by Nigerian Agip Exploration (NAE), a subsidiary of Italy's energy company (Eni). It was the first deepwater field developed in Nigeria. Eni holds 85% in the field and the remaining 15% is owned by Oando, which acquired its working interest in 2008. The storage capacity of Abo FPSO is 900,000 barrels of oil with an oil treatment capacity of up to 45,000 bopd, a water injection capacity of 30,000 bwpd and a gas injection capacity of 35 MMSCFD. Figure 3 shows a map indicating the location of Abo field (Alaibe, 2008).

Kokori is one of the six suburban 'states' of the Agbon 'kingdom' in Ethiope East local government area of Delta State, Nigeria. Its land area is 196 square kilometers. The estimate terrain elevation above sea level is 15 metres with a latitude of 5°37'52.39" and a longitude:of 6°2'6.25". The entire Kokori land is flat and situated in the evergreen tropical forest zone which is dominated by the oil palm tree. Kokori oil field is one of the nine flow stations of OML 30. On 4th November 2012, Shoreline Natural Resources acquired 45% Participating Interest of

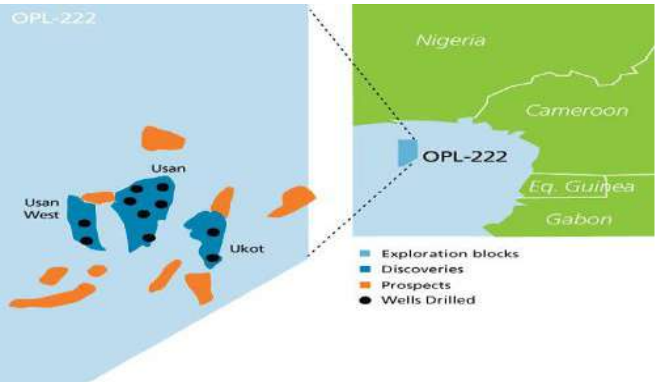

Fig. 1: Map Indicating the Location of Usan Field

Fig. 2: Map Indicating the Location of Ogbele Field

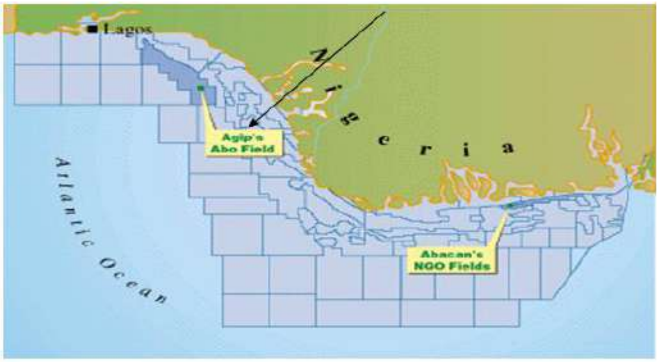

Fig. 3: Map Indicating the Location of Abo Field

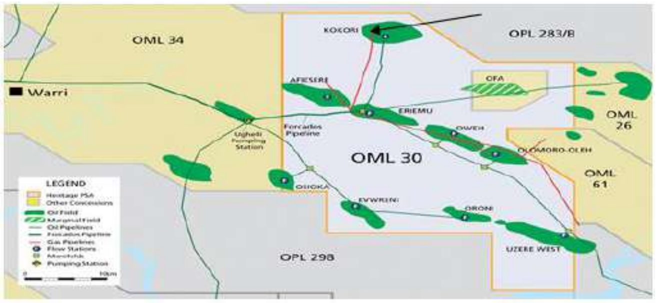

Fig. 4: Map Indicating the Location of Kokori Field

SPDC in Oil Mining Lease (OML) 30. Nigerian Petroleum Development Company (NPDC) a subsidiary of Nigerian National Petroleum Cooperation holds the remaining 55%. Figure 4 shows a map indicating the location of Kokori oil field (Chokor, 2008).

Sample collection, preparation and analyses

Samples were obtained from each of the fields with glass sampling bottles previously rinsed with xylene and dried. Samples were sandwiched between two plates of a sodium chloride salt. The plates are transparent to the infrared light as such do not introduce any lines onto the spectra. Spectra of samples were obtained using a Nicolet IS5 Fourier Transform Spectrometer. Samples were identified as A, B, C, D and E according to their locations.

A beam of infrared light was passed through an interferometer which splits into two separate beams. Each of the beams passes through the sample and reference respectively. The distribution of the infrared light that passes through the interferometer was altered using a moving mirror placed inside the apparatus. The signal directly recorded, called an "interferogram", represents light output as a function of mirror position. The beams pass through a splitter and then reflected back towards a detector, the splitter alternates the beams that enter the detector. A data-processing technique called Fourier transform turns this raw data into the desired result which is the sample's spectrum (Demirdöven et al., 2004).

RESULTS AND DISCUSSION

Prominent functional groups and compounds in Sample A were listed in Table 1. The major functional groups as identified in the infrared (IR) spectrum as shown in figure 5 include a medium vibration N-H stretching of primary amine at a wavelength of 3500-3400 cm^{-1} and a strong vibration S=O stretching of sulfate at a wavelength of 1415 – 1380 cm-1 indicating the presence of 2,5-diamino toluene sulfate. A medium C-H stretching of alkane at a wavelength of 3000 – 2840 cm^{-1} confirms the presence of dodecane, tetradecane and octadecane while a weak C-H bending of aromatics at a wavelength of 2000 – 1650 cm^{-1} and a strong C-Br stretching of halo compound at a wavelength of 690 – 515 cm^{-1} confirms the presence of bromo triphenyl methane. The presence of 1-decane thiol in Sample A was confirmed by a weak S-H stretching of thiol at a wavelength of 2600 – 2550 cm-1 while the presence of 3- methyl octane was confirmed by a medium C-H bending of methyl group at a wavelength of 1465 – 1450 cm^{-1}.

Prominent functional groups and compounds in Sample B were listed in Table 2. The major functional groups as identified in the infrared (IR) spectrum as shown in figure 6 include a weak C-H bending of aromatics at a wavelength of 2000–1650 cm^{-1} and a strong C-Br stretching of halo compound at a wavelength of 690–515 cm^{-1} indicating the presence of bromo triphenyl methane, while a medium C-H

Table 1: Functional groups/compounds obtained from IR spectrum of sample A

Peak	Wavelength (cm^{1})	Mode of Vibration	Functional Group	Compounds
P1	3500-3400	N-H stretching	primary amine	2,5-diaminotoluene sulfate,
P2	3000 - 2840	C-H stretching	alkane	dodecane, tetradecane octadecane
P3	2600-2550	S-H stretching	thiol	1-decane thiol
P4	2000-1650	C-H bending	aromatic compound	bromo triphenyl methane
P5	1465-1450	C-H bending	methyl group	3-methyloctane
P2	1415-1380	S=O stretching	sulfate	2,5-diaminotoluene sulfate
P7	690-515	C-Br stretching	halo compound	bromo triphenyl methane

Table 2: Functional groups/compounds obtained from IR spectrum of sample B

Peak	Wavelength (cm^{1})	Mode of Vibration	Functional Group	Compounds
P1	3000-2840	C-H stretching	alkane	octadecane, docosane
P2	2000-1650	C-H bending	aromatic compound	bromo triphenyl methane
P3	1465-1450	C-H bending	methyl group	2.6-dimethyloctane, 4-methyloctane
P4	915-905	C=C bending	monosustituted alkene	1-tetradecene, 1-octadecene
P5	690-515	C-Br stretching	halocompound	bromotriphenylmethane
P6	600-500	C-I stretching	halocompound	1-Iodoheptane

Table 3: Functional groups/compounds obtained from IR spectrum of sample C

Peak	Wavelength (cm^{1})	Mode of Vibration	Functional Group	Compounds
P1	3500-3400	N-H stretching	primary amine	2,5-diaminotoluene sulfate
P2	2600-2550	S-H stretching	thiol	1-decane thiol
P3	3000 - 2840	C-H stretching	alkane	dodecane, octadecane, docosane
P4	1465-1450	C-H bending	methyl group	4-methyl octane, 3-methyl octane
P5	1415-1380	S=O stretching	sulfate	2,5-diaminotolune sulfate

Table 4: Functional groups/compounds obtained from IR spectrum of sample D

Peak	Wavelength (cm^{1})	Mode of Vibration	Functional Group	Compounds
P1	3500-3400	N-H stretching	primary amine	2,5-diaminotoluene sulfate, dodecylamine
P2	3000 - 2840	C-H stretching	alkane	dodecane, tetradecane, octadecane, docosane
P3	2600-2550	S-H stretching	thiol	1-decane thiol
P4	1465-1450	C-H bending	methyl group	4-methyl octane, 3-methyl octane
P5	1415-1380	S=O	sulfate	2,5-diaminotoluene sulfate

stretching of alkane at a wavelength of 3000–2840 cm^{-1} confirms the presence of octadecane and docosane and a medium C-H bending of methyl group at a wavelength of 1465–1450 cm^{-1} confirms the presence of 2,6-dimethyl octane and 4-methyl octane while a strong C=C of mono substituted alkene at a wavelength of 915-905 cm^{-1} confirms the presence of 1-tetradecene and 1-octadecene. The presence of 1-iodoheptane was affirmed by a strong C-I stretching of halo group compound at a wavelength of 600–500 cm^{-1}.

Prominent functional groups and compounds in Sample C were listed in Table 3. The major functional groups as identified in the infrared (IR) spectrum as shown in figure 7 include a medium vibration N-H stretching of primary amine at a wavelength of 3500-3400 cm^{-1} and a strong vibration S=O stretching of sulfate at a wavelength of 1415–1380 cm-1 indicating the presence of 2,5-diamino toluene sulfate. A medium C-H stretching of alkane at a wavelength of 3000–2840 cm^{-1} confirms the presence of dodecane, octadecane and docosane. The presence of 1-decane thiol in sample C was confirmed by a weak S-H stretching of thiol at a wavelength of 2600–2550 cm-1 while the presence of 3-methyl octane and 4– methyl octane were confirmed by a medium C-H bending of methyl group at a wavelength of 1465–1450 cm^{-1}.

Prominent functional groups and compounds in Sample D were listed in Table 4. The major functional groups as identified in the infrared (IR) spectrum as shown in figure 8 include a medium vibration N-H stretching of primary amine at a wavelength of 3500-3400 cm^{-1} and a strong vibration S=O stretching of sulfate at a wavelength of 1415–1380 cm-1 indicating the presence of 2,5-diamino toluene sulfate and dodecylamine. A medium C-H stretching of alkane at a wavelength of 3000–2840 cm^{-1} confirms the presence of dodecane, tetradecane, octadecane and docosane. The presence of 1-decane thiol in Sample D was confirmed by a weak S-H stretching of thiol at a wavelength of 2600–2550 cm-1 while the presence of 3- methyl octane and 4 – methyl octane were confirmed by a medium C-H bending of methyl group at a wavelength of 1465–1450 cm^{-1}.

Table 5 shows that more prominent peaks eluted from Sample E. The major functional groups as identified in the infrared (IR) spectrum as shown in figure 9 include a medium vibration N-H stretching of primary amine at a wavelength of 3500-3400 cm^{-1}, a strong C-H stretching of thiazole at a wavelength of 3110–3050 and a strong N-O stretching of nitro compound at a wavelength of 1550–1500 indicating the presence of 2-amino-5-nitrothiazole and 4-nitro aniline. A medium sharp O-H stretching of free alcohol at a wavelength of 3819–3451 cm^{-1} confirms the presence of stigmasterol while the presence of paraffin oil is confirmed by a medium C-H stretching at wavelength of 2924–2855 cm^{-1}. A strong broad N=C=O stretching of isocyanate at a wavelength of 2275–2250 cm^{-1} indicates the presence of dichloroisocyanuric acid sodium salt while a strong S=O stretching at a wavelength of 1350–1342 cm^{-1} and a medium C-H bending of gem dimethyl at a wavelength of 1385–1365 cm^{-1} confirms the presence of 2-(-4-pyridyl) ethanesulfonic acid.

Petroleum is a complex chemical compound with complicated mixtures of hydrocarbons (hydrogen and carbon) and small amounts of heteroatoms (oxygen, nitrogen, sulphur, halogens) and very small amounts of coordination series metals. Though the percentage composition of elements other than hydrogen and carbon is < 6.0%, studies have shown that no two petroleum samples are identical in their physical appearance and behavior (Gough and Rowland, 1990). To be able to anticipate the behavior of crude oil it is very important to know the chemical composition of the crude. The presence of polar components is the most important factor applied in understanding the behavior of crude oils (Farooq et al., 2013). Polar compounds are compounds with heteroatoms, they include functional groups such as carboxylic acid, hydroxyl, amines, thiols, isocyanates, nitro and halo compounds etc. Polar compounds are surface active components of crude oil; they are acidic in

Table 5: Functional groups/compounds obtained from IR spectrum of sample E

Peak	Wavelength (cm^{1})	Mode of Vibration	Functional Group	Compounds
P1	3819 - 3451	O-H stretching	free alcohol	stigmasterol
P2	3500-3400	N-H stretching	primary amine	2-amino-5-nitrothiazole, 4-nitro aniline
P3	3110-3050	C-H stretching	thiazole	2-amino-5-nitrothiazole
P4	2924 - 2855	C-H stretching	alkane	paraffin oil
P5	2275-2250	N=C=O stretching	isocyanate	dichloroisocyanuric acid sodium salt
P6	1550-1500	N-O stretching	nitro compound	2-amino-5-nitrothiazole, 4-nitro aniline
P7	1465-1450	C-H bending	methyl group	4-methyl heptane, 3 methyl heptane, isopropyl cyclohexane
P8	1350-1342	S=O stretching	suiphonic acid	2-(-4-pyridyl) ethanesulfonic acid
P9	1385-1365	C-H bending	gem dimethyl	2-(-4-pyridyl) ethanesulfonic acid

nature and play important role in emulsion stability, wet ability, interfacial tension and viscosity of the crude (Hoeiland et al., 2001). The force of adhesion between the crude oil and the reservoir rocks increases with increase in the polarity and hydrophilicity of the functional groups present in the crude and this is very important in oil recovery. The higher the surface active components of the crude the lower the recovery efficiency during enhanced oil recovery (EOR) operations (Farooq et al., 2013). Results show that all the crude samples analyzed indicated the presence of different polar components. Infrared (IR) spectrum of Sample E indicated the presence of more hetero atoms such as free alcohols, primary amines, isocyanates, sulphonic acids and nitro compounds while infrared (IR) spectrum of Sample B indicated the least number of heteroatoms, IR spectrum of Sample B indicated the presence of halo compounds in addition to saturated alkanes, monosubstituted alkenes and alkanes as well as aromatics.

The functional groups present in the crude are also an indication of the biodegradability of the crude. Biodegradation involves the aerobic or anaerobic consumption of organic matter by bacteria either as in an oil reservoir (Meredith et al., 2000). Bacteria have a preference for the aliphatic saturates, followed by branched saturates, cyclic saturates and finally aromatic hydrocarbons. The presence of aliphatic saturates in the crude is an indication that the crude is not heavily biodegraded as such the higher the amount of aliphatic saturates in the crude the lower the level of biodegradability (Larter et al., 2006). Comparing the results obtained from the analyses of the samples under study it can be deduced that Sample D is the least biodegraded crude with four aliphatic saturates namely dodecane, tetradecane, octadecane, docosane, followed by Sample C with three aliphatic saturates (dodecane, octadecane, docosane) and two branched saturates (4-methyl octane, 3-methyl octane). This is closely followed by Sample A with three aliphatic saturates (dodecane, octadecane, docosane) and one branched saturate (3-methyl octane). Results show that Sample E is the most biodegraded crude with no straight chain saturate.

Apart from preferentially reducing the amount of aliphatic saturates in crude oil, studies have also shown that biodegradability also increases the concentration of acidic components in crude oil, this explains why Sample E is the only sample with acidic compounds such as dichloroisocyanuric acid sodium salt and 2-(-4-pyridyl) ethanesulfonic acid (Barth et al., 2004). Nitrogen and sulphur are both problematic elements in crude as oxides of both elements are acidic. Sulphur compounds also increases the activities of sulphate reducing bacteria which reduces sulphate to hydrogen sulphide and sulphuric acids (Wenger, 2002). Results show that apart from Sample B all the other crude samples contain nitrogen and sulphur compounds.

There are other factors apart from biodegradability that controls the amount of acidic components present in petroleum products such as the type of sediment the petroleum stems from, how long it has been buried and most importantly how deep it was buried (Schobert, 2013).

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CONCLUSIONS

Functional group identification in crude samples using infrared absorption spectrometric technique is very essential in characterizing crude oil based on surface active properties and biodegradability. The presence of polar components in crude samples increases the interfacial tension of the crude and this is very important during oil recovery. The higher the surface active components (polar components) of the crude the lower the recovery efficiency during enhanced oil recovery (EOR) operations. With the preference of bacteria towards aliphatic saturates, it is obvious that the higher the amount of aliphatic saturates in the crude the lower the level of biodegradability. Sample E has the most polar components (hetero atoms) of all the crude analyzed as such has the most surface active components which increases its adhesion to reservoir rocks thereby reducing its recovery, It is also the most biodegraded as confirmed by the presence of acidic components, nitrogen and sulphur compounds as well as the absence of aliphatic saturates. Assessing if a crude oil is biodegraded has become an important factor for the oil industry due to reduced recovery efficiency, reduced product quality and increased problems under production and handling. Sample D is the least biodegraded crude with the highest concentration of aliphatic saturates while sample B has the least amount of surface active components (hetero atoms) which gives it the most advantage during oil recovery.

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