



# **Laser-induced Fluorescence with Multivariate Analysis in Characterizing Crude Oils from Some Selected Oil Fields in Ghana**

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## **Authors' contributions**

*This work was carried out in collaboration among all authors. All authors read and approved the final manuscript.*

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## **ABSTRACT**

Developing scientific practices and procedures for finding the characteristics of various crude oils from different geological sources based on fluorescence spectra fingerprints would be beneficial to the petroleum industry. Laser-Induced Fluorescence (LIF) has gained relevance worldwide because of its advantages in crude oil analysis. Presently, the use of this technique in the characterization of crude oils from the oil fields in Ghana has not been studied. The study employed the LIF technique to determine some physical qualities of crude oils from Jubilee Oil Field, Tweneboa Enyenra Ntomme (TEN) Oil Field and Saltpond Oil Field. Specifically, this study used multivariate analysis methods to link the spectral signatures of the crude oils to their properties for identification and classification. The LIF technique was applied on four crude oil samples. Fluorescence spectra were obtained using a continuous wave 405.0 nm laser. The excitation source revealed five (5) peak wavelengths after deconvolution. Using Principal Component Analysis (PCA), Linear Discriminant Analysis (LDA) and Hierarchical Cluster Analysis (HCA), the crude oil samples were classified accurately.

*Keywords: Crude oil; laser-induced fluorescence; principal component analysis.*

## 1. INTRODUCTION

Crude oil is made up of mainly organic carbon (85-87%) and hydrogen compounds (12-14%) [1] with different complex mixes such as naphthenes, methane, butane, paraffin and other aromatic hydrocarbons. Based on its hydrogen predominance, crude oil can be categorized as a naphthenic base, intermediate base or paraffin base. These natural oils are found in different parts of the world and can be obtained from an extensive range of geological sources. The Sedimentary rocks of the oil fields are of unique geological composition and, therefore, the need to identify their physical characteristics [2,1]. Crude oil analysis is essential for characterization and standardization in the petroleum industry. Therefore, an interesting area that calls for research with efficient techniques to provide essential information on the geochemical qualities of crude oils. Studies have shown that chemical methods such as the Grote-Krekeler, Herman, Mortiz and other methods may be used to determine the quality of crude oil [3]. They require several hours of heating the samples to temperatures as high as 750.0 °C to obtain results. These methods are destructive to the sample and involve more than one process, making it tedious [4]. Simplicity in analyzing samples in optical spectroscopic methods, such as Laser-Induced Fluorescence (LIF), is very rapid and it provides quality assurance results. LIF method of spectral analysis prevents unavoidable impurity additions to samples as compared to chemical wet methods of analysis. LIF has aided new development and approaches to determine the physicochemical properties of crude petroleum and its products. This spectroscopic technique has unique diagnostic potential and has gained relevance in crude oil analysis. Laser-Induced Fluorescence (LIF) is one of the most widely utilized non-destructive spectroscopic techniques in petroleum technology for possible spectral measurements and analysis [5]. It offers rapid response, high sensitivity and selectivity, and contain valuable information related to the intrinsic physicochemical characteristics of each examined sample. In this technique, sample preparation is nondestructive and spectral line interference is relatively uncommon (El-Hussein et al., 2015). LIF is another method of fluorescence spectral profile that can be used to develop a standard wavelength fingerprint for light and heavy crude oils [5,6].

Other researchers have utilized LIF to predict crude oil quality based on their fluorescence emission spectra profiles (El-Hussein et al., 2015). Bujewski and Rutherford used a rapid optical screening tool coupled with LIF to ascertain the physicochemical characteristics of petroleum contamination at toxic waste sites. Also, Takumi et al, demonstrated the applicability of Time-Resolved Laser-Induced Fluorescence (TRLIF) in detecting metal ions in petroleum products, soil and contaminated seawater samples (Takumi et al., 2012; Collins et al., 2011).

The main objective of this study was to use LIF to characterize the crude oil samples by determining the fingerprint spectral qualities of crude oils from Jubilee Oil Field, TEN Oil Field and Saltpond Oil Field of Ghana. Specifically, multivariate analysis methods were used to link the spectral signatures of the crude oils to predict their properties, identify and classify them. The use of the LIF technique for the determination of some physical properties of crude oils from the active oil fields in Ghana would provide valuable information to the petroleum stakeholders for quality assessment. Therefore, there is the need to use this non-destructive technique to study these crude oils from Ghana.

## 2. LITERATURE REVIEW

The fundamental study of interactions of radiation with matter has become an enthralling developing research area in the petroleum industry. A thorough understanding of the sulphur content using fluorescence techniques can, therefore, not be underestimated [7]. The recent discovery of crude oil across several regions in Ghana has awoken a growing attentiveness to research in the petroleum industry. Therefore, a research work to estimate the concentration of elements in crude oil and hence the sedimentary rock of various oil fields in Ghana for standardization of crude petroleum and its products in Ghana is very important.

### 2.1 Crude Oil Formation

When animals and micro-organisms die and settle to sedimentary rock after a very long period, several layers of mud cover them. With changes in heat and pressure over a long period, these layers are converted to kerogens which are

estimated to be about  $10^{16}$  tons of carbon. It is the most abundant content of living matter by 10,000-fold (Evdokimov & Losev, 2007). Due to heat and pressure, the kerogens are cracked to form crude oil which may migrate from one layer to another till it is trapped by a cap rock that is neither porous nor permeable, hence called the source rock (Ante, 2013).

Crude oil is made up of mainly organic carbon (85 – 87 %) and hydrogen compounds (12 – 14 %) with different complex mixes such as naphthenes, methane, butane, paraffin and other aromatic hydrocarbons (Evdokimov & Losev, 2007). Petroleum is categorized as a naphthenic base, intermediate base or paraffin base depending on its hydrogen predominance. Tables 1 shows detailed information about the hydrocarbon and non-hydrocarbon composition of petroleum.

## 2.2 Non-Hydrocarbons

Information on crude oil composition has increased based on the recent development of apparatus and techniques. Not only scientists are able to ascertain the group of hydrocarbon structures in crude oil but also the whole make-up of the hydrocarbons and their respective structures (Hook, 2009). Examples of these new techniques include Laser-Induced Fluorescence, nuclear magnetic and paramagnetic resonances and X-ray fluorescence-based methods (Qasim, 2016). Contents of the trace elements in crude oil differ significantly, hence the upsurge in trace element studies by researchers across the global frontier (Hook, 2009). The discovery and recovery of trace elements from crude oil are technically complex and has not been regularly used, although scientific experimentation is in progress (UNEP/PCFV, 2009). Table 2 shows the elemental percentage composition range by weight in crude oil.

The physical and chemical properties of any given hydrocarbon species do not only depend on the number of carbon atoms present in the molecule but also the nature and type of chemistry existing between them (Abdalla, 2015). The different proportions of the vast hydrocarbon class and distribution determine the yield and quality of refined petroleum products. Also, the percentage composition of single and other multi-elements may influence crude oil and its products. The market price evaluation of crude oils varies according to the prevailing accepted standards (Appenteng et al., 2013). The classification of crude oil into light and heavier grades is used to determine yield quality since lighter grades produce quality yields than heavier grades. The heavier have to undergo the process of carbon removal and hydrogen addition through catalytic cracking units (Abdalla, 2015). Petroleum quality is linked to the concentration of sulphur present in it. The API gravity of crude oil plays a major role in determining its value on the world market. High API gravity oils command higher prices globally. Crude oil with low sulphur content crude oils is rated as sweet crude. Sulphur concentrations differ around 0.1% to 0.5% for sweet and 1% to 3.3% for sour crude (UNEP/PCFV, 2009). In the refinery process, heavy and sour crude oils require relatively much energy, time and maintenance cost than light and sweet crude oils.

## 3. MATERIALS AND METHODS

### 3.1 Crude Oil Sample

Four different composite run-down petroleum crude oil samples were obtained from Jubilee Oil Field, TEN Oil Field and Saltpond Oil Fields from the Research and Development Department of Ghana National Petroleum Corporation (GNPC). Table 3 summarizes the crude oil samples, oil field locations and sample codes used in this study.

**Table 1. Non-Hydrocarbon Composition of Petroleum (Hook, 2009)**

Non-Hydrocarbons	Products	Observations
Compounds of sulphur	Hydrogen Sulphide	Unwanted due to foul odour (0.5 % to 7%)
Compounds of Nitrogen	Quinoline Pyridine	After exposure to sunlight, the presence of nitrogen compounds in gasoline and kerosene degrades the colour of the fuel.
	Pyrrole Indole	Gum formation can be caused by them. Less than 0.2 %, usually.
Compounds of Oxygen	Naphthenic acids, Phenols	At different stages of manufacturing, these acids cause corrosion and contamination issues. Traces of materials are up to about 2 %

**Table 2. Elemental Percentage Range: (Bawazeer & Zilouchian, 1997)**

Element	Composition (wt %)
Carbon	38.0 – 87.0
Hydrogen	10.0 – 14.0
Nitrogen	0.1 – 0.2
Oxygen	0.05 - 1.5
Sulphur	0.05 – 6.0
Metals	< 0.1

**Table 3. Summary of the Crude Oil samples, Oil Field Locations and sample Codes used**

Crude Oil Sample	Oilfield/Drilling	Sample Code
FPSO MV21 Kwame Nkrumah	Jubilee	JF
FPSO MV25 Atta Mills	Tweneboa Enyenra Ntomme	TF
Saltpond Well 2	Saltpond	SF-1
Saltpond Well 4	Saltpond	SF-2

### 3.2 Experimental Setup

The spectral analytical setup consists of a radiation source, an analyte and a detection system. The radiation source is a continuous-wave diode laser with 100.0 mW maximum output power emitting at a wavelength of 405.0 nm. It is equipped with a laser diode driver (TTL modulation of 0 to 20.0 kHz) requiring an output voltage of 12.0 V at 2.0 A. Also, bifurcated optical fibre-probe with a core diameter of 600.0  $\mu\text{m}$  (Ocean Optics, U.S.A) was connected to the fibre port micro-positioner (PAF-SMA-5B, Thorlabs, U.S.A) which directed the beam out of the microscope objective lens (CP09/M, Thorlabs, U.S.A).

The detection system comprises CCD-based USB2000 spectrometer (USB2000 Ocean Optics, U.S.A) and 450.0 nm cut-off long-pass filter. It operates on low power characteristics of 100.0 mA at 5.0 V and sensitive to photon activity within the range of 200.0 nm to 1100.0 nm. Data from the charged coupled detector (CCD) is acquired by choosing from 1.0 ms to 60.0 ms as integration time. The collected data is converted to electric pulses and passed to the 001Base32 spectrometer on the PC for visualization.

### 3.3 Fluorescence Measurement

Laser-Induced Fluorescence measurements of the four crude oil samples were taken at LAFOC, Department of Physics, University of Cape Coast. The 405.0 nm CW diode laser was incident on each sample. The emitted fluorescence was detected and transmitted through an optical fibre with a long-pass filter

connected to a USB2000 spectrometer (Ocean Optics, U.S.A). The fibre was positioned behind a converging lens 90 degrees from the sample, and the spectrum was displayed on a computer screen. After the desired spectrum was acquired, the intensity and wavelength data of that spectrum were exported from the 001Base32 software into MATLAB version R2019a (MathWorks Inc., U.S.A) for further processing to obtain the fluorescence spectra.

### 3.4 Data Analysis

The fluorescence spectrum of each sample was deconvolved to extract information about the hidden peaks contributing to the entire spectrum. Two unsupervised pattern recognition techniques, Principal Component Analysis (PCA) and Hierarchical Cluster Analysis (HCA) were used to determine the differences in the four crude oil samples used. The score plot of the PCA was used as input variables to generate a Linear Discriminant Analysis (LDA) model using the principal components (PC1 and PC2) for the identification and classification of the crude oil samples. Six hundred (600) LIF spectra were obtained from the four crude oil samples measured from four different oil wells. Four hundred (400) of the LIF spectra representing 66.67 % of the entire LIF spectra obtained were used for the training set. Two hundred (200) of the remaining LIF spectra representing 33.33 % of the total spectra were used for the verification set.

### 3.5 Data Pre-processing Method

The fluorescence spectra data were pre-processed using maximum normalization to get

all the data on the same scale [8]. Maximum normalization involved dividing each fluorescence spectra by its maximum value instead of its average (Ozaki et al., 2006). This technique was employed to remove the undesired spectral variations resulting from the light interaction with the samples, the rigidity of the environment and noise from the detector [9,8]. The entire spectrum was subjected to the maximum normalization technique to obtain the mean and normalized fluorescence spectra for the four crude oil samples.

### 3.6 Peak Deconvolution

Fluorescence was made up of bands representing the constituents of the samples, and these bands cover ranges wavelength. When the bands are very broad, they end up overlapping and tend to be closer to each other hence the need to deconvolve the spectra (Subhash & Mohanan, 1995). Peak deconvolution can be described as the process of decomposing peaks that overlap with each other and extracting information about the hidden peaks depending on the existence of the baseline [10]. The procedure was implemented using the PeakFit software (PeakFit, Seasolve, Version 4.12) with the fluorescence spectra data as input points.

### 3.7 Principal Component Analysis (PCA)

Principal Component Analysis (PCA) is an exploratory and unsupervised pattern recognition statistical method used to divulge hidden structures within large data sets [11]. PCA decreases the data set by plotting each fluorescence spectrum as a single point in PC space, based on the variations across the entire data set [12]. It presents a visual illustration of the correlation between sample and variables and gives insight into how measured variables cause samples to vary or show similarities among each other. For more information, refer to these authors [13,14].

### 3.8 Linear Discriminant Analysis (LDA)

Linear Discriminant Analysis (LDA), also called Fisher's linear discriminant or mapping is used for dimension reduction. LDA works by finding the linear combination of features that spell out the precise ratio of between-class variance and reduces the ratio of within-class variance [15,16,17].

## 3.9 Hierarchical Cluster Analysis (HCA)

Hierarchical Cluster Analysis (HCA) is another unsupervised pattern recognition technique that identifies groups or clusters of data due to measuring the proximity between the elements. Data groups can be defined based on similarity and dissimilarity.

## 4. RESULTS AND DISCUSSION

### 4.1 Fluorescence Emission

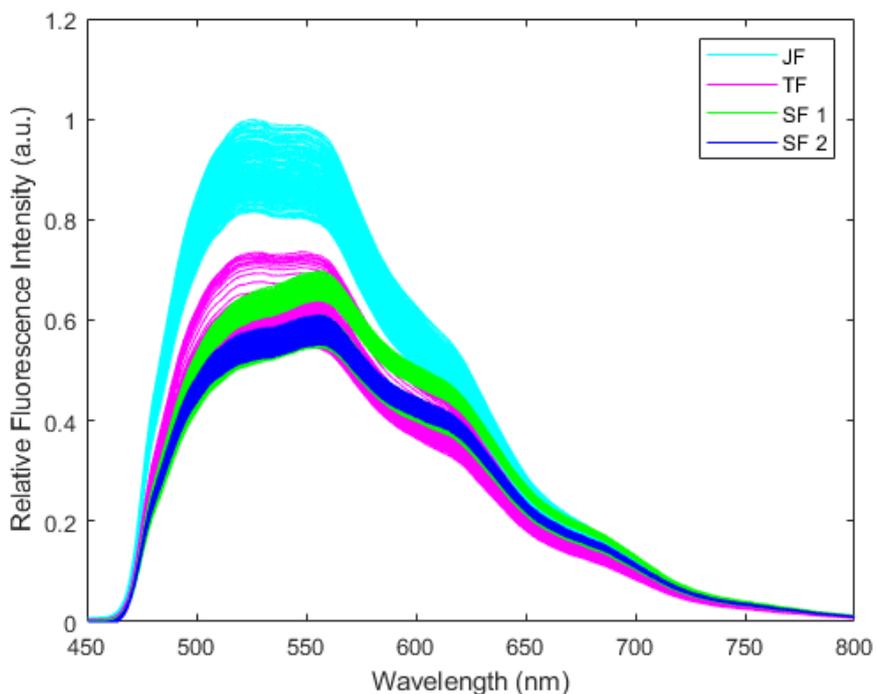
Unprocessed laser-induced fluorescence (LIF) spectra obtained from each of the four samples, JF, TF, SF-1 and SF-2, are shown in Fig. 1. The fluorescence peaks are observed near 500.0 nm and 560.0 nm, with shoulder fluorescence emission peaking around 620.0 nm and 650.0 nm. The fluorescence intensities for TF, SF-1 and SF-2 crude oil samples appear to be closer to each other than the crude oil sample from JF [5].

Fig. 2 shows the average normalized fluorescence spectra obtained for each of the four samples. The fluorescence spectra profile observed for TF, SF-1 and SF-2 crude oil samples in Fig. 2 fits the general characteristics of light oils with low specific gravities and lower fluorescence band intensities [5]. JF is characterized by a higher fluorescence band intensity, which implies that crude oil from JF is heavier than those from TF, SF-1 and SF-2 [5,18,19].

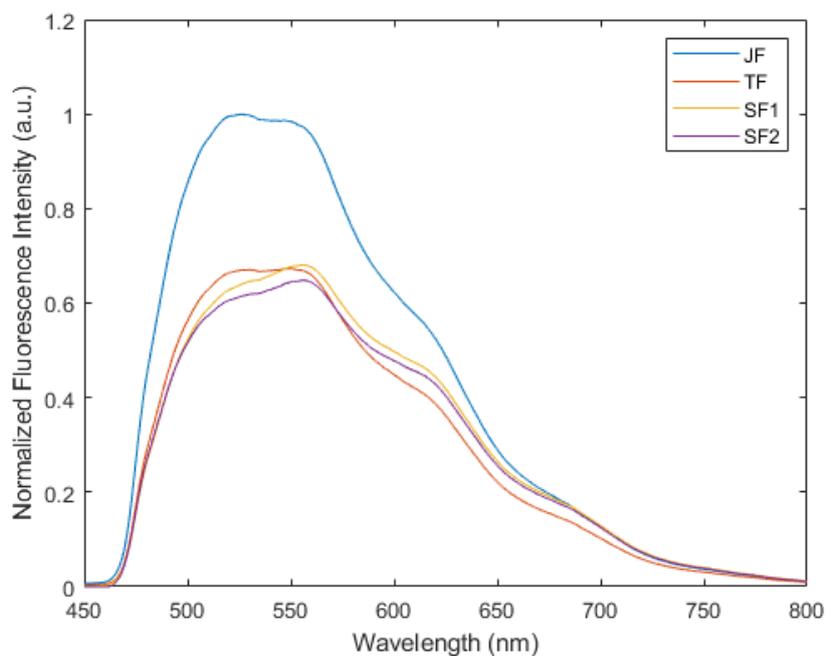
### 4.2 Fluorescence Spectra Deconvolution

Fig. 3 shows the deconvoluted fluorescence spectra of the crude oils from the four crude samples of the three oil fields. The deconvoluted fluorescence spectra of JF, TF, SF-1 and SF-2 consisted of Gaussian components observed at different peak wavelengths and respective intensities. It can be observed that the significant fluorescence peaks occur within a range of 510.0 – 750.0 nm. These fluorescence peaks are similar to other fluorescence emission results obtained for crude oils observed by other researchers [5,17], (Owens and Ryder, 2011; Schultze et al., 2004; Asiamah et al., 2013). The similarities in peak wavelengths for SF-1 and SF-2 suggest that they possess the same fluorophores and could have the same molecular characteristics since they are both drilled within the same field. The peak wavelengths and

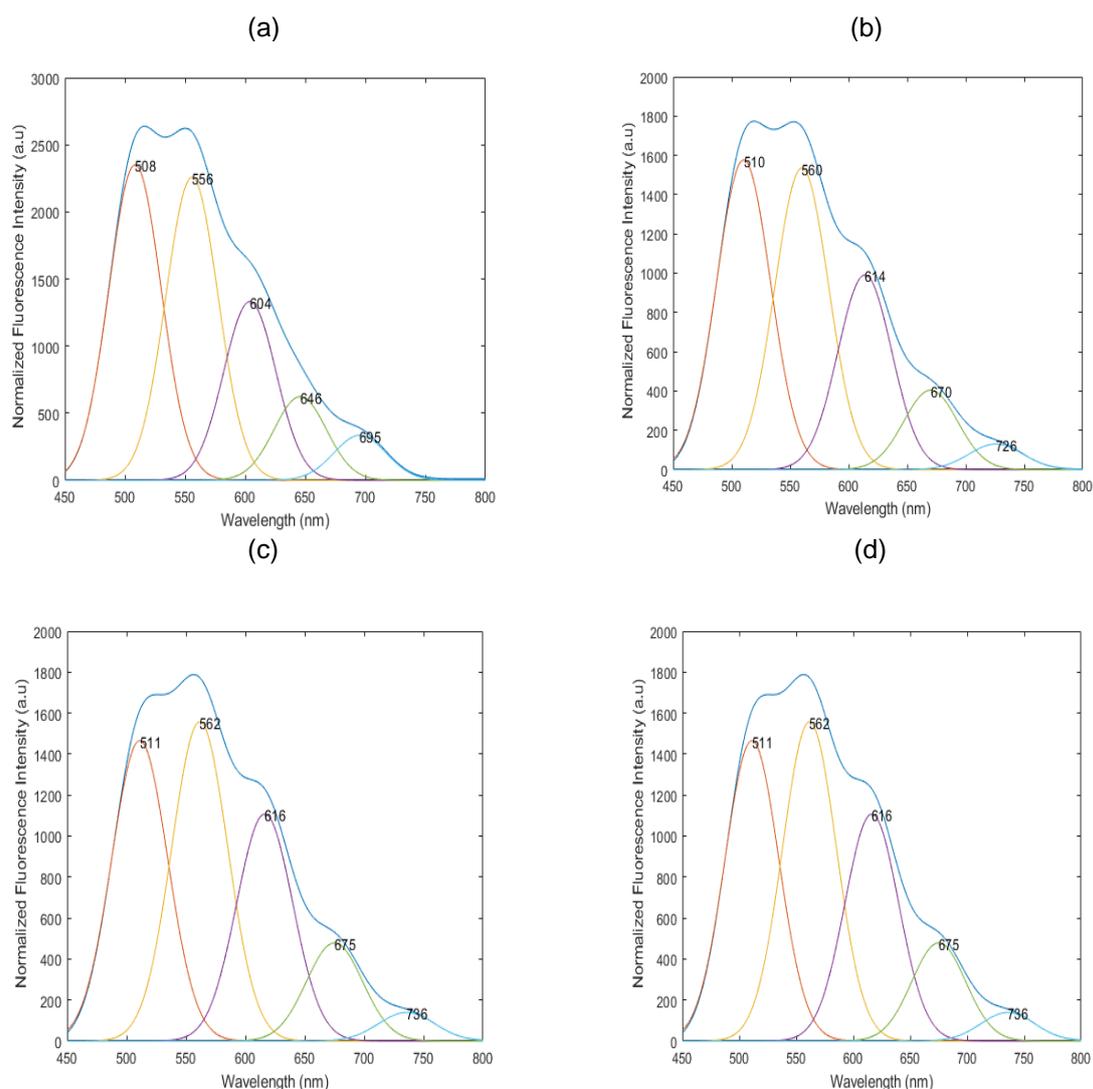
intensities of the deconvoluted fluorescence spectra of the crude oil samples from the four (4) oil wells JF, TF, SF-1 and SF-2 are shown in Table 4.



**Fig. 1. Unprocessed Relative Fluorescence Spectra of all the Crude Oil samples, JF, TF, SF-1 and SF-2 from the four Oil fields showing the Dominating Spectral Signatures**



**Fig. 2. Average Normalized Fluorescence Spectra from four Crude Oil samples from four (4) Oil Wells**



**Fig. 3. Deconvoluted Fluorescence Spectra of (a) JF, (b) TF (c) SF-1 and (d) SF-2 showing the Hidden Peaks**

**Table 4. Results of Deconvoluted Fluorescence Spectra for Crude Oil samples from all four (4) Oil Wells**

PEAKS	JF		TF		SF-1		SF-2	
	$\lambda$ (nm)	I (a. u.)						
1	508	2350	510	1576	511	1465	511	1438
2	556	2257	560	1535	562	1557	562	1495
3	604	1331	614	990	616	1107	616	1075
4	645	623	670	404	675	479	675	468
5	695	335	726	130	736	139	736	138

The differences in the peak wavelengths suggest that the crude oil samples from the oil fields of Saltpond TEN and Jubilee have different fluorophore compositions and do not have the same molecular characteristics [5,17]. The average peak wavelength values of

deconvoluted fluorescence spectra for crude oil samples from the three oil fields JF, TF, SF-1 and SF-2 are shown in Table 5. These average peak wavelengths provide the standard deconvoluted peak wavelength ranging from 510.0-750.0 nm. This determines the specific

peak wavelengths range at which fluorescence was occurring. The peak wavelengths also suggest that there are five (5) main different fluorophores responsible for fluorescence emission in the crude oil samples.

### 4.3 Principal Component Analysis (PCA) of the Fluorescence Spectra

Principal Component Analysis (PCA) was worth exploring to find the variations in the fluorescence spectra to allow for more insight into the difference between JF, TF, SF-1 and SF-2. The PCA simplified the large data set obtained from the fluorescence measurements by reducing the dimensionality, extracting the principal component coefficients and the eigenvalues of the covariance matrix of the data. Each spectrum was then plotted as a single point in PC space based on the variance across the entire data set.

Fig. 4 shows the screen plot containing the eigenvalues of the covariance matrix of the fluorescence spectra from the crude oil samples. It determines the number of factors to retain in the exploratory analysis (EA).

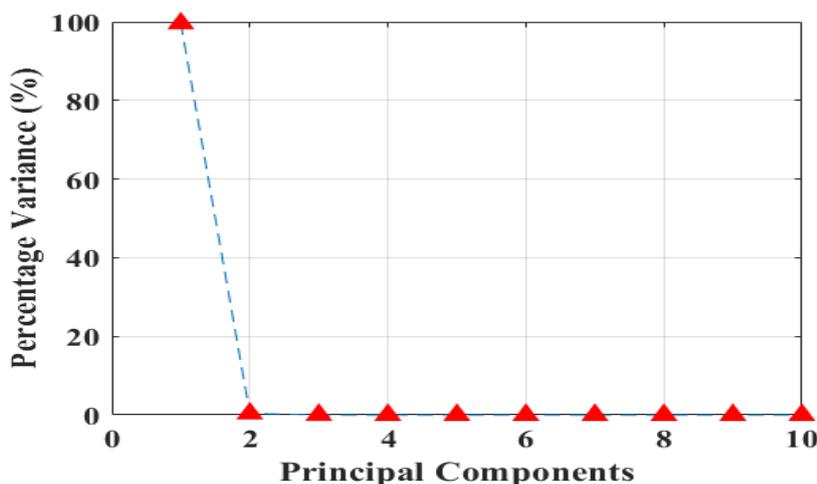
It has been suggested that the point where the screen plot levels off and flattens is where the best possible principal components (PCs) can end [20,21,22]. And also, may predict the amount of explained variance of the eigenvalue (Zhao et al., 2016). So, two PCs were selected to represent the real data. The two PCs (PC1 and PC2) preserved the maximum variance in the fluorescence spectra. Out of 99.97 % discrepancy in the fluorescence spectra, PC1 contributed 99.59 % and PC2 0.38 %.

The loadings or coefficient plot is shown in Fig. 5. It is made up of the coefficients of the principal component. The rows of the principal components correspond to the wavelengths, while the columns correspond to the principal components. The loadings plot shows the regions where the differences and similarities between the fluorescence spectra can be observed within the 450.0 nm – 800.0 nm range. It also reflects the unique grouping of the four crude oil samples. This plot helps to apprehend the trend of the observations. From the plot, the significant differences in the fluorescence spectra are set to occur within the 470.0 – 570.0 nm range.

**Table 5. Average Peak Wavelength Values of Deconvoluted Fluorescence Saltpond (SF 1 & SF 2) Oil Fields**

Peaks	Wavelength (nm)
1	510.00 ± 1.41
2	560.00 ± 2.83
3	612.50 ± 5.74
4	666.50 ± 13.87
5	723.25 ± 19.41

*Data behind ± for this work are standard deviations*



**Fig. 4. Number of Principal Components and their Contribution to the Variation in Data Set Explained Percentagewise**

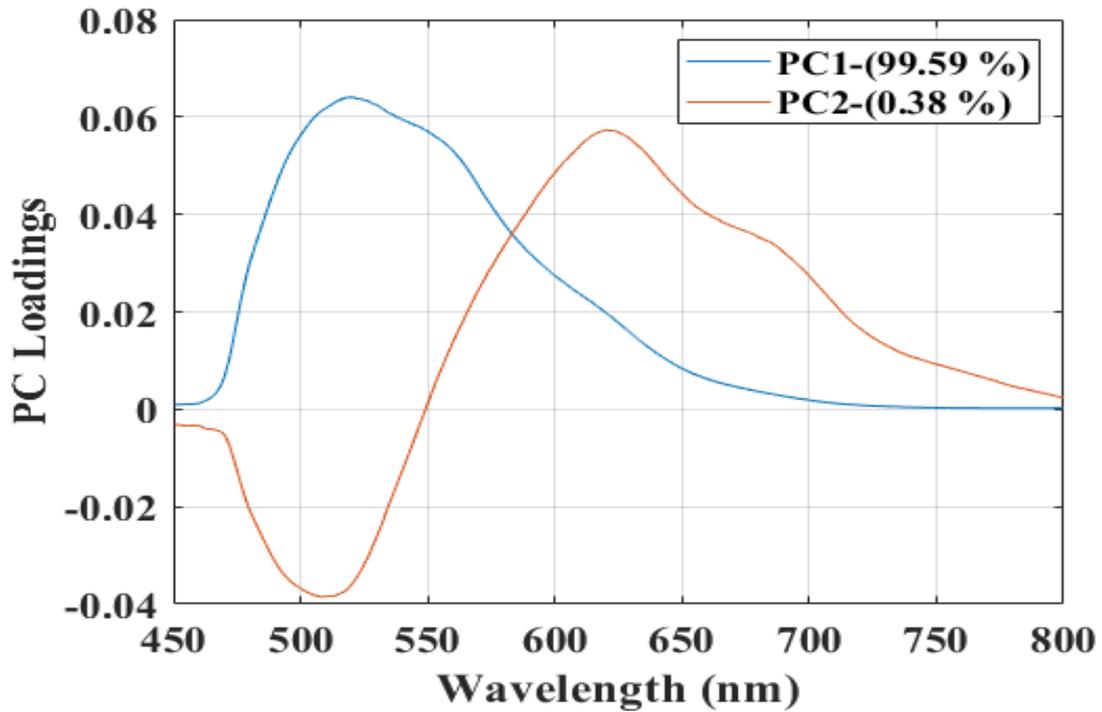


Fig. 5. Loadings Plot from the PCA of the LIF Spectra giving information about the significant sources of Variation in the Data

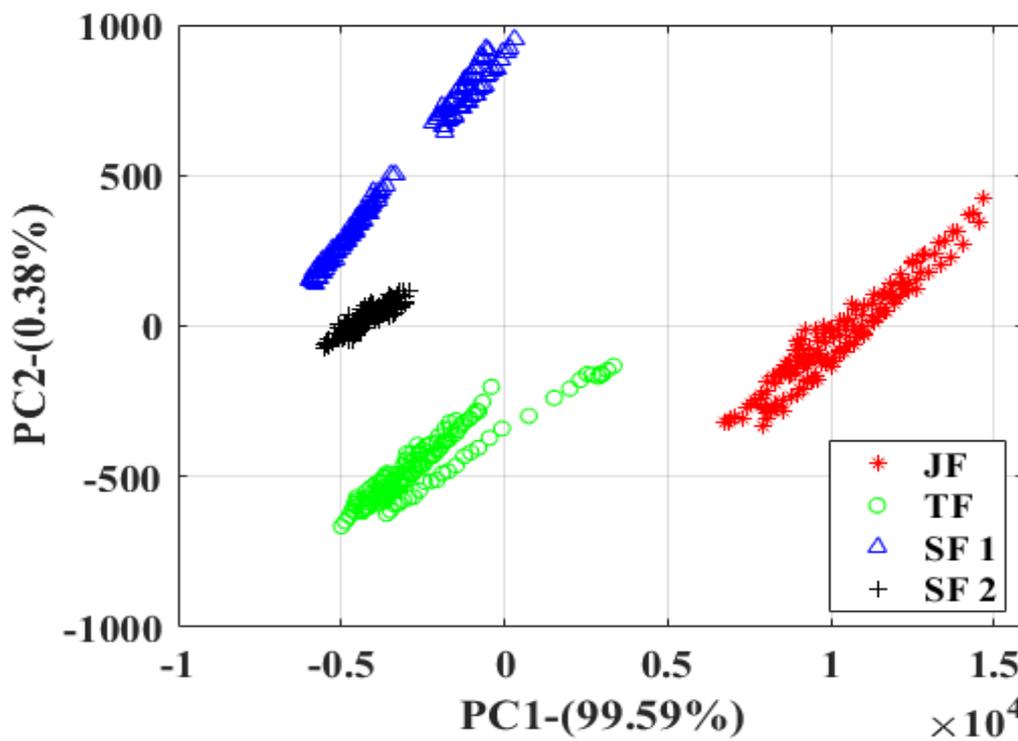


Fig. 6. Score with only two PCs Plot for PC1 and PC2 from Principal Component Analysis of the LIF Spectra Obtained from the Four Crude Oil Samples, JF, TF, SF-1 and SF-2

The score plot in Fig. 6 shows the clustering of the fluorescence spectra in PC space based on the two significant principal components selected and it gives the variance across the entire data set. Sample JF separated from TF and SF along PC1, which suggests that crude oil from JF is heavier than TF and SF. The negative loadings for PC2 show a trough, which explains the differences observed in the fluorescence spectra of JF, TF, SF-1 and SF-2 samples in the score plot. It is, therefore, feasible to report that, due to the different geological sources from which the crude oil samples were obtained, it was observed that crude oil composition varied from oil field to oil field. The PCA has shown that the crude oils from these four different oilfields can be discriminated against with only two PCs.

#### 4.4 Linear Discriminant Analysis (LDA)

Fig. 7 shows the confusion matrix of the LIF spectra trained and test sets using the LDA model from the four crude oil samples. The confusion matrix provides a summary of what the discriminant function has done for each class. Each row shows the correct label, and each column shows one type of prediction. Two hundred (200) LIF spectra were used for the test set. The classification results for both trained and

test sets were 100 % using the LDA. This means that there was no misclassification of the crude oil samples based on their LIF spectra.

#### 4.5 Hierarchical Cluster Analysis (HCA) of the Fluorescence Spectra

Hierarchical Clustering Analysis (HCA) works by generating a data hierarchy leading to the formation of a dendrogram. Fig. 8 shows the dendrogram of the LIF spectra of the four crude oil samples from JF, TF, SF-1 and SF-2 oil wells.

The cluster labelled I consist of SF-1, SF-2 and TF, while cluster II comprises JF. The HCA has been able to discriminate between the crude oil samples by calculating the linkage or Euclidean distance between JF, TF, SF-1 and SF-2 and classifying them into two major clusters. The shorter linkage distances observed in cluster 1 suggest that TF and SF are lighter oils since they form under one cluster. Also, JF separates from the rest of the samples with a longer linkage distance, suggesting that JF could be heavier in molecular composition. The ability of the HCA to discriminate between the crude oil samples suggests that HCA can also be used to classify the crude oils from the three different oil fields.

(a)

True Class	JF	97			
	SF 1		94		
	SF 2			106	
	TF				103
		JF	SF 1	SF 2	TF
		Predicted Class			

(b)

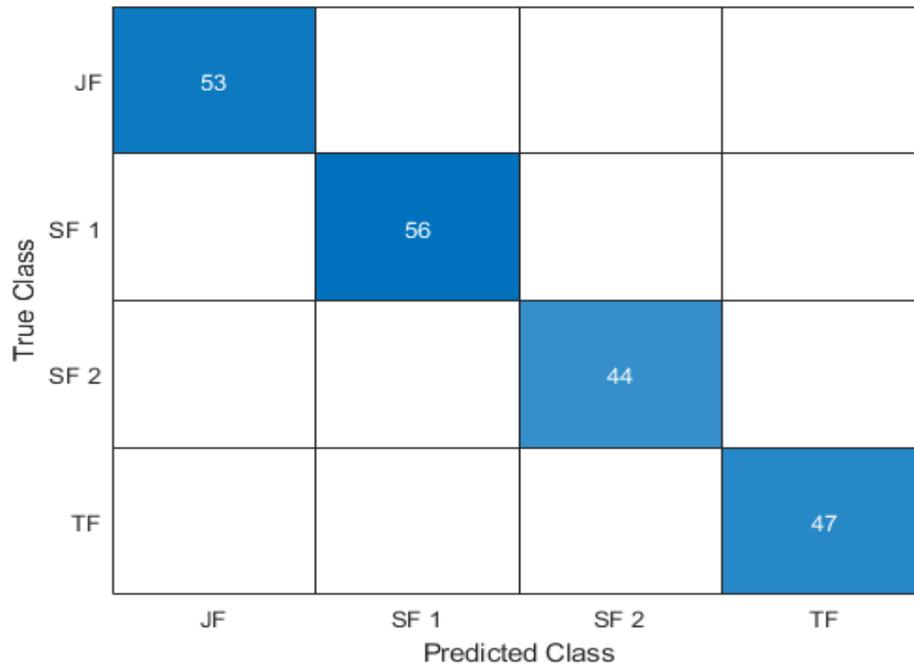


Fig. 7. Confusion Matrix for the LIF (a) Trained Set and (b) Test Set of Crude Oil samples for the LDA Model

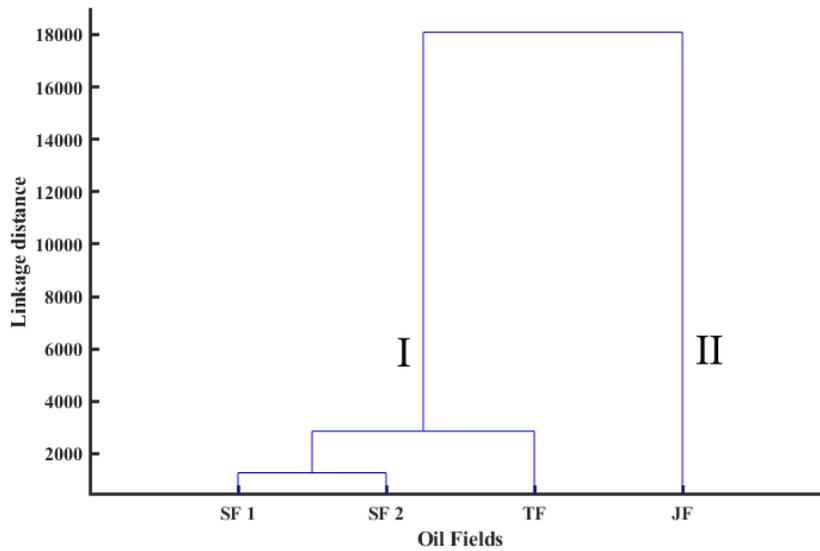


Fig. 8. Dendrogram of the Laser-Induced Fluorescence Spectra of the four Crude Oil samples from JF, TF, and SF

## 5. CONCLUSION

This research has shown the potentials of LIF in combination with multivariate analysis for

measurement, identification and classification of crude oil samples. PCA and HCA identified cluster trends in the spectra data and classified JF, TF, SF-1 and SF-2 according to their

geological locations based on the LIF spectra obtained. The results generated by the confusion matrix on both trained and test sets were 100 % using the LDA. There was no misclassification of the crude oil samples based on their LIF spectra. Therefore, LIF coupled with PCA, HCA and LDA was used as a fast and straightforward spectrochemical analytical method to characterize crude oil effectively. It is recommended that state-owned corporations, petroleum research institutes and laboratories adopt the use of the LIF technique to identify and classify petroleum oil samples because of its cost-efficient, simple design, rapid result and quality assurance outcome. The technique can also be used to detect adulteration of crude oils and other petroleum products in the petroleum manufacturing industries, thus making it an effective tool for quality assessment.

### COMPETING INTERESTS

Authors have declared that no competing interests exist.

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