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X-ray Diffraction and Fourier Transform Analysis of Corrosive Components of Crude Oils Used by Kaduna Refinery for Corrosion Control

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ABSTRACT

Kaduna refinery is a critical segment of the Nigerian economy but is subject to costly corrosion problems. The refinery obtains crude oils from various sources and processes them under peculiar conditions into essential chemical products. This research was undertaken in order to gain insight into the component proportions of the refinery's employed crude oils that contribute to corrosion problems. The component proportions of a homogeneous admixture of the employed crudes at the refinery were analyzed using XRD and FTIR techniques. Obtained results and analysis indicate that crude oils employed at the refinery are generally significantly corrosive to most materials due to their appreciable total component contents of sulfur, carbon, idrialite, refikite, flagstaffite, and silicon dioxide up to 35%, which can be corrosive per se or can undergo reactions, especially at higher refining temperatures, to produce abundant quantities of corrosion agents that include acids, chlorides, water, carbon dioxide, hydrogen sulfide, etc.

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1. INTRODUCTION

Crude oil media can be more corrosive and cause much higher levels of corrosion in refineries than other industrial media because most known factors that cause various types of material corrosion to an extreme are abundantly present in crude oils' physicochemical composition and are augmented in corrosivity level by the various refineries' processing conditions. Every form of crude oil contains almost the same types of chemicals, but in various amounts that depend on the source of the crude and how it is processed, transported, and stored. Differently sourced, stored, processed, and transported crude oils can therefore be differently corrosive to specific materials [1], [2].

The Kaduna refinery is an important part of Nigeria's industry and economy. The refinery uses Nigerian and foreign crudes from various countries, such as Venezuela, Saudi Arabia, Kuwait, Basra, etc., and processes them under peculiar refining conditions to produce a variety of fuels and chemical industrial raw feedstocks for the general Nigerian economy. Corrosion is a critical problem the refinery has been contending with over the course of its existence since 1976 [3]. Corrosion has indirectly contributed to the refinery's maintenance costs, shutdowns, low production output, and product scarcity. These problems have significantly contributed to the untold hardship of Nigerians and huge economic losses in the country. It is challenging to combat corrosion problems in the refinery despite its excellent design against corrosion problems and the state-of-the-art corrosion management and control in its domain. The development of any fundamental strategy for controlling corrosion to the barest level at the refinery would, however, require information on the corrosivity of the crudes employed at the company [3].

Energy-dispersive spectroscopy, scanning electron microscopy, Fourier transform infrared (FTIR) spectroscopy, wavelength-dispersive spectroscopy, Auger electron spectroscopy, x-ray photoelectron spectroscopy, Raman spectroscopy, time-of-flight secondary ion mass spectrometry, chromatography, and atomic emission spectroscopy are commonly used methods in corrosion and corrosivity analyses [4]. FTIR spectroscopy is used to chemically characterize organic, polymeric, and inorganic materials. It is also used for identifying their contaminations by measuring the infrared absorption or emission spectra from them. FTIR spectroscopy works by scanning test materials and examining their chemical characteristics using infrared light. It is an established analytical method for quality control of industrially produced materials. It is frequently used as the initial step in the material examination process. A material's composition and the presence of any contamination in it are clearly shown by a change in the distinctive pattern of absorption bands in FTIR spectroscopy. The technique can be used to identify the chemical composition of samples of both macroscopic and microscopic nature, typically 10 to 50 microns in size. The technique is used to identify and characterize unknown materials such as films, solids, liquids, and powders; contamination on or in materials such as particles, fibers, powders, and liquids; additives after being extracted from a polymer matrix; and oxidation, decomposition, or uncured monomers when conducting failure analysis investigations [5], [6]. The merits of FITR spectroscopy include [5], [6], [7]:

i. The ability to identify chemical species as tiny as 10 to 20 microns.
ii. Inability to harm the sample or be harmed by the sample.
iii. The possibility of gathering a lot of analytical data from it because its mid-infrared spectrum's absorbance bands are evaluated simultaneously in usage.
iv. Possibility of finishing analysis with the method in as little as 24 to 48 hours.
v. Suitability for the analysis of many different organic and certain inorganic compounds.
vi. Extremely quick and accurate analyses like spectral subtraction are possible due to the lack of uncertainty regarding wavelength measurement.

However, FTIR spectroscopy has drawbacks such as [5], [6], [7]:

i. Exposing the sample to all of the mid-infrared frequencies simultaneously during the process results in developing noise in one region of the infrared source's radiation and spreading it throughout the spectrum.
ii. A possible challenge in conducting the analysis on highly sensitive samples or samples that must be investigated over a long period of time due to changes in atmospheric conditions.
iii. It’s a bulk analytical technique that works best for locating and identifying broad categories of compounds within a compound but is difficult for distinguishing minute amounts of substances that are dissolved in mixtures with other substances.

On the other hand, XRD analysis is one of the most flexible and popular analytical methods for describing the chemical compositions of various materials, including minerals and soils. The benefits of XRD analysis include quick and accurate material identification, simple sample preparation, and computer-aided chemical species identification using a large database of structural data [8], [9].

The aim of this paper is to present the quantitative values of corrosive components of the crude oils employed at the Kaduna refinery by XRD and FTIR as versatile complementary techniques. The objective of the research is to provide the fundamental information that predicts the total corrosivity level of the employed oils on structures such as piping, vessels, furnace tubes, equipment, etc. in the refinery for consideration in any comprehensive corrosion control strategy or approach at the refinery.

2. RESEARCH METHOD

2.1 Materials and Equipment

The materials used for the research were all crude oils that had ever been sourced and employed at the Kaduna refinery. These were the Nigerian, Venezuelan, Saudi Arabian, Kuwaiti, and Basran crudes. The crude oils were sourced through the refinery. The facilities used for the research were the made-in-Japan Shimadzu 1200 diffractometer and FTIR-8400S spectrometer.

2.2 Experimental Processes

A liter of each of the sourced Nigerian, Venezuelan, Saudi Arabian, Kuwaiti, and Basran crudes were admixed in a 10-liter plastic container. The container was then tightly closed and vigorously shaken manually for five minutes to obtain a homogeneous mixture of all the sourced crudes for the study.

The XRD of a sample of the homogeneous mixture of the crudes was taken at room temperature using the Shimadzu-corporation-made 1200 model diffractometer with counter monochromatic Cu-Ka radiation from a Cu tube of wavelength 0.1406 nm [8], [9]. The voltage and current settings were 40 kV and 30 mA, respectively. The crude oil was examined in a continuous mode across the angle range of the goniometer, which was 2-theta (2θ) = 0 to 60°. The scanning speed, sampling pitch, and preset time were 2θ = 7°/min, 0.02 degrees, and 0.17 seconds, respectively. With these, the diffractogram, reference peak intensities, reference high and low intensity peaks, the performed search, match, and accept information, and the analyzed chemical components in the crude oil sample were produced with the diffractometer's integral computer accessory. This was repeated three times, and the results that were consistent at least twice were taken as the XRD-analyzed chemical components of the representative crude oil.

An FTIR analysis of a sample of the homogeneous mixture of all the crudes was carried out using the Shimadzu-cooperation-made FTIR-8400S spectrometer in accordance with the facility's settings, workpiece positioning, and calibration requirements. The diffractometer was properly set and operated. It sent infrared radiation of about 10,000 to 100 cm-1wave numbers through the homogeneously admixed sample of the crude oils, with some of the radiation being absorbed and some passing through the sample. The absorbed radiation was set into rotational and/or vibrational energy by the sample molecules. The resulting energy signal at the detector manifested as a spectrum of wave numbers, representing a molecular fingerprint of the sample. Each molecule or chemical structure produced a unique spectral fingerprint. This was printed and used for the analysis and identification of the chemical components or compounds in the representative sample of the crude oils.

3. RESULTS AND DISCUSSIONS

3.1. Results

The X-ray diffraction analysis of a sample of the admixed crude oils employed at the Kaduna refinery is presented in Figures. 1, 2, and 3; and Tables 1, and 2. On the other hand, the FTIR wave spectrum of a sample of the admixed crude oils showing critical absorption peak values of wave numbers for identified chemical compounds in it is presented in Figure 4.

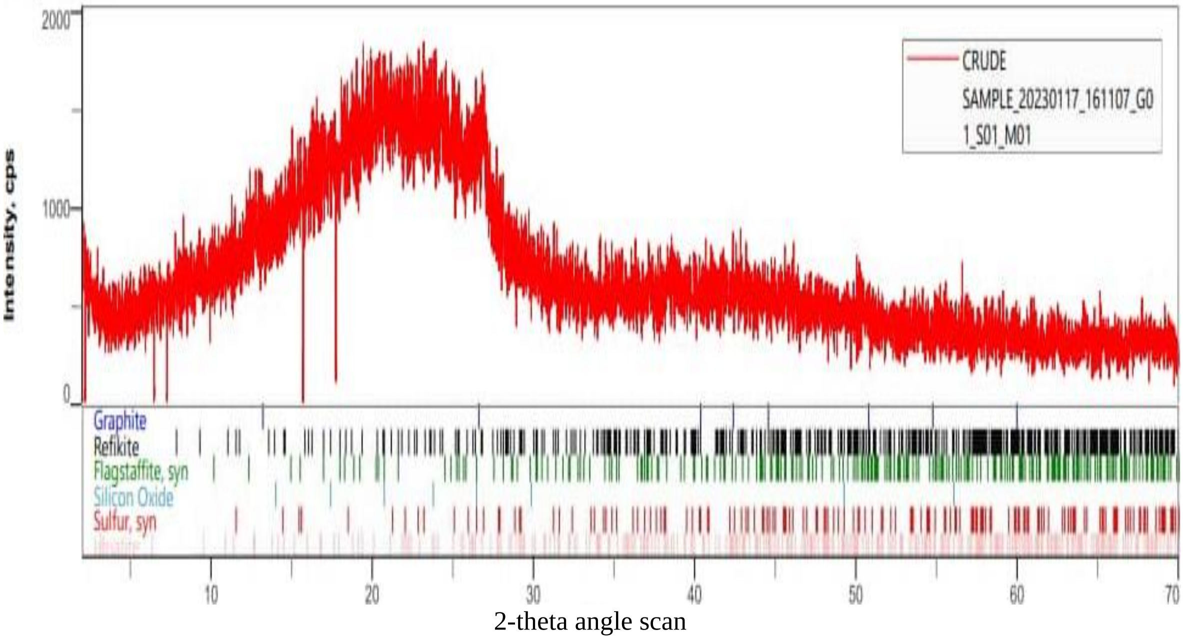


Figure 1: The obtained XRD diffractogram of the admixed crude oils at Kaduna refinery showing the principal components in the oils

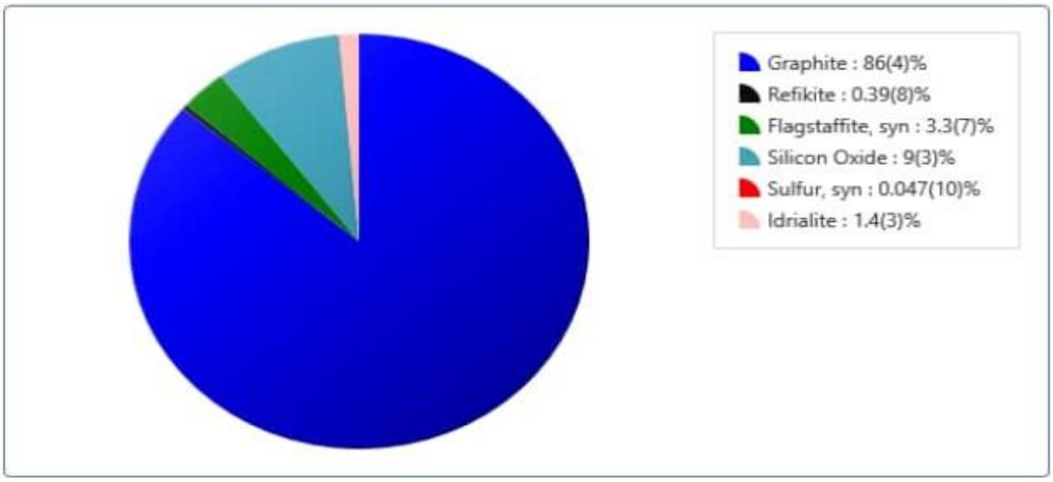
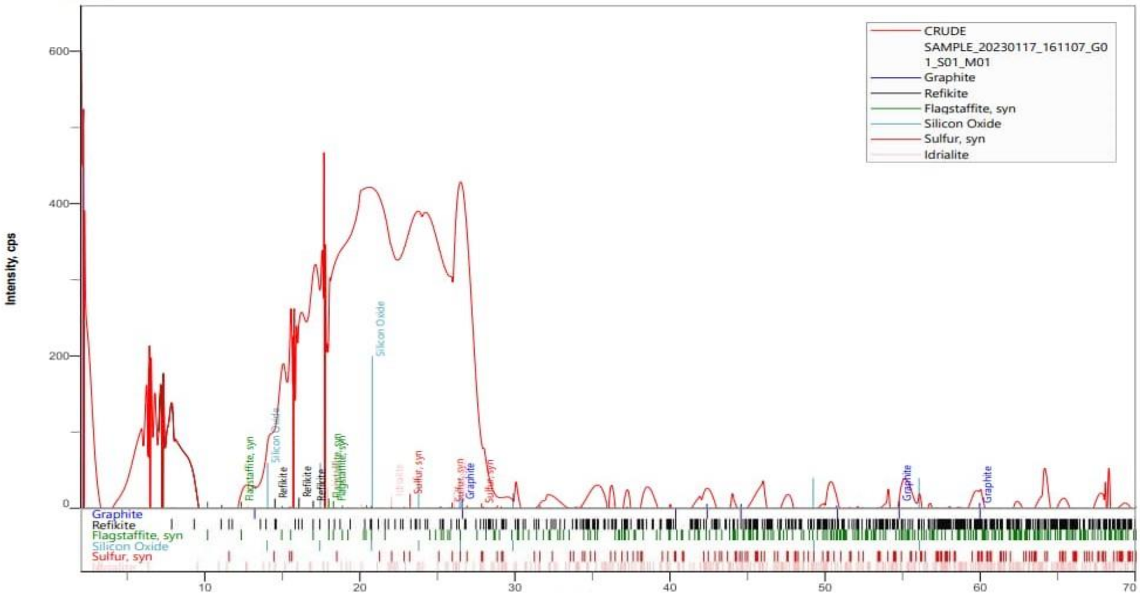


Figure 2: A pie chart of the component weight percentages of the crude oils

Table 1: Component weight quantity

Dataset/Weight Fraction	Value, Unit	Graphite	Refikite	Flagstaffite, syn	Silicon Oxide	Sulfur Syn.	Idrialite
Crude oil Sample	0	86(4)	0.39(8)	3.3(7)	9(3)	0.047(10)	1.4(3)



2-theta angle

Figure 3: The diffractogram patterns of the principal components found at the 2-theta angles in the representative crude oils' sample

Table 2: XRD specification of the main components found in the representative crude oils' sample

Phase name	Formula	Figure of merit	Phase reg. detail	Space Group	DB Card No.
Graphite	C	2.777	S/M	147:P-3	00-013-0148
Refikite	C20H34O2	2.682		18: P21212	00-028-2009
Flagstaffite Syn.	(C10H20O2) (H2O)	2.731		43: F2dd	01-072-5057
Silicon Oxide	SiO2	2.066		-	00-034-0717
Sulfur Syn.	S8	2.671		47: Pmmm	01-073-5065
Idrialite	C22H14	2.730		47: Pmmm	00-028-2006

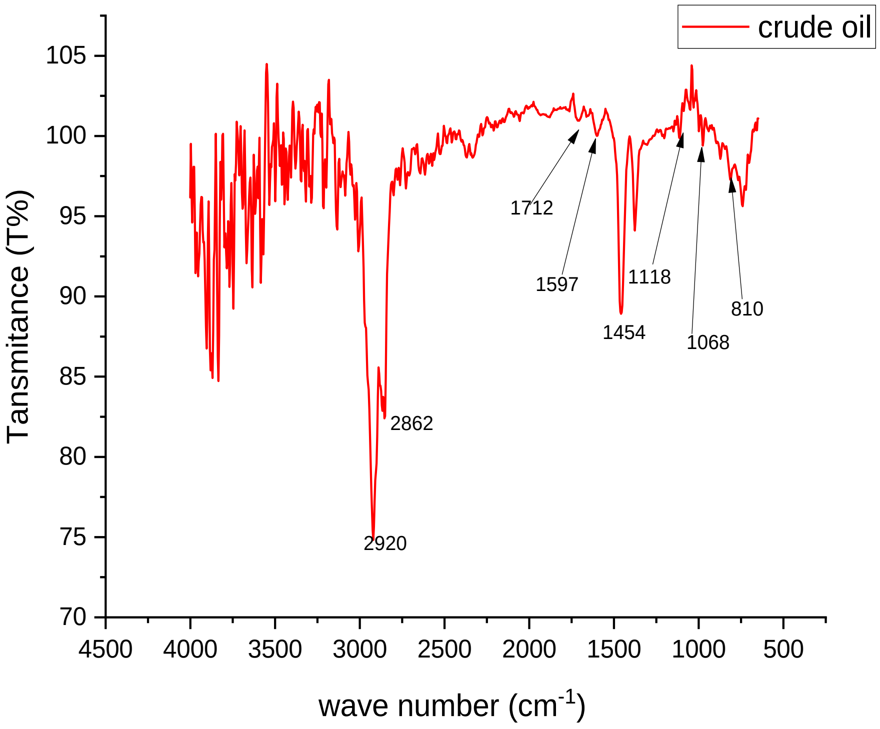


Figure 4: The FTIR wave spectrum of the representative crude oils' sample showing critical absorption peak values of wave numbers for identified chemical compounds in it

3.2. Discussions

It is evident from the XRD diffractogram shown in Figures 1 and 3 that a sample of the homogeneously admixed crude oils employed at the Kaduna refinery contains graphite, refikite, flagstaffite, silicon oxide, sulfur, and idrialite. However, the diffractogram in Figure 1 shows that the components are not uniformly but randomly distributed within the crude oil sample. This is observable from the variation in the diffractogram's peaks and the random distribution of the components within the 2θ-scan angle from 0 to 70°. As can be observed, the distribution of the components and their proportions in the admixed crude oils as depicted in Figures 1 and 3 are also shown in Table 1, Figure 2. From Table 1 and Figure 2, it is evident that the respective weight percentages of graphite, refikite, flagstaffite, silicon dioxide, sulfur, and idrialite components present in the admixed crude oil sample are 4, 8, 7, 3, 10, and 3%. The total percentage of the weight components is 35% and seen to be much higher or different from those of other refinery crudes analyzed by more or less similar methods by Fadlelmoula et al. [7]; Xia et al. [8]; Fahim et al. [14]; and Azam et al. [15] in FTIR analyses of their samples.

Graphite in crude oil comes from oil deposits that have been transformed with temperature, time, and pressure. Crystalline graphite has properties such as high temperature resistance, conductivity, lubrication, and heat conduction. It is an anti-rust primer made of carbon black and talc powder and has excellent corrosion resistance to chemicals. This element is not, per se, very corrosive, as it is mainly used in anti-corrosion coatings. However, graphite is more noble than all metals, so when it is in contact with any metal, it becomes the cathode, and the metal becomes the anode and starts to corrode. The metal's corrosion depends on the quantity of graphite in contact with the metal [15], [16]. Refikite is an orthorhombic-di-sphenoidal white mineral that contains carbon, hydrogen, and oxygen. It has an empirical formula of $C_{20}H_{34}O_2$. It is a derivative of abietic acid [16]. Flagstaffite is a yellowish-white mineral with the chemical formula $C_{10}H_{22}O_3$. The corrosivity levels of flagstaffite and refikite per se are not clearly documented, but as chemical compounds of the form $C_xH_yO_z$, flagstaffite and refikite can undergo combustion in the refinery to produce carbon dioxide and water according to the chemical equation (1). Water and carbon dioxide are great agents of corrosion, depending on their formed quantities, as determined by the quantities of flagstaffite and refikite present in the crude oils. As can be seen from Tables 1 and 2, the quantities of refikite and flagstaffite in the average crude oil employed at the Kaduna refinery are appreciable, with a total value of 15% and respective values of 8 and 7%.

$$C_xH_yO_z + \left[x + \frac{y}{4} - \frac{z}{2}\right] O_2 \rightarrow xCO_2 + \left(\frac{y}{2}\right) H_2O \tag{1}$$

Idrialite is a rare polycyclic aromatic hydrocarbon mineral that has a chemical formula $C_{22}H_{14}$. Like any other hydrocarbon of the forms C_xH_y or $C_xH_yO_z$, idrialite can also undergo combustion at high refining temperature in accordance to equation 2 to form carbon dioxide and water. The reaction products can cause appreciable corrosion of refinery structural components depending on their formed quantities. The level of carbon dioxide formed and attendant corrosion is proportional to the average 3%-weight fraction of idrialite present in the crude oils.

$$C_xH_y + N(O_2) \leftrightarrow x(CO_2) + \frac{y}{2}(H_2O) \tag{2}$$

In the presence of water or any moisture, silicon dioxide component of the crude oil can cause scaling and fouling corrosion of surfaces of heat exchangers, boilers, and other process equipment [17]. Sulfur is a non-metallic chemical element that belongs to the oxygen group. Sulfur occurs in crude petroleum in different chemical compounds such as sulfides, hydrogen sulfide (H_2S), polysulfides, thiophenes, mercaptans, and elemental sulfur [18], [19]. The sulfur concentration of crude oils is quite variable. The concentration depends on the oil's geographic origin, and is usually in the average range of 0.2 to 2.5% by weight but can be up to 14% by weight. Crude oils with less than one percent by weight of sulfur content are generally known as "sweet crudes". These crude oils are of good quality in contrast to the so-called "sour crudes" with more than 0.5 percent by weight of sulfur. Sour crudes are known to be of lower quality than sweet crudes. Sour crudes are also the principal culprit behind sulfidic corrosion in refineries [20], [21], [22]. Moreover, sulfur is very corrosive as it is one of the main elements that enhance the corrosivity of a given crude oil. The sulfur in crude oil reacts with water at temperatures greater than 80°C and lead to acidified solution that can be very corrosive to steel and other materials. The equation of the reaction of S_8 with water to produce corrosive acid and hydrogen sulfide which corrode ferrous materials at about 230°C is given by the reactions in equations 3 and 4 [23]

$$S_8(s) + 8H_2O(l) \rightarrow 6H_2S(aq) + 2H_2SO_4(aq) \tag{3}$$

$$8Fe + S_8 \rightarrow 8FeS \tag{4}$$

Elemental sulfur can per se cause localized corrosion of mild steel when they are in direct contact [13], [24].

From the FTIR spectrum of the prepared crude oil sample shown in Figure 4, a sharp absorption peak occurs at the wave number change of 2920 to 2862 cm^{-1}. This peak can be attributed to the stretching vibration of the hydroxyl group in carboxylic acid. An absorption peak also occurs at the 1712 cm^{-1} wave number. This absorption peak can be attributed to the stretching vibration of the C=O bond in carboxylic acid. The absorption peak at 1597 cm-1 wave number corresponds to the bending vibration of hydroxyl in carboxylic acid. The presence of these acidic peaks suggests the presence of petroleum acids, which are likely naphthenic acids found in the crude oil. These components' results strongly correlate with similar observations made by He et al. [25] on similar components in their FTIR spectral features of plant biomass components during cotton organ development and their biological implications. He et al. [25] observed that carboxylic acids are fundamental to the chemistry of crude oils and are collectively referred to as naphthenic acids. During crude oil refining, the presence of naphthenic acid is a major contributor to the corrosion of surfaces such as pipelines, vessels, and refinery equipment, especially at higher temperatures. The obtained component composition results of the analyzed crude oil sample used in this work and the samples used by He et al [25], however, contrast with the results of other analyzed material components by researchers from the literature, such as the results of the clay minerals in a clastic reservoir in FTIR tests obtained by Jozanikohan and Abarghooei [26] with the wavenumbers of 3621, 3432, 1034, and 515 cm^{-1} for illite, 3567, 1088, 990, and 463 cm^{-1} for glauconite, 3567, 3432, 1613, 1088, 990, 687, 651, and 515 cm^{-1} for magnesium-rich chlorite, and 3700, 3621, 3432, 1034, 687, and 463 cm^{-1} for kaolinite.

The absorption peak at 1454 cm^{-1} wave number suggests the S=O bond of sulphate ester, which implies that the crude oil contains sulfur or sulfur compounds. The corrosiveness of the oil is also affected by the type of sulfur-containing compounds that are present. From the FTIR spectrum, it can be seen that the crude oil contains a presence of sulfur compounds, which could likely be mercaptans, thiophenes, or elemental sulfur. Sulfur compounds that have a fraction or functional group tend to react with metallic compounds and cause corrosion [6], [19], [27].

The peak at 1068 cm^{-1} wave number denotes a C-O stretching vibration of carbon dioxide. CO_2 will mix with the water, forming carbonic acid, making the crude oil acidic and leading to corrosion of metallic surfaces. CO_2 corrosion is influenced by temperature, an increase in pH value, and metal characteristics [7], [8], [14], [15], [27].

The infrared absorption band at 1118 cm^{-1} and 810 cm^{-1} wave numbers is characteristic of alkyl halides possible from the reaction of the salts with other compounds. Crude oil could contain metallic salts such as NaCl, $CaCl_2$, and $MgCl_2$ based on its geological location. The presence of water or moisture leads to the reaction of these salts at elevated temperatures to produce hydrochloric acids and hydrogen sulfides and thus cause corrosion at low temperatures [8], [25], [27].

4. CONCLUSION

The proportions of corrosive components in all the differently-sourced crude oils employed at the Kaduna refinery in Nigeria were investigated using XRD and FTIR analytical techniques. The obtained results indicated that the crude oils contained sulfur, carbon, idrialite, refikite, flagstaffite, and silicon dioxide, up to a total proportion of about 35%. The analyses have shown that these components can per se cause corrosion of the refinery's structures and equipment to various levels or undergo reactions at higher refining temperatures and produce an appreciable quantity of corrosion agents that include acids, chlorides, water, carbon dioxide, hydrogen sulfide, etc. The presented information is useful in fundamental corrosion control strategies such as the use of corrosion inhibitors for better corrosion control of structural systems at the refinery.

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