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Comparative Soil Analysis by Scanning Electron Microscope: A Forensic Perspective

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ABSTRACT: Soil is a mineral that is found on the surface of this planet which is unconsolidated and is the final product caused by organisms, parent material, change in climate and relief acting together over time. It contains minerals, organic matter, liquids and gases. There are various ranges due to the effects of flora and fauna and the effects of wind, rain and the sun. Soil varies in more than just one property such as texture, porosity, morphology, color, temperature etc. (1) This report investigates the morphology and elemental composition of soils from Canterbury (UK), Dubai (UAE), Kerala (India). These soil samples were studied under a scanning electron microscope after heating at a certain temperature in order to remove moisture, which in turn was a challenge as to make sure none of the samples burned as each soil sample had different water retention capacities. A general comparison is done among each of these soils. We can see the difference in their texture, color, composition etc. The back scattered images of each of these soils are shown in this report along with the elemental composition from specific areas in the sample to the bulk analysis of the sample. An elemental sum spectrum is also taken to show exactly which of the components dominate in the soil sample and which of them are found in minute traces. Each of these samples slightly vary from each other as they come from different parts of the world. We observe a difference in their elemental composition and also we can see that there is a difference in their texture, particle size and color. This investigation helps us to see the difference in soils from different parts of the world, how they vary in size, shape, color and composition. This investigation helps us to see the difference in soils from different parts of the world, how they vary in size, shape, color and composition. The results proved that the method could be used in forensic cases and provide new insights into the forensic analysis of soil samples.

Keywords: Aluminum, EDX, SEM, Silicon Soil.

Abbreviations: SEM, scanning electron microscope; EDX energy – dispersive x-ray, SDD – silicon drift detector; BSE back scattered electron.

I. INTRODUCTION

Soils vary and have a couple of characteristics as an aftereffect of the regular which are altered due to its impact and trades made by us people and animals alike. So the assessment of soil isn't just the assessment of rocks, minerals, vegetation, and animal waste, it is also the study of different particles from designed composts, components like nitrates, phosphates and sulfate etc. which will help us narrow down the type of to a particular region [1]. There are various methods and systems which help us identify these components which can help us separate and isolate the soil samples from one another [2].

Composition of soil is an essential part of nutrient administration. While soil minerals and natural matter hold and store the nutrients, soil water is the thing that promptly gives nutrients for plant uptake.

The basic components of soil consists of water which would be approximately around 25% of the soil, then water which would again contain about 25% of the soil, organic matter which is the least in the soil i.e. 5% and finally about 45% of minerals. This is a general approximation of the percentages of each of these components that is present in the soil [3].

The color of the soil can differ from one soil to the other. This depends on the amount of organic matter, the oxidation degree and also due to drainage conditions. The colour of the soil can be due to the various iron minerals found in the soil. These changes in the colour can be due to chemical or biological weathering processes. Mostly the colour in soils range from yellow-red to brown and black. The yellow –red color in the soil can be due to the presence of iron in the soil, the decomposition of organic matter can lead to brown or black compounds and the presence of sulfur, manganese and nitrogen could lead to the formation of black deposits in the soil. These pigments can cause a change in the soil colour with the help aerobic and anaerobic reactions that occur in the soil [4].

The soil contains many elements and nutrients such as Silicon, Aluminium, Nitrogen, Phosphorus, Oxygen, Potassium, Calcium, Sulfur, Iron, Titanium, Manganese, Chlorine, Chromium, Zinc, Copper etc. Out of these elements few of these are very useful for the growth of vegetation. Calcium, phosphorus, nitrogen, potassium, sulfur, magnesium etc. are considered to be essential for the growth of plants or to enhance its growth [4].

Soils are significant evidence that can set up or reject the connection between a suspect, casualty, or an article at a specific scene, which could add to building a case [5]. The accessibility of using a scanning electron microscope has been so easy nowadays that it is being

widely used to study many minute traces, to identify its morphology, understand what maybe in the sample. Soil was analyzed using the SEM analysis program, then the minerals in the soil samples were identified from their chemical components [6].
This experiment is done to investigate soils. The soil samples are from Canterbury (UK), Kerala (India), Dubai (UAE). The reason this experiment was chosen was to see the difference in the morphology of each of these soil samples under a scanning electron microscope and also learn their elemental composition and study the differences found in each of the soil samples. This experiment helps us to understand the difference in texture, size, and elemental composition among the soil samples taken from three different countries.

II. LITERATURE REVIEW

Different experiments using a scanning electron microscope have been done for the study of soils as scanning electron microscope is widely used to analyse the micro structures of the soils.
Sonja Brodowski did a study on carbon in soil particles were viewed under a scanning electron microscope (SEM) and an energy dispersive x-ray (EDX) spectrometer [7]. It was found that this device was useful to examine the black carbon morphology and composition. It concluded that black carbon had a variety of surface properties due to the fact that it had both (i) distinctive inception discovered chiefly from coal combustion and vegetation fires, and also (ii) partial oxidation at harsh surface components.
Cengiz *et al.*, (2014) did a study on 17 soil samples collected around Istanbul. The purpose of this study was to show how 9 tonnes/cm^2 of pressure on soil would affect the elemental composition of soil by using SEM/EDX [2].
The samples were heated at 100-200 degrees and then placed on a stub after it was sieved. 100-150mg of the sample then was placed under a 9tonnes/cm^2 pressure by a KBr disk preparation apparatus. They concluded by saying that the sample under pressure had a gentler matrix which thus brought about the homogenization of the sample and the elemental composition of the soil sample had lesser standard deviation which implied more separation intensity on the pressed samples.
In an another study conducted by Gilkes *et al.*, (1980), samples were taken from a granitic parent material which is adamellite, along with saprolite and the pallid zone of 3 bauxitic- laterite profiles from a railway path in the West of Australia [8]. The sample was divided and one of these portions was used for examination under the Scanning electron microscope. Each of the mineral samples and fracture surfaces of the clods were coated with gold prior to the examination in the Philips Scanning Electron Microscope. The results were found to be that SEM examination of single grains and broken clods uncovered a vast assortment of morphologies. Single fracture surfaces normally contain different blends of quartz, feldspar, biotite and magnetite, kaolinite, halloysite, and gibbsite. These patterns for the most part mirror the diverse weathering results of feldspar.

III. MATERIALS AND METHODS

Each of the soil samples were collected from garden or parks in each of the countries. The soil from Canterbury was collected just outside the Darwin Houses rose garden in University of Kent. The soil from Kerala was collected from a garden of a house and finally the soil from Dubai was collected from Safa Park. Each of the soils were collected with gloves and put into a plastic bag.
The soils varied in colour and texture. The soil from Kerala was a bright red-orange and were in clumps. The soil from Dubai was a dark brown and was dry in nature. The soil from Canterbury was dark brown with more of a clay like texture.

Preparation of Soil sample

– Each of the soils were taken one by one by a clean spatula and put onto a small piece of foil paper with the use of gloves in order to avoid any kind of contamination.
– These foil paper containing each of the samples were then placed into an oven at a temperature of 50°C -80°C in order to remove any moisture content that may have been present in the soil. This is an appropriate temperature as increase in the temperature may lead to burning of the sample.
– Once it is taken from the oven, these foil papers containing each of the soil samples are placed between two glass slides. These are then slowly grinded with the help of these glass slides to get a finer powder form of the sample.
– Each of these samples are once again placed into the oven for another hour.
– Once taken out of the oven each of these samples are placed into tiny containers and labelled accordingly.
– Each sample is taken from their respective container and mounted onto a stub using different pairs of gloves and spatula.
– These stubs are first cleaned and a piece of carbon tape is stuck on top in order to hold the sample.
– One by one a small amounts of each of the soil sample is mounted onto the stubs.

– The height and width of the stub is measured before placing it into the Scanning electron microscope and it was considered to be +1mm in height and 15mm in width.
– These measurements are fed onto the computer and the sample stage is adjusted based on these measurements before closing the Scanning electron microscope. The machine used here is the Hitachi

S3400 SEM with an Oxford Instrument 'X-MAX' 80 mm^2 SDD.

– The accelerating voltage is 20kV and the variable pressure is 100Pa.

– Each of the soils bulk analysis image was taken at a magnification of x27.

– A region of 2.05mm/2mm was used for further analysis of each of samples.

– Two areas were studied from each of the samples with a magnification of x100 and an emission current ranging from 6.9µA – 7.1µA.

– Features which stood out in the whole sample were also studied.

– The elemental composition from each of these areas and features were also studied by clicking different points on the back scattered image in order to get an elemental spectrum.

– Finally an elemental mapping was done for all the soil samples.

IV. RESULTS AND DISCUSSIONS

A. Canterbury Soil

Fig. 1 shows us the overall back scattered image of the Canterbury soil sample. As seen in the image, we can observe that the particles contain powdered particles along with small pieces of grains. They seem to have many aggregates.

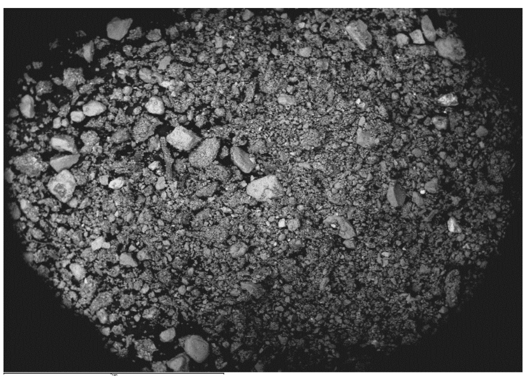

Fig. 1. Full Backscattered Image of Canterbury Soil sample.

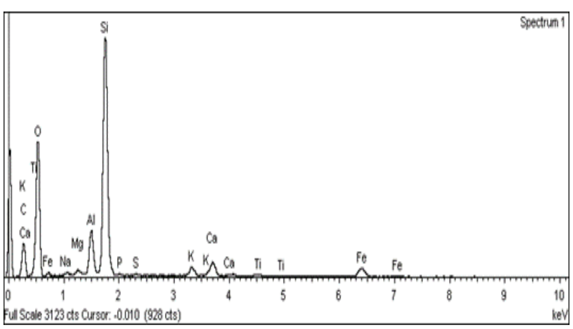

Fig. 2. Bulk Spectrum of Canterbury Soil.

Fig. 2 shows us the bulk spectrum of the soil. We can see from this spectrum that the soil from Canterbury is Silicon dominated and contains other elements like Aluminium, Oxygen, Magnesium, Calcium, Titanium, Iron, Potassium and Calcium from moderate to mild traces.

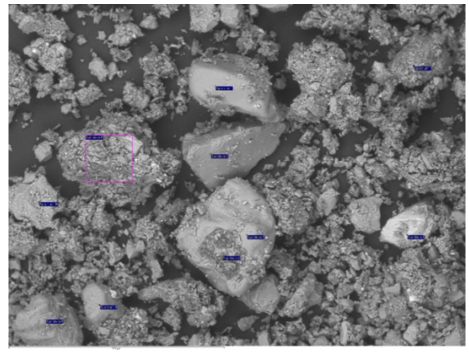

Fig. 3. Back scattered Image of Area 1.

Fig. 3 shows us the back scattered image of a particular area in the sample. As we can see it consists of smooth grains as well as powdered aggregates.

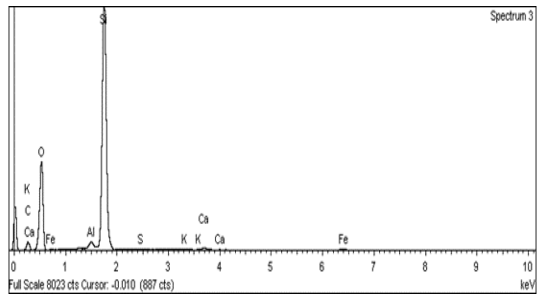

Fig. 4. Spectrum 3 of Area 1.

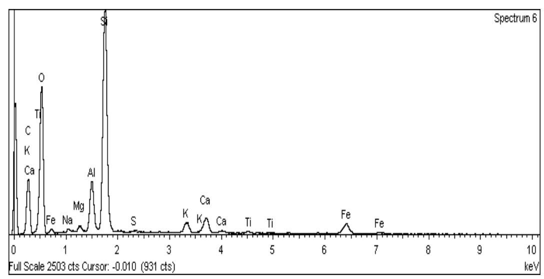

Fig. 5. Spectrum 6 of Area 1.

Fig. 4 and 5 shows us the spectral image of two different points taken from Area 1. Fig. 4 and 5 both show us that Silicon is the main component present along with Aluminium, Oxygen, Calcium and Phosphorus. The bright parts seen in the back scattered images are found to be iron rich silicates.

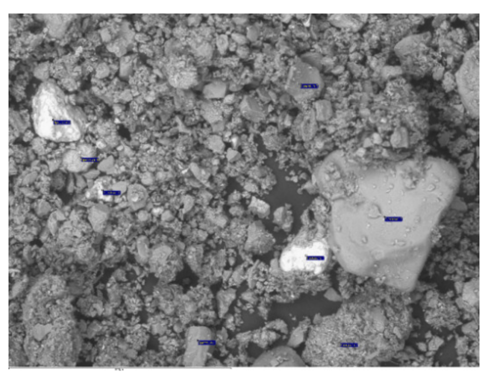

Fig. 6. Back scattered image of Area 2.

Fig. 6 shows us the back scattered image of Area 2 in the Canterbury soil. According to this image we can see that the particles are powdered and mostly aggregates with a few larger particles.

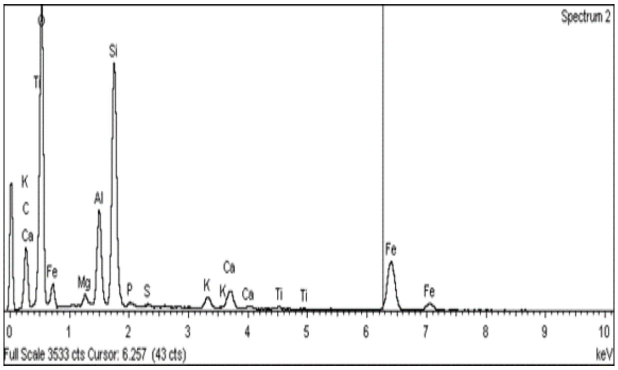

Fig. 7. Spectrum 1 of Area 2.

Fig. 7 is the spectrum of the shiny particle on the right end corner found in Fig. 6, we can see that this is a titanium rich silicate.

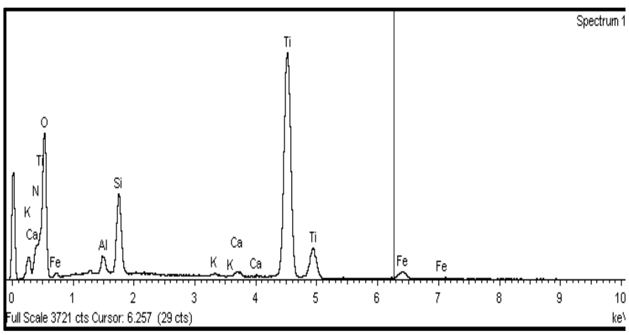

Fig. 8. Spectrum 2 of Area 3.

Fig. 8 is the spectrum of the shiny particle found on the upper left corner of Fig. 6, according to the spectral analysis we can see that this particle contains Iron mixed with particles of silicon, aluminium, calcium etc.

We observe that in this area, two different shiny particles are found with different elemental compositions. These could be the result of exhaust products.

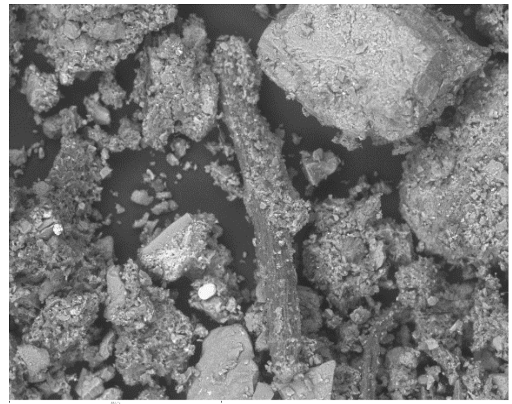

Fig. 9. TWIG.

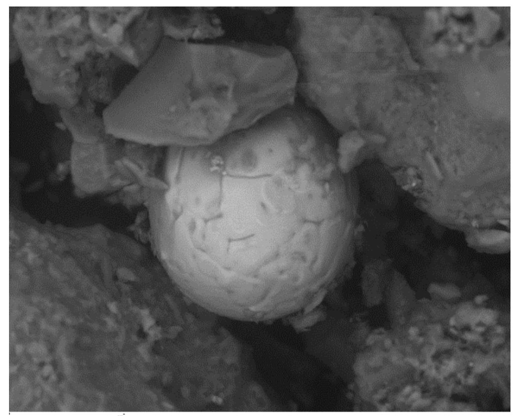

Fig. 10. Round Sphere.

Figs. 9 and 10 were two particular features which stood out in the back scattered image of the Canterbury soil sample. Fig. 9 most likely seems to contain a piece of twig covered with grains. Fig. 10 seems to be a round sphere and upon elemental analysis was found to be an Iron rich sphere. This may be a product of combustion or may be even a micro meteorite product.

Fig. 11 shows us the elemental mapping of the Canterbury soil sample. We can see that this soil is most rich in silicon. Silicon is the second most abundant element found on earth's crust. This sample contains traces of Aluminium, Magnesium, Oxygen, Titanium and Iron.

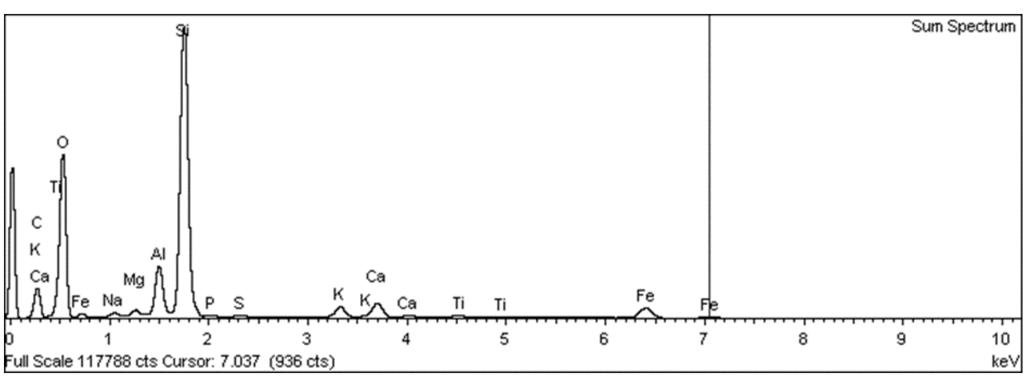

Fig. 11. Elemental Mapping of Canterbury Soil Sample.

B. Kerala Soil

Fig. 12 shows us the back scattered image of the Kerala Soil. As we can see this soil tends to contain bigger grains mixed with a bit of powdered grains. Fig. 13 shows us the bulk spectral analysis of this particular soil and we can observe that it has similar features that of the Canterbury Soil. It contains Silicon and also high amounts of Aluminium with traces of Iron, Magnesium, and Calcium etc.

Fig. 12. Full Back scattered Image of Kerala soil.

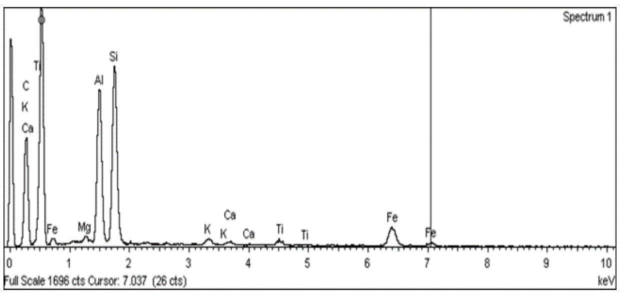

Fig. 13. Bulk Spectral analysis of Kerala Soil.

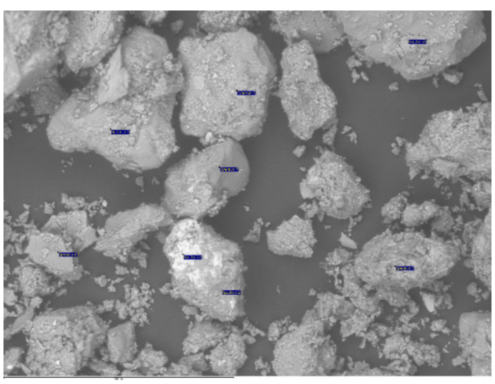

Fig. 14. Back scattered image of Area 1.

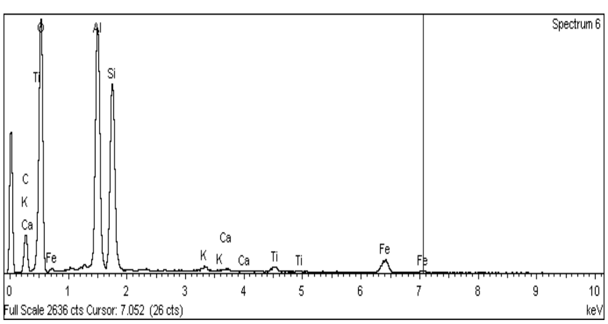

Fig. 15. Spectrum 6 of Area 1.

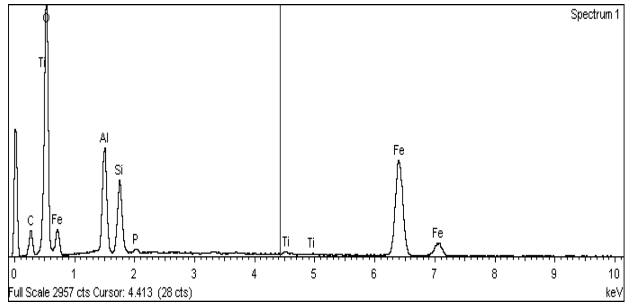

Fig. 16. Spectrum 1 of Area 1.

Figs. 15 and 16 show us the spectrums taken from Area 2. These spectrums show us that this area contains more amount of Aluminium and Silicon. The bright spots seem to be Iron rich particles as seen in the spectral analysis. Figs. 18 and 19 Spectrum show us that there are again huge differences in the amount of elements found in the same area. The bright spots found on the smooth grains which are covered with aggregates are most likely to be iron. From the above images we can see that both the spectrums taken from the same area consist of different elements. We can see that Ca found in one spectrum isn't found on the other and Mg found in spectrum 5 isn't found on spectrum 3.

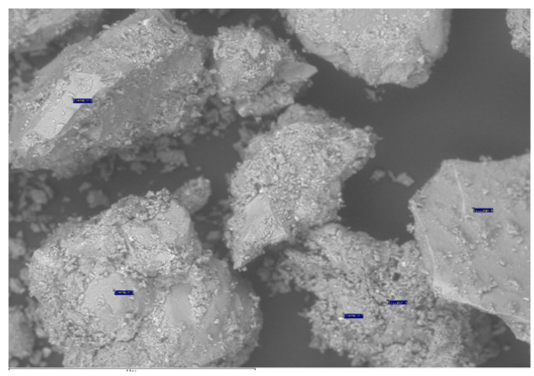

Fig. 17. Back scattered image of Area 2.

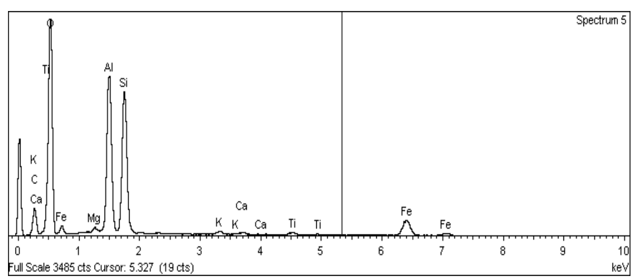

Fig. 18. Spectrum 5 of Area 2.

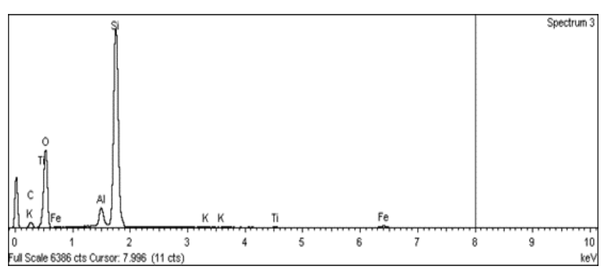

Fig. 19. Spectrum 3 of Area 2.

The aggregates in some places are found to be rich in Aluminium but in other places are found to be rich in silicon. This depends on the weathering process of the soil.

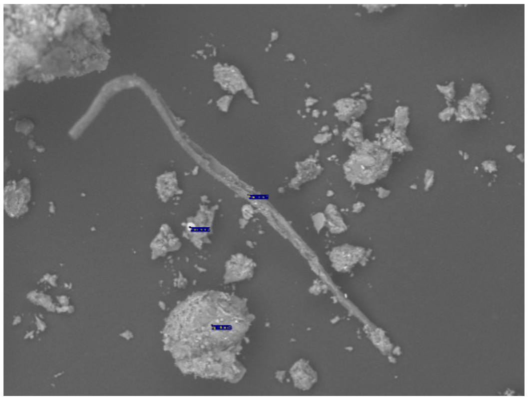

Fig. 19. Suspected Biological element.

Fig. 19 is suspected to be a biological element. Three spectrums were taken from this particular area. The long piece which is suspected to be some biological element had an elemental composition of Si, Al, Cl, K, S, Ca, etc. The bright spot on the image had an elemental composition of Ti, Mn and Fe in higher amounts with Al and Si. And finally the grainy smooth grain had an elemental composition similar to the rest of the sample; Si and Al with traces of Fe and Ti.

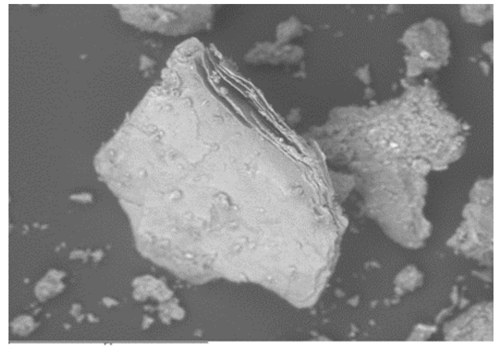

Fig. 20. Platy Mineral.

Fig. 20 is suspected to be a platy mineral. This looked different from the other minerals found on the sample. It was platy and looked layered. The elemental composition of this particular mineral were found to be slightly different from the rest of the sample as it contained high amounts of Fe, K, Ti, along with Si and Al.

Fig. 21 shows us the elemental mapping of the Kerala Soil Sample. This soil contains high levels of Silicon, Aluminium, Iron, Calcium etc. As we have seen earlier, for this particular soil the levels of aluminium and silicon differ from one area to another. In some places Silicon is high and other places Aluminium is found to be high. The bright spots found in the soil are found to be Iron rich elements.

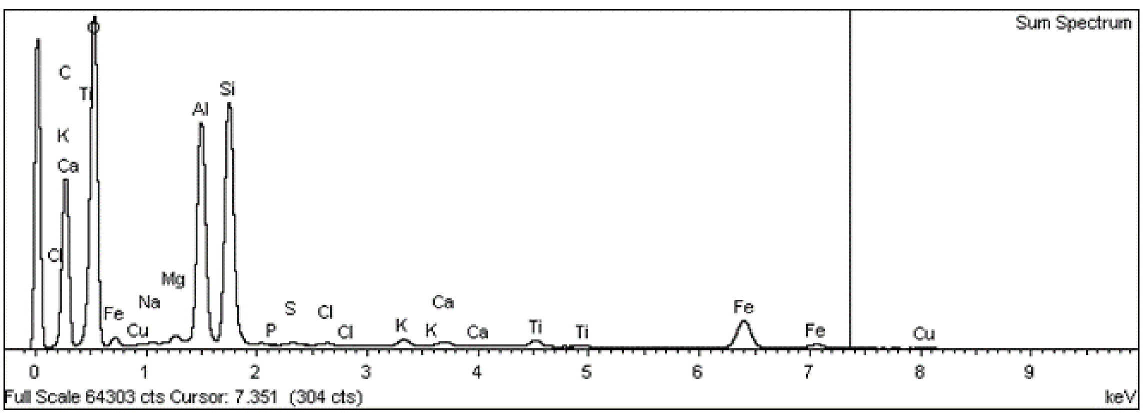

Fig. 21. Elemental Mapping of Kerala Soil Sample.

C. Dubai Soil

Fig. 22 shows us the back scattered image of the Dubai soil. It seems to be fine pebble like material.

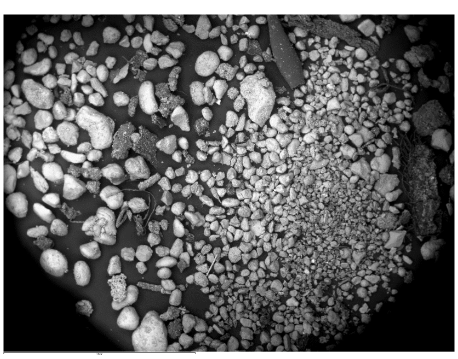

Fig. 22. Full backscattered image of Dubai soil.

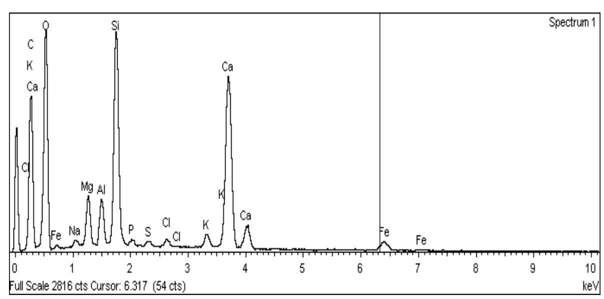

Fig. 23. Bulk analysis spectrum of Kerala soil.

There hardly seems to be any aggregates. The grains seem to be smooth and finely shaped when we look at this image. Fig. 23 shows us the bulk spectrum of the soil. It seems to contain high levels of Silicon along with calcium, oxygen and traces of Aluminium, magnesium, iron etc.

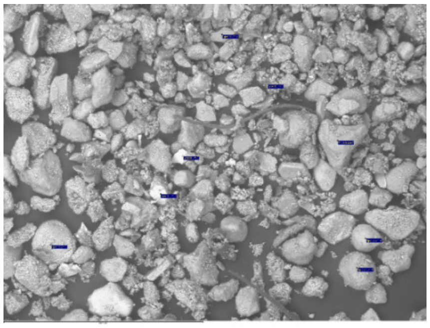

Fig. 24. Back scattered image of Area 1.

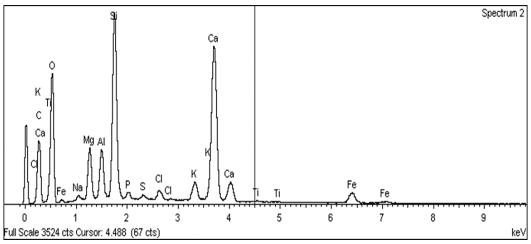

Fig. 25. Spectrum 2 of Area 1.

Fig. 24 shows us the back scattered image of Area 1. Fig. 25 shows us the elemental composition of spectrum 2 in this area. It consists of silicon, calcium, oxygen, magnesium and aluminium with few other trace elements.

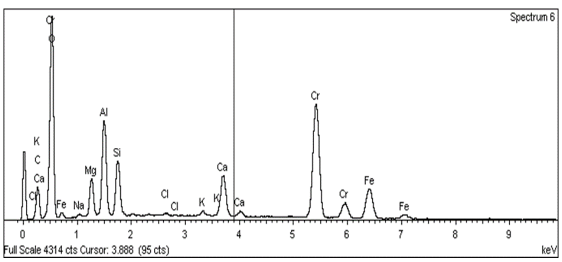

Fig. 26. Spectrum 6 of Area 1.

Fig. 26 is a spectrum taken from the bright spot found in Fig. 24. It is found to composed of mostly chromium, iron mixed with aluminium and silicon with other trace elements.

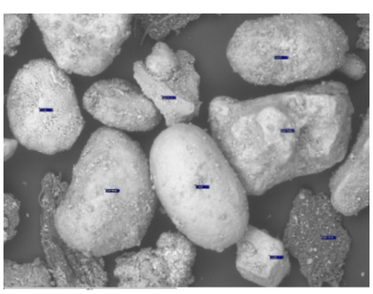

Fig. 27. Back scattered image of Area 2.

Figs. 28 and 29 shows us the spectral analysis of Area 2 in the Dubai soil. This area mainly contains of smooth grains with few powdered grains stuck on top of the smooth grains. It is found from the spectrum that the smooth grains mainly are composed of Silicon and the ones with grains on them are found to contain higher amounts of Calcium than Silicon.

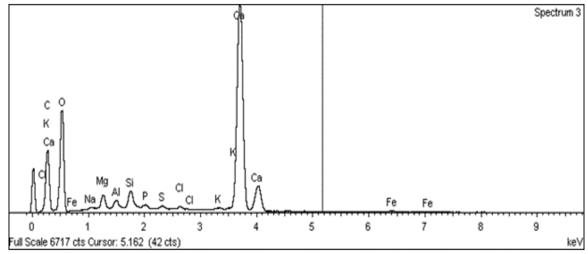

Fig. 28. Spectrum 3 of Area 2.

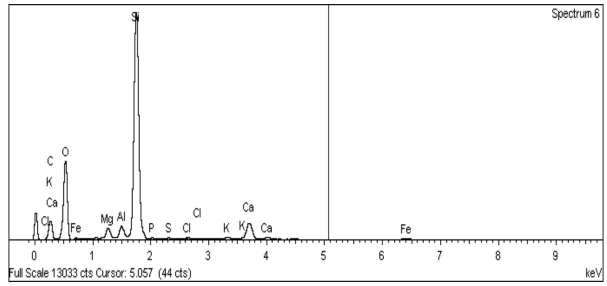

Fig. 29. Spectrum 6 of Area 3.

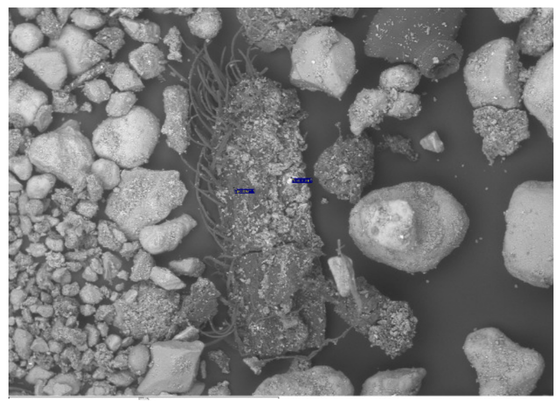

Fig. 30. Suspected Biological Element.

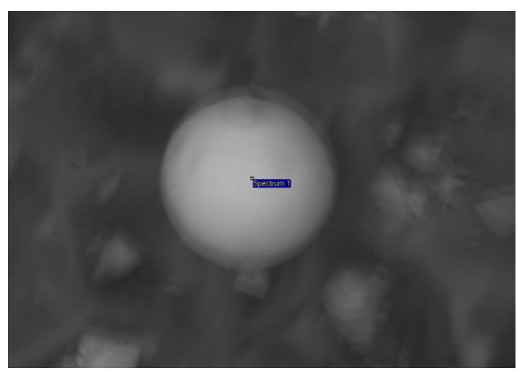

Fig. 31. Rounded Sphere.

Figs. 30 and 31 are features which stood out in the back scattered image of the Dubai soil sample. Image 30 is suspected to a biological element. The two spectrums that were taken on this biological were found to contain Ca, Si, Al, etc. and the bright spot which is the second spectrum was found to contain a high amount of Iron.
Fig. 31 is a rounded sphere. Upon elemental analysis it is found to be an Iron rich sphere. This may be a result of a combustion process.

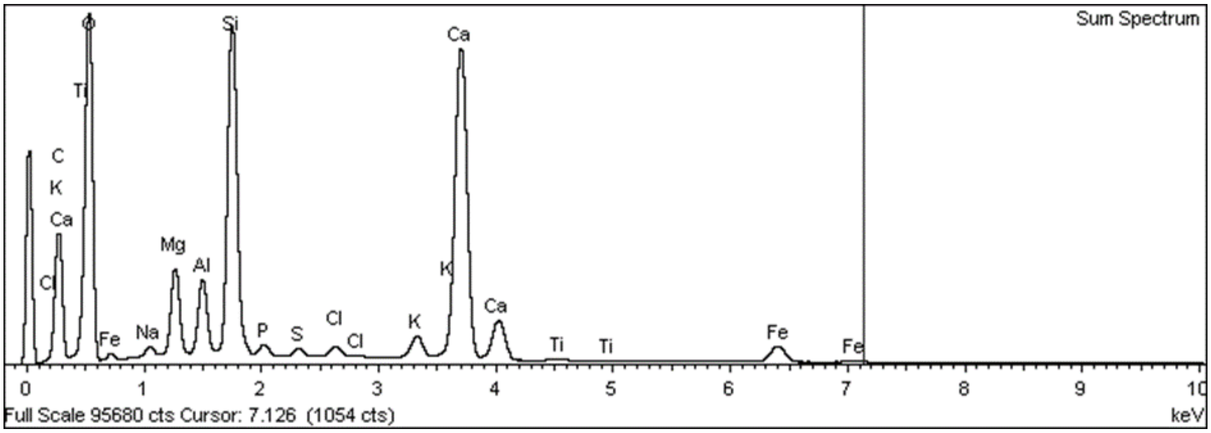

Fig. 32. Elemental Mapping of Dubai Soil.

Fig. 32 shows us the elemental mapping of the Dubai soil. As we can see it contains high levels of Silicon along with Calcium. The other trace elements that are found with this are Aluminium, Magnesium, Iron, Phosphorus, Chlorine etc.

We can see from the above images that each of these soils vary from each other. The soil from Canterbury contained aggregates mixed with larger grains and their colour was a dark brown. Upon examination under the Scanning electron microscope, we saw that they contained elements such as Silicon, Aluminium in a high amount. They also contained Iron in few places in abundance.

The soil from Kerala was found to be red in colour, and contained mostly smooth grains with a few powdered material stuck on top of smooth grains when examined under the scanning electron microscope. The elements found in them were also mainly silicon and aluminium with bits of titanium and iron in a few places. The levels of silicon and aluminium differed in different parts of the analysed sample.

The soil from Dubai was very dry in nature, when examined under the scanning electron microscope it was observed that they looked like fine pebbles with a bit of aggregates. This soil contained high amounts of Silicon and Calcium which were quite different from the other two samples. This sample also contained peculiar features which could have been biological features.

V. CONCLUSION

All the three soils that were analysed had very different texture from each other. The soil from Canterbury (UK) at first had a clay like texture but after it was dried in the oven and grinded gently between two glass slides they seemed finely powdered. But under the scanning electron microscope, they contained lots of aggregates among bigger grains. The main elements found in this soil was Silicon and Aluminium. Silicon is found to be the most abundant element on earth's crust and found in high amounts in soils. It is found that silicon helps plants when they are under stress and also improve drought tolerance and wilting in certain crops [9]. It is found in all soils, clay, sand and silt combined with oxygen and other elements like Aluminium, magnesium, iron etc. as found in this soil.

The soil from Dubai was generally dry, mostly cause of the high temperatures in this area. Under the scanning electron microscope these seemed like fine smooth pebbles. The soils found in Dubai are mostly imported from other places as it is a deserted place and contains sand more than soil. As we have seen, this soil contains high amounts of silicon and calcium. Calcium in soils helps increase their porosity. There were also few suspected biological features found in this sample which had elements such as chromium, magnesium, aluminium etc.

Finally the soil from Kerala was almost red-orange in colour. This could be due to the fact that there must be a wide diffusion of iron infused in the soil. When observed under the scanning electron microscope, they were smooth grains with few powdered particles stuck on top and scattered around. This sample contained high amounts of Aluminium along with Silicon. In different areas the levels of aluminium and silicon varied. They also had other elements like iron, titanium where bright spots were observed along with magnesium, phosphorus, in trace amounts.

All the three soils had features different from each other that were well defined when studied under the scanning electron microscope.

All the three soils had features different from each other that were well defined when studied under the scanning electron microscope. So to conclude we can say that each soil shows a particular type of characterization which makes it unique. This can be very useful in the field of forensic science in order to narrow down as to where different soil samples originate from because of their elemental composition, color, texture etc. Criminals are normally aware that their DNA, finger prints etc. can be used as evidence but they have very less knowledge as to how soil can be an identifier. Therefore it can serve as crucial evidence in most of cases [9].

VI. FUTURE PROPOSED WORK

SEM has known to be a technique to provide high resolution images along with the variations in chemical composition of the sample it analyses. It also has the ability to analyze the morphological, compositional and topographical information of the sample [10].

For future work we could collect different soil samples from different places in the world and analyse them under the scanning electron microscope and understand their morphology and their chemical components and see which of them contain high elements such as calcium, phosphorus etc. which are used to promote the growth of vegetation. We could combine the use of scanning electron microscope with other soil analysis methods for a deeper look into it.

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