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Classification of Malaysian Soil Species Based on FTIR and Laser Raman Spectroscopy Analysis

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Abstract: This study focused on the characterization from two groups of soil species which is three species of soil from alluvial soil and two species of soil from sedentary soil. The soil samples are taken from 5 different places around Johor Bahru and Batu Pahat, and name for each soil species that taken is Keranji (thionic fluvisols), Selangor-Kangkong (Dystric Gleysols), Peat (Dystric histosols), Kulai- Yong Peng (Rhodic Nitosols-Ferric Acrisols) and Rengam- Jerangau (haplic acrisols-rhodic ferralsol). The objectives of this study were to measure the elemental composition in several morphology of soil in Johor state using SEM-EDX and XRD, to characterize the functional group and molecular structure for each soil species using FTIR and Laser Raman Spectroscopy and to compile the information of elemental composition, functional group, and molecular structure for Malaysian soil species database. The soil samples were dried in the oven and crunch with pestle and mortar until the samples turned into fine paste. There are 5 different instruments to identify the characterization for each soil sample. The crystallography of cubic, orthomobic and others are obtained through the X-Ray Diffraction analysis. The percentage of elements composition and morphology surface were recognized by SEM-EDX analysis. Next, FTIR analysis identified the presence of functional groups within the powder soil. Laser Raman spectroscopy was identifying the molecular vibrational and revealed that Keranji soil was rich in organic molecules, which are good solvents for other organic compounds because of their low reactivity.

Keywords: Characterization Of Soil, Morphology Of Soil, Functional Group

1. Introduction

In Malaysia generally, there are three main groups which are alluvial soil, sedentary soil, and urban and mined land. The alluvial soil is surface water deposited soil. They are to be found near rivers, in floodplains and deltas, on stream terraces and regions termed flood fans. Next, sedentary soil is that remaining on the rock from which it has developed. The characteristics of each soil are different by their texture, properties, structure, and colour of the soil [1]. In this research XRD, Laser Raman Spectroscopy, and SEM-EDX were used to find the characteristic of soil. The elemental composition

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properties are characterised using SEM-EDX. This technique is resourceful and quickly used for each macro element in soil [2]. XRD can identify the characteristic of metal oxide and also can characterise crystalline and poor crystalline metal oxide [3]. Laser Raman Spectroscopy is a procedure to identify the molecular structure for each soil. Raman was used in discriminating against various soil samples and this technique is not sufficient for sample discrimination and suffers from matrix interferences, in particular during soil sample analysis [4].

This study is important due to the fact that we can create awareness to the public that one database for all soil species in Malaysia will help students and researchers in the future. The collection of soil species needs to be specific and complex and to reach this target, there are many steps to figure out and more challenging. Part of the problem encountered by non-soil scientists when writing about soil classification is the frequency with which soil scientists themselves change the system. Soil classification has a poor reputation because it has too many classification of soil, changing frequently, data too difficult to obtain and the specialists disagree on the exact name of soil [5]. A classification may only be built if a large number of soils are known and suitably described. Soils outside of the primary crop growing regions, in particular, are less well recognised. Soil surveys conducted across the world regularly feed the database with information on new soils, allowing for continuing improvement of the existing categorization systems. This method of functioning will continue for a long time [6].

The aim of this research was to measure the elemental composition in several morphologies of soil in Johor state using SEM-EDX. Next, to characterize the functional group and molecular structure for each soil species using FTIR and Laser Raman Spectroscopy and at the end of the experiment, we need to compile the information of elemental composition, functional group, and molecular structure for Malaysian soil species database. The soil samples are collected from 5 selected locations in Johor state which is Pasir Gudang, and Batu Pahat according to the map of soil species. The information of elemental composition, functional group, and molecular structure for all species need to be compiled in a database for the research.

2. Methodology

There are 3 steps of preparation for all samples which are to remove unwanted particles (such as dry leaves), dried in the oven (at temperature 110ºC for 24 hours), and grinding by using pestle and mortar (powder form).

2.1 Sample preparation for XRD

Each dry soil sample with weight 0.2 g, was placed in the XRD holder. The sample needs to be pressed and compressed until it flat.

2.2 Sample Preparation for SEM-EDX

The sample needs to undergo the coating process with gold sputtering. The soils will fill on the pin stub and then put in the coating machine. The gold sputtering process is to protect the variety of surface safe and prevent corrosion.

2.3 Sample dissolve in liquid form for FTIR analysis

The sample for each soil will be dissolved in the distilled water and this process is to prepare the sample that will be tested in FTIR. The amount of soil sample is 1g and the amount of distilled water is 10mL. The sample of soil is placed in a small beaker of glass and put in a little of distilled water and then stir the sample until it dissolves and becomes darker.

2.4 Pressing pellet for Raman Spectroscopy analysis

The sample needs to undergo the pressing pellet. Raman scattering can be used in powder form, because pellet samples can provide Raman spectra with a better Signal-to-noise ratio. The sample needs to be compressed for 1 minute. The weight for each sample needed is 2g and size for the pellet is 1 cm× 1cm × 1cm.

3. Results and Discussions

The properties that were analyzed were the elemental composition, functional group, and molecular structure for Malaysian soil species.

3.1 Crystallography analysis using XRD

XRD diffractograms from Figure 1-5 show the soil particle. There were several soil particles. In Figure 1, there are 2 of crystallography which is the higher peak showing the bikitaite compound. This compound of crystallography is monoclinic while Genthelvite, has cubic crystallography. Monoclinic crystal has three unequal axes, two are inclined to one another, the third is perpendicular. The cubic crystal is three perpendicular axes of equal length.

From Figure 2, the XRD spectrum identified 4 crystallography in Rengam-Jerangau which is the highest peak is Fourmarierite and has orthorhombic crystal while the lowest peak is Nordstrandite and has triclinic crystallography. For knowledge, an orthorhombic crystal known as three mutually perpendicular axes of different lengths and triclinic crystal is Three unequal axes with oblique angles. The highest peak for Kulai- Yong Peng soil as shown in Figure 3, is Kaolinite and has triclinic crystallography.

Figure 4 shows only 2 crystallography which is hexagonal (quartz low) and cubic (Canfieldite) and Figure 5 is peat soil, and shows many of crystallography which has 8 crystallography. The higher peak of peat soil is Sicklerite. The crystallography for Sicklerite is Orthorhombic.

To compile the data received from figure 1-5, each diffraction peak shows a plane in the lattice of that material. Heights of a plane show how much x-ray gets diffracted from a particular lattice plane and that show dominance of that plane in that material and the peat soil show that this soil sample has many materials in it.

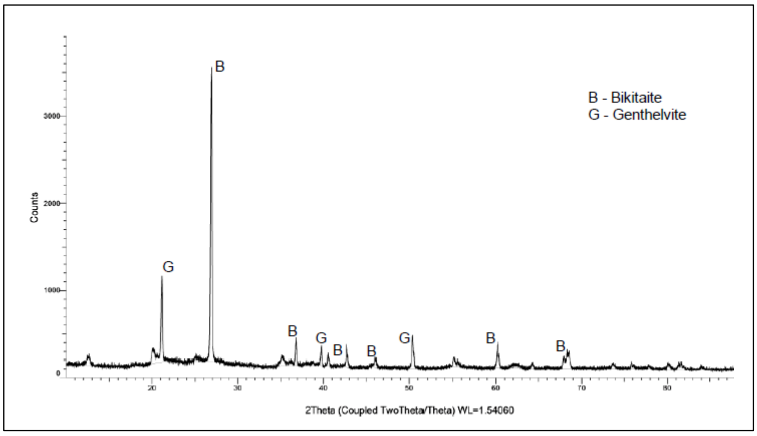

Figure 1: The XRD spectrum of Selangor- Kang Kong soil species

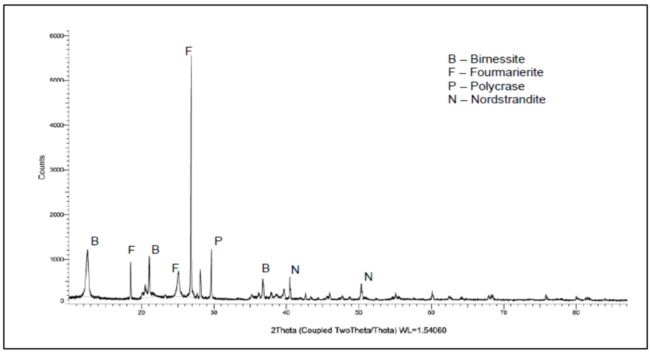

Figure 2: The XRD spectrum of Rengam- Jerangau soil species

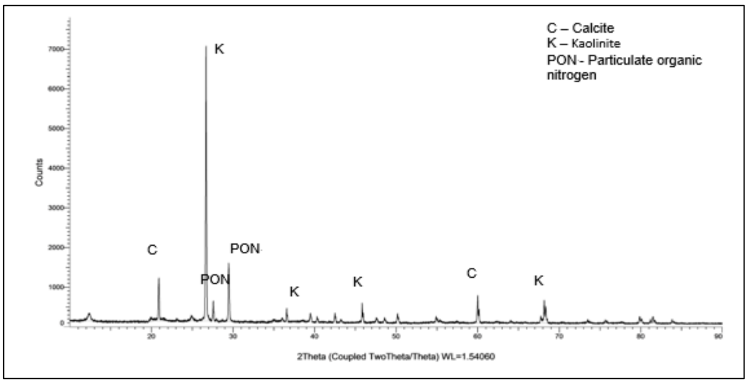

Figure 3: The XRD spectrum of Kulai- Yong Peng soil species

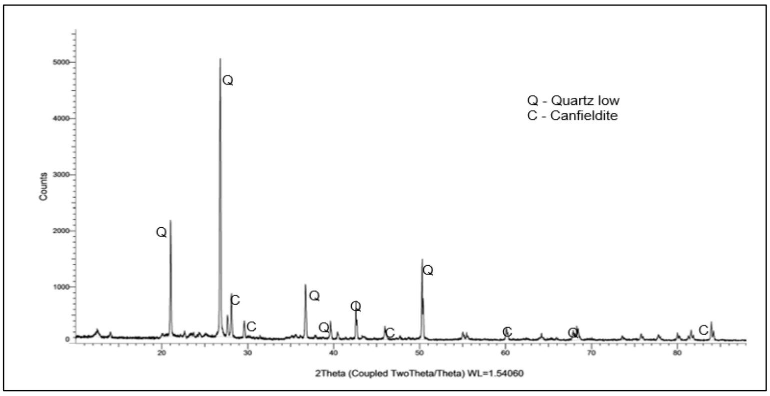

Figure 4: The XRD spectrum of Keranji soil species

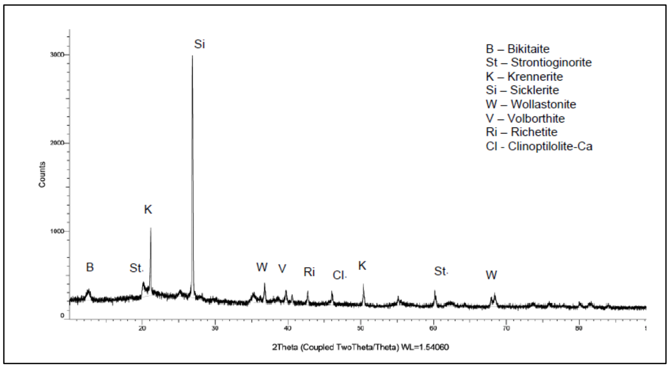

Figure 5: The XRD spectrum of Peat soil species

3.2 Elemental composition and morphology analysis using SEM-EDX

From the SEM analysis, the surface morphology of soil powder samples was examined and elemental composition of the soil powder was analysed by EDX analysis. Figure 6 represents the SEM micrographs of the Peat and Selangor- KangKong soil powder. The image shows that the surface of the peat soil powder is rough, and large pores where organic fragments of various sizes were seemingly strewn in a random fashion. This inherent porous fabric of the peat could bring effects to the filtration process. Moreover, large pores are usually associated with high shrinkage, indicating possible volume change of the Peat media over time. Other than that, peat samples have irregular structure due to metal ions that were trapped on the open pores while the shiny big atoms represent the presence of heavy metals on the surface of the Peat and Selangor – KangKong soil samples.

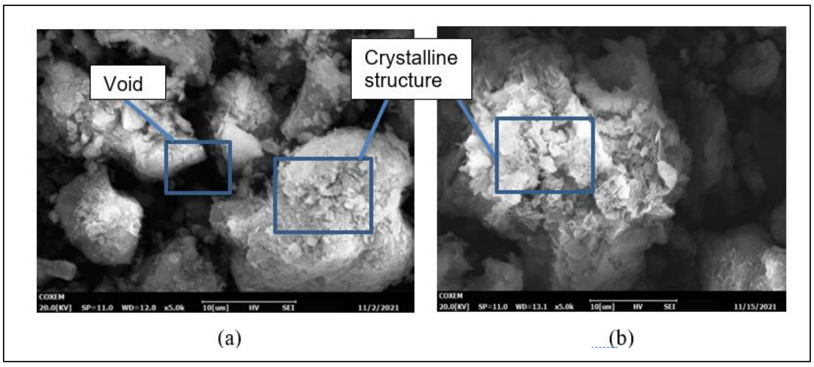

Figure 6: (a) The morphology structure of peat from SEM analysis, (b) The morphology structure of Selangor-kangkong from SEM analysis

Table 1: Elements of Peat soil sample and atomic percentage

Elements	Atomic %
Si	10.60
O	61.21
Al	7.57
Fe	1.22
Ca	1.05
K	0.28
C	17.78
Mg	0.15
Cu	0.13

Table 2: Elements of Selangor-Kangkong soil sample and atomic percentage

Element	Atomic %
Si	9.28
O	50.40
Al	4.51
Fe	0.56
K	0.34
C	34.56
Mg	0.22
Cu	0.12

Several elements were discovered in the Peat and Selangor-Kangkong soil samples, as shown in Tables 1 and 2. Copper (Cu) has the lowest atomic percentage of any heavy metal, at 0.13%. While oxygen (O) has the largest atomic percentage observed in peat soil samples (61.21%), it is not a heavy metal element. It is due to the rapidly expanding industrial areas, mine tailings, disposal of high metal wastes, leaded gasoline and paints, land application of fertilisers, animal manures, sewage sludge, pesticides, wastewater irrigation, coal combustion residues, petrochemical spillage, and atmospheric deposition. Because oxygen atoms are consumed by plant roots and soil bacteria during respiration, oxygen is a higher element in soil, and oxygen is a vital element for plant development. Reduced concentrations in the soil will have an impact on plant physiological activities such as nutrient and water absorption, as well as respiration.

The number of carbon elements presented from the EDX spectra is less than oxygen, because all carbon has completely burned during the dried process and turned into gas products. Carbon atoms react with the oxygen gas to form carbon dioxide after the pyrolysis process is completed. Pyrolization process is the process of chemical decomposition of organic matter at high temperature. A strong reaction between carbon and oxygen causes more carbon atoms loss to the environment at the highest temperature. Oxygen could be produced from the oven ambient during the heating process. Carbon particles turn into gas during the heating process and cause the reduction of carbon content in Peat and Selangor- KangKong soil samples.

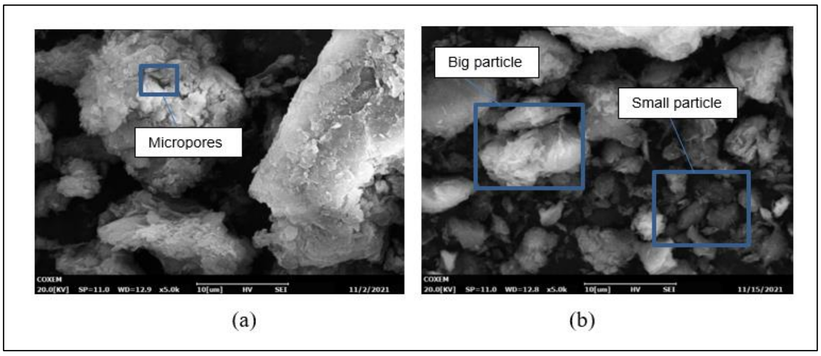

Figure 7: (a) The morphology structure of Kulai-Yong Peng from SEM analysis, (b) The morphology structure of Rengam- Jerangau from SEM analysis

Second, Figure 7 shows SEM micrographs of soil samples from Kulai-Yong Peng and Rengam-Jerangau. The picture from the Kulai-Yong Peng soil sample shows macropores, whereas the one from Rengam-Jerangau shows a blend of tiny and large particles. Micropores are microscopic soil holes that often exist between aggregates. Micropores drain easily by gravity, allowing for simple passage of water and air, as well as providing a home for soil creatures and allowing plant roots to develop into them. The majority of soil is made of sand, silt, clay, and decomposing plant and animal debris. Sand, silt, and clay are all created from various rocks and minerals, but the size of their particles is the most significant distinction between them. Sand is the largest particle in soil, clay is the smallest, and silt is somewhere in between.

Table 3: Elements of Kulai- Yong Peng soil sample and atomic percentage

Elements	Atomic %
C	56.11
Al	2.70
Si	3.03
O	36.98
Ca	0.33
Fe	0.43
Mg	0.13
Cu	0.19
K	0.10

Table 4: Elements of Rengam – Jerangau soil sample and atomic percentage

Elements	Atomic %
C	56.11
Al	2.70
Si	3.03
O	36.98
Ca	0.33
Fe	0.43
Mg	0.13
Cu	0.19
K	0.10

Several elements were discovered in soil samples from Kulai-Yong Peng and Rengam-Jerangau, as indicated in Tables 3 and 4. Carbon (C) has the greatest atomic percentage in the Kulai-Yong Peng soil sample, at 56.11%. Carbon is the most abundant component of soil organic matter, and it contributes to the soil's water-retention capacity, structure, and fertility. This is due to the fact that soil organic matter enhances soil particle agglomeration. Improved aggregation leads to improved soil structure, which allows for the passage of air and water through the soil as well as improved root development. Following that, oxygen (O) has the greatest atomic proportion in Rengam- Jerangau, at 50.54%. This is due to the fact that when soils are saturated with water, all pores are full of water and the oxygen concentration is very low. After the soils were dried, all pores were virtually filled with air, therefore the soil moisture content was very low and the oxygen level in the soil sample was high. Potassium (K) has the lowest atomic percentage in both soil samples from Kulai- Yong Peng and Rengam- Jerangau, at 0.10% and 0.06%, respectively.

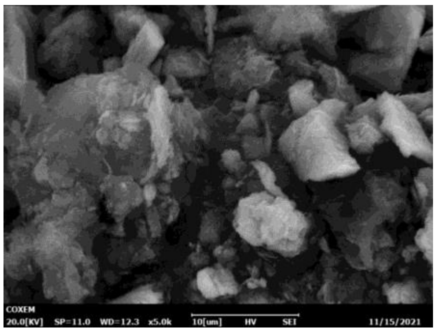

Figure 8: The morphology structure of Keranji from SEM analysis

Finally, Figure 7 depicts the morphological structure of a Keranji soil sample as determined by SEM examination. The photograph depicts the rough surface of Keranji soil. Soil surface roughness, also known as soil surface irregularities, is created by elements such as soil texture, aggregate size, rock fragments, plant cover, and land management.

Table 5: Elements of Keranji soil sample and atomic percentage

Element	Atomic %
Si	17.00
O	76.00
Al	7.00

Table 5 shows the EDX spectrum analysis of Keranji soil sample, and there were only three elements that can be detected in EDX spectrum analysis. Atomic percentage of Keranji soil sample is shown in Table 5. The highest atomic percentage in Keranji soil sample is oxygen (O) with 76.00% while the lowest atomic percentage is aluminium (Al) with 7.00% and silicon has 17.00%. The number of oxygens was the higher element in the sample, it is because the sample was drying in the oven for 24 hours. Soil will release carbon gas during the heating process and the oxygen gas will be absorbed. There were several elements that were not detected, this because of some error during the preparation of the sample.

3.3 Functional group analysis using FTIR

The peak from all soil species shows the same functional group finding. The initial greatest intensity peak with absorption ranges between 3470~3200 cm^{-1} is due to H-bonded and O-H stretch where the substances that are present are hydroxyl compound, alcohol, and phenol. The hydroxyl compounds formed a hydrogen bond, which led them to cling together, resulting in a greater boiling and melting temperature. Alcohols have a hydroxyl functional group that is linked to carbon, resulting in a higher boiling point. Furthermore, the carboxylic acids group exists in the absorption range of 3500~2400 cm^{-1} due to infrared vibration of O-H stretch, carboxylic group, and acidic. Carboxylic acid have higher boiling points than water due to their larger surface area and proclivity to form stabilised dimers. As a result, they may be evaporated or boiled as dimers more simply by breaking the dimer link or evaporating the entire arrangement.

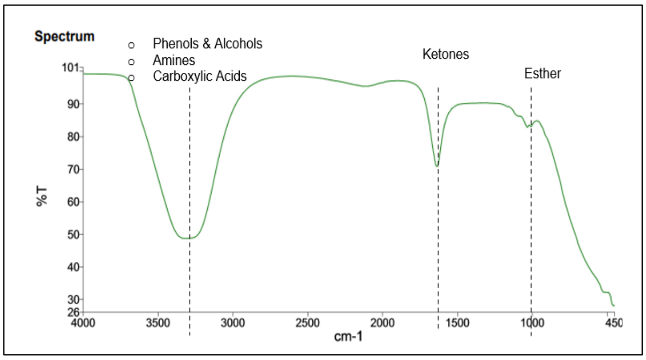

Figure 8: FTIR spectra for Rengam-Jerangau, Selangor-Kangkong, Keranji, Peat and Kulai-Yong Peng

The functional group that resembles the wavenumber ranges between 1650~1600 cm^{-1} is ketone compound due to the C=O stretching. The O-H bend and alcoholic group are produced between 1675~1600 cm^{-1} ranges of vibration while the aldehydes are caused by 1750~1625 cm^{-1} ranges of vibration. The aldehyde compound possesses a hydrogen atom that is bonded to the carboxyl group and easy to oxidise. The oxidation occurs when combined chemically with oxygen and undergoes an exothermic reaction such as combustion process. By gathering the data received from the FTIR analysis, it is concluded that the functional group that is present in the powders are almost identical for the raw peat sample with or without the additives and the wavenumber between range 1300 ~ 1000 cm^{-1} is Esther compound due to the C-O stretching.

By combining together, the data received using FTIR analysis, it is concluded that the functional group showed that the samples have ketones, esther and the combination of phenols, carboxylic acids and amines. Phenolic compounds are well-known phytochemicals found in all plants. They consist of simple phenols, benzoic and cinnamic acid. Phenolic compounds can be defined as plant substances, which possess in common an aromatic ring bearing one or more hydroxyl groups. Consequently, the most common form in which they are found in nature is as glycosides, which are soluble in water and organic solvents. They may also be found joined to carboxylic acids, organic acids, amines, lipids, and other phenolic compounds. This is in good agreement with the chemical compound of soil.

3.4 Laser Raman Spectroscopy data for soil particle

Figure 9 shows the Raman spectrum of Peat soil species. There were several chemical molecular structures in the Peat soil sample. The molecular structure at 625.46 cm^{-1} is S-S bond, and also called disulfide bonds. Function of disulfide bonds is to stabilise the tertiary or quaternary structures of proteins. Other than that, there was another bond which is C-S bond. The C-S bond is the foundation of organic synthesis, and it is crucial in the synthesis of natural products, biomolecules, and functional materials. In contrast to a circumstance in which the carbon atom is connected to the rest of the molecule by a double bond, the CH_2 bond or methylene may be found at 1341.79 cm^{-1}. At 1581.37 cm^{-1}, the molecular structure was methanide (CH_3). Methanide is a methyl group which contains one carbon atom bonded to three hydrogen atoms. In the 2377.56 cm^{-1} spectrum, there were two bonds which is C ≡ N and C ≡ C. On the spectrum under reading 2527.61 cm^{-1}, there were has S-H bonds and on the 2926.91 cm^{-1} has CH_3 and CH_2.

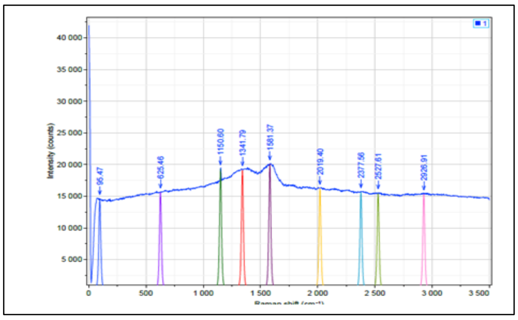

Figure 9: Raman spectrum of Peat soil

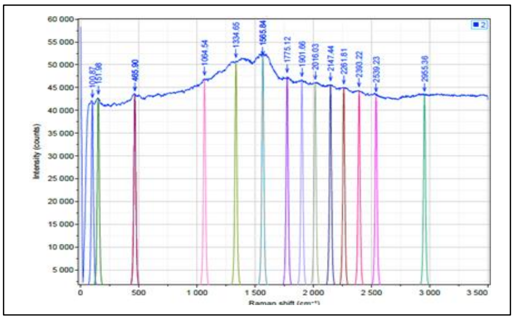

Figure 10: Raman spectrum of Keranji soil

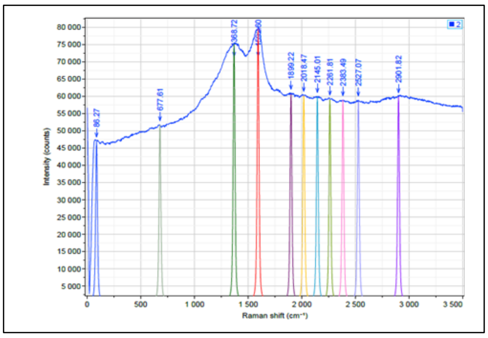

Figure 11: Raman spectrum of Kulai- Yong Peng soil

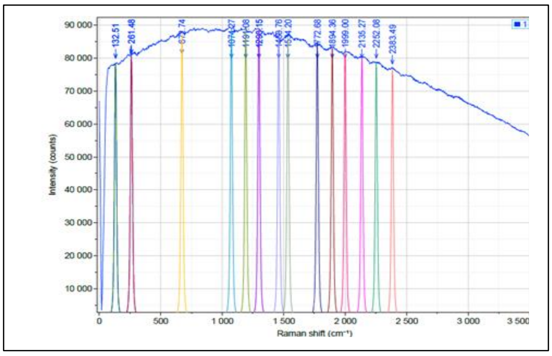

Figure 12: Raman spectrum of Rengam-Jerangau soil

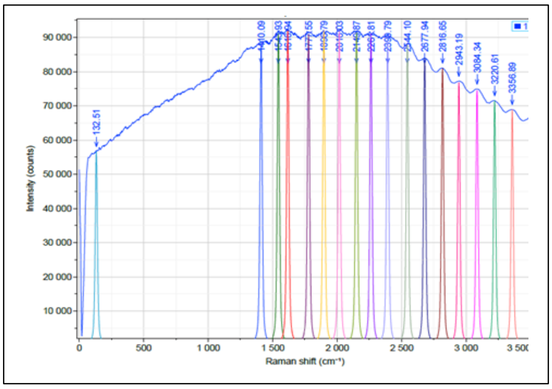

Figure 13: Raman spectrum of Selangor- Kangkong soil

Figure 4.10 shows the spectrum of Keranji soil that was examined in the Raman spectroscopy. The first molecular structure that can be found at 1334.65 cm^{-1} is the C-O-C bond. C-O-C bond is known as ether, which are good solvents for other organic compounds because of their low reactivity. The functional group at point 1775.12 cm^{-1} is alkene (C = C) and ketone (C = O). Alkenes are a kind of hydrocarbon that solely contains carbon and hydrogen, whereas ketones are organic compounds that have a carbonyl molecular structure and the carbon atom of this group has two remaining bonds that can be filled with hydrogen, alkyl, or aryl substituents. The last molecular structure can be found is CH_3 and CH_2.

There were several molecular structures in the spectrum of Kulai- Yong Peng soil in Figure 4.11. The first molecular structure is S-S bond which is disulfide bond. The second bond at 1368.72 cm-1 is C-O-C bond or called ether. Second bond at 1899.22 cm-1 is the C = O bond which is ketone. Next, at 2261.81 cm-1 has alkene (C ≡ C) while at 2527.07 cm^{-1} has S-H bond. The last bond at 2901.82 cm^{-1} is CH_3 and CH_2.

From Figure 4.12, the first molecular structure at reading 1074.27 cm-1 is Ph-H bond while the second functional group at peak 1191.08 cm-1 is C-O-C bond. Other than that, at peak 1772.68 cm^{-1} and 1894.36 cm^{-1} has C = C and C= O bonds. The last molecular structure is at peak 2252.08 cm^{-1}. Figure 4.13 shows the spectrum and peak of Selangor- KangKong. The first molecular structure group at peak 1616.94 cm^{-1} has C = N and C = C bonds. At peak 1896.79 cm^{-1} has C = O bond meanwhile at peak 2261.81 has two bonds which is C ≡ N and C ≡ C. Next, at peak 2544.10 cm-1 has S-H bond while at peak 2816.65 cm^{-1} and 2913.49 cm^{-1} has CH_3 and CH_2 bond.

4. Conclusion

To sum up, in this research the EDX analysis revealed that peat soil samples had a similar elemental composition to soil samples and carbon is the main component of soil organic matter. Other than that, the peak of FTIR from all soil species, show the same result of functional group and has good agreement with the chemical compound of soil. Next, Raman spectroscopy revealed that Keranji soil was rich in organic molecules, which are good solvents for other organic compounds because of their low reactivity. Last but not least, XRD diffractograms show the crystallography of soil particles. The higher peak from Keranji soil and Peat soil is Quartz low and Sicklerite, respectively.

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