

# Minerals and Elemental Status and Thermal behavior of the industrial area soil at Attur Region

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## Abstract

The soil samples were collected from various locations in an industrial area. Mineralogical identification was carried out on the soil samples using the Powder X-Ray Diffraction (XRD) method. This study gives the mineralogical composition to analyze the quartz, Kaolinite, hematite, aragonite, illite and calcite. SEM-EDX gives an insight of morphological and elemental analysis. Thermal analysis techniques are employed for the characterization and assessment of the endothermic and exothermic behavior. Results were discussed and more reliable in mineral analysis.

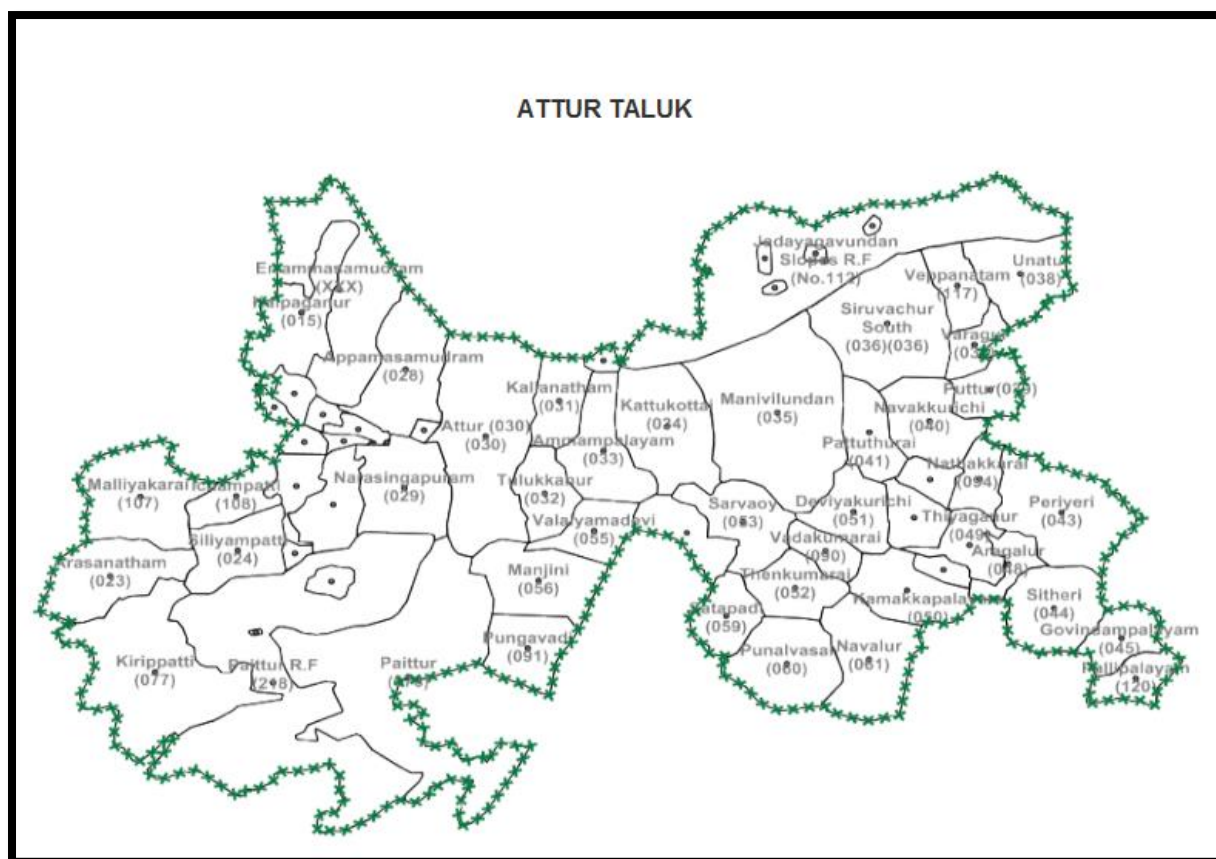
**Keywords:** Soil, XRD, TG/DTA and SEM-EDX

## 1. Introduction

Pollution is the introduction of contaminants into a natural environment that cause instability disorder, disorder to the ecosystem i.e physical systems or living organisms. Pollution denotes the presence of impurities like organic matter, minerals, alkalinity and objectionable colour, odour and taste. Although pollution is not necessarily a health hazard always. Pollution of water, air and soil environment due to industrial and other waste is one of the problems. Soil salination, one of the most common land degradation processes. X-ray diffraction studies are used for mineral analysis. XRD method is one of the mineral analysis and non-destructive. Soil varies widely in composition and physical characteristics as a function of depth, distance from land variation in soil source and the physical, chemical and biological characteristics of the environment. The SEM-EDX analysis is usually used to provide qualitative analysis. With the help of the thermal characteristics reaction such as dehydration, decomposition and transformations. The soil samples were collected, one at the surface level, second at 15cm depth, third at 30cm depth.

### 1.1 Location of Study area

The present study was concentrated in Attur taluk of Salem District, Tamilnadu, India. Which is located in north latitude between 11° 14' and 12° 53' East longitude between 77° 44' and 78° 50'. Few factories are located in and around in Attur. For the Present work the soil samples were collected from industrial area in attur region. Which is shown in Fig 1.



**Figure: 1. Sampling locations and map of study area**

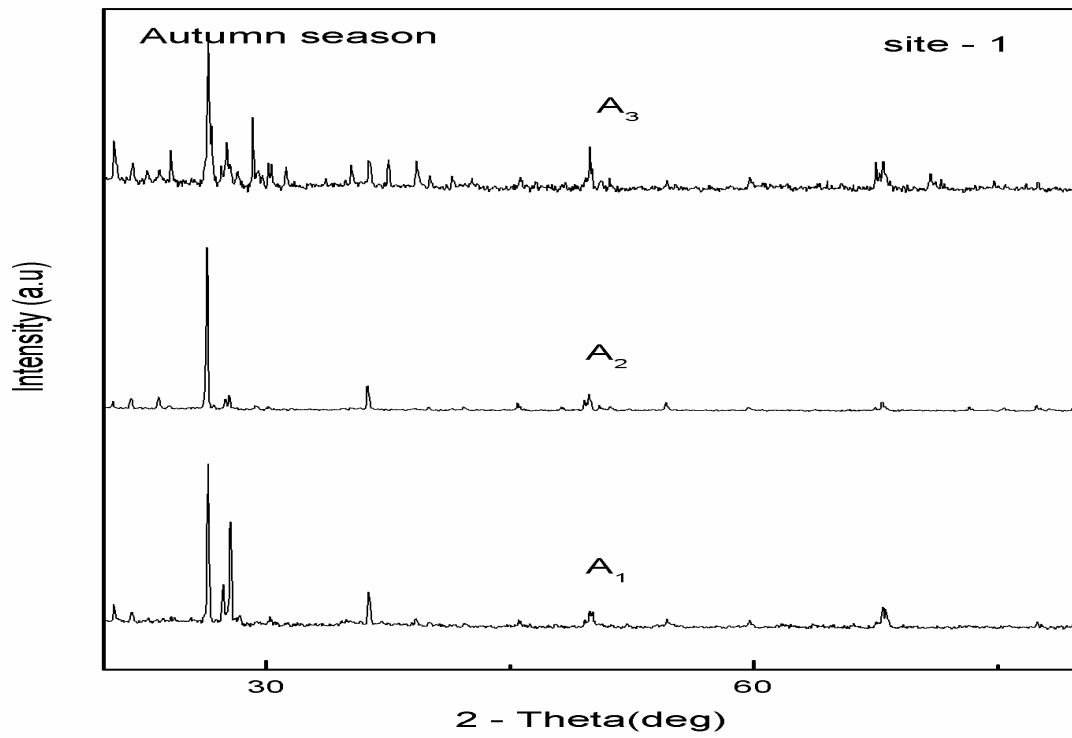
## 2. Materials and Methods

In the present study, soil samples were collected from three sites flowing around the industry located near Attur port at salem, Tamilnadu state. Using standard procedures each location is separated by distance 100m approximately. All the samples were collected in 2020. In each and every site, three samples were collected, one at the surface level, second at 15cm depth, third at 30cm depth. Nine samples were selected for the present study. Those all samples were dried at room temperature in open air for two days and stored in plastic vials. The soil samples are ground well into a powder by using an agate mortor. The soil samples were well powdered and remove the moisture content.

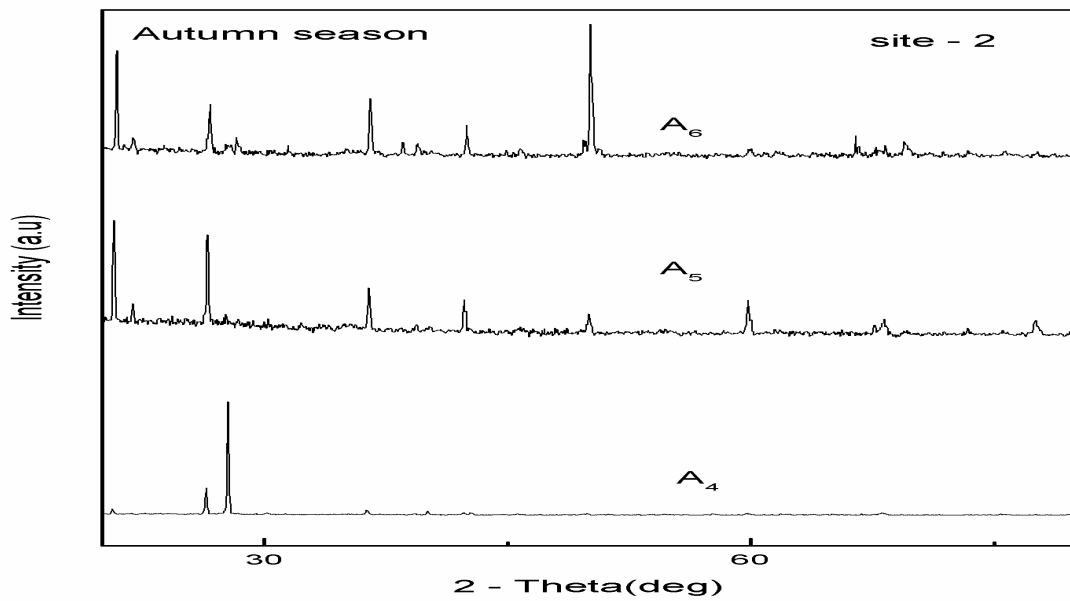
## 3. Result and Discussion

### 3.1. XRD analysis

XRD of soil samples (at the surface level, at 15 cm depth and at 30cm depth) are studied. The samples were numbered as (A<sub>1</sub> – A<sub>9</sub>). XRD studies analyze the mineral composition. The XRD soil samples in figures are shown in fig 3.1(a-c). The observed data from are given in tables 1-9. Soil samples experimental  $2\theta$  values were compared with the standard value, relative intensity and hkl values are indicate the minerals are quartz, kaolinite, hematite, aragonite and feldspar given in the



**Figure: 2. XRD pattern of soil samples at site - 1 autumn season (A<sub>1</sub> - A<sub>3</sub>)**



**Figure: 2. XRD pattern of soil samples at site - 2 autumn season (A<sub>4</sub> - A<sub>6</sub>)**

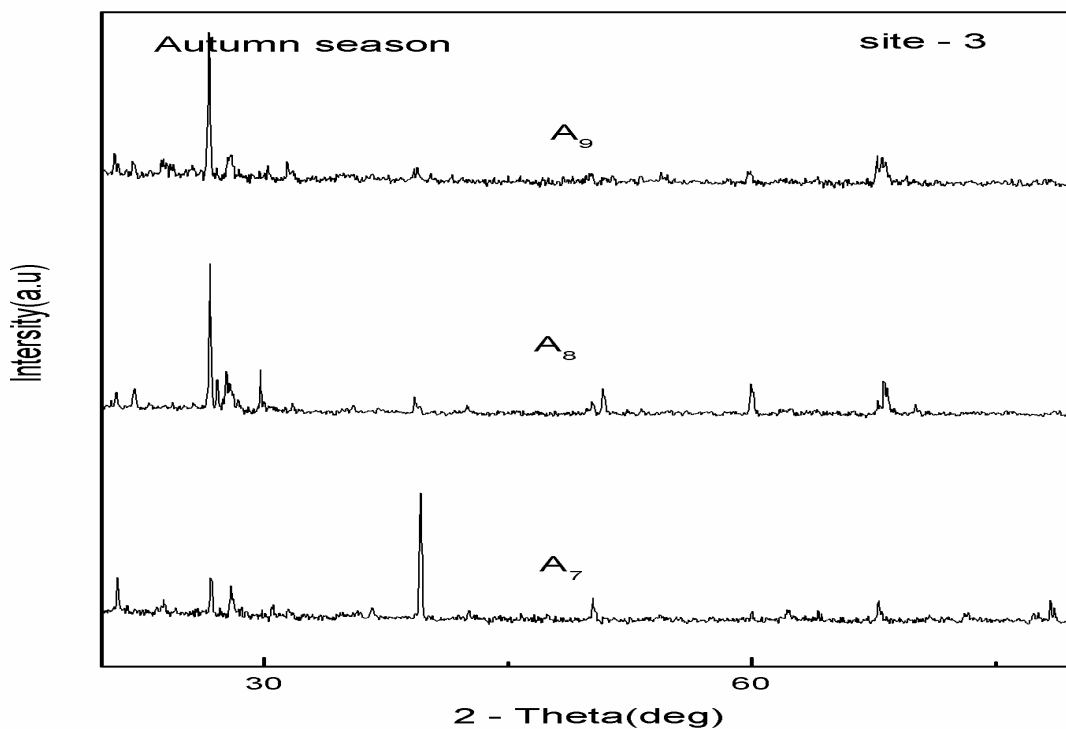


Figure: 3. XRD pattern of soil samples at site - 3 autumn season (A<sub>7</sub> - A<sub>9</sub>)

Table: 1 Comparison between observed and standard value of '2θ' for soil samples (A<sub>1</sub>)

Observed values			Standard values			hkl values	Mineral names	JCPDF number
2θ	I/I <sub>0</sub>	d- (Å <sup>0</sup> )	2θ	I/I <sub>0</sub>	d - (Å <sup>0</sup> )			
20.647	13	4.30	20.870	22	4.25	100	quartz	85 - 0930
21.756	7	4.08	21.623	17	4.10	201	feldsper	70 - 2121
26.435	100	3.37	26.696	100	3.33	101	Quartz	85 - 0865
28.376	6	3.14	28.251	76	3.15	112	kaolinite	83-0971
36.326	21	2.47	36.551	7	2.4564	110	quartz	85 - 1053
39.239	5	2.29	39.475	7	2.28	102	quartz	85 - 1053
45.553	3	1.99	45.898	3	1.97	201	quartz	85 - 1053
54.737	4	1.67	54.872	3	1.67	022	quartz	85 - 0798
67.925	13	1.37	67.766	4	1.38	212	quartz	85 - 0930

**Table: 2 Comparison between observed and standard value of '2 $\theta$ ' for soil samples (A<sub>2</sub>)**

Observed values			Standard values			hkl values	Mineral names	JCPDF number
2 $\theta$	I/I <sub>0</sub>	d - (A <sup>0</sup> )	2 $\theta$	I/I <sub>0</sub>	d - (A <sup>0</sup> )			
20.570	5	4.31	20.870	22	4.25	100	quartz	85 - 0930
21.687	6	4.09	21.623	17	4.10	201	Feldsper	70 - 2121
23.399	8	3.80	23.783	9	3.73	111	Calcite	87 - 1863
26.372	100	3.37	26.696	100	3.33	101	quartz	85 - 0865
27.765	8	3.24	27.702	78	2.97	002	Feldsper	70 - 2121
36.272	15	2.47	36.551	7	2.45	110	quartz	85 - 1053
45.515	4	1.99	45.803	3	1.97	201	quartz	85 - 1053
59.670	2	1.54	59.958	8	1.54	121	quartz	85 - 1054
67.890	6	1.38	67.766	4	1.38	212	quartz	85 - 0930

**Table: 3 Comparison between observed and standard value of '2 $\theta$ ' for soil samples (A<sub>3</sub>)**

Observed values			Standard values			hkl values	Mineral names	JCPDF number
2 $\theta$	I/I <sub>0</sub>	d - (A <sup>0</sup> )	2 $\theta$	I/I <sub>0</sub>	d - (A <sup>0</sup> )			
20.653	28	4.30	20.960	23	4.23	100	Quartz	85 - 1780
22.725	7	3.91	22.990	10	3.86	012	Calcite	86 - 2343
24.156	17	3.68	24.318	4	3.66	112	illite	29 - 1496
26.439	100	3.37	26.696	100	3.33	101	Quartz	85 - 0865
27.564	27	3.23	27.702	76	3.21	002	Feldsper	70 - 2121
29.202	29	3.05	29.065	4	3.06	112	kaolinite	89 - 5695
30.245	8	2.95	30.374	6	2.94	222	Feldsper	84 - 0710
31.242	12	2.86	31.532	6	2.83	022	kaolinite	89 - 6538
35.264	11	2.54	35.598	74	2.52	110	Hematite	89 - 2810
36.373	15	2.47	36.551	6	2.45	110	Quartz	85 - 1053
37.543	17	2.39	37.429	9	2.40	110	Quartz	83 - 2473
39.271	17	2.29	39.475	7	2.28	102	Quartz	85 - 1053
45.649	6	1.98	45.917	6	1.97	201	Quartz	85 - 0865
59.738	8	1.54	59.958	8	1.54	121	Quartz	85 - 1054
67.519	17	1.38	67.766	4	1.38	212	Quartz	85 - 0930

**Table: 4 Comparison between observed and standard value of '2θ' for soil samples (A<sub>4</sub>)**

Observed values			Standard values			hkl values	Mineral names	JCPDF number
2θ	I/I <sub>0</sub>	d - (A <sup>0</sup> )	2θ	I/I <sub>0</sub>	d - (A <sup>0</sup> )			
20.630	5	4.30	20.870	22	4.25	100	Quartz	85 - 0930
26.422	23	3.37	26.696	100	3.33	101	Quartz	85 - 0865
27.774	100	3.21	27.702	76	3.21	002	Feldsper	70 - 2121
30.154	2	2.96	30.374	6	2.94	222	Feldsper	84 - 0710
36.338	4	2.47	36.551	7	2.45	110	Quartz	85 - 1053
40.082	3	2.24	40.321	20	2.23	113	Hematite	88 - 2359
42.289	2	2.13	42.459	5	2.12	200	Quartz	85 - 1053
59.780	1	1.54	59.958	8	1.54	121	Quartz	85 - 1054
68.077	2	1.37	68.497	4	1.72	231	Aragonite	03 - 1067

**Table: 5 Comparison between observed and standard value of '2θ' for soil samples (A<sub>5</sub>)**

Observed values			Standard values			hkl values	Mineral names	JCPDF number
2θ	I/I <sub>0</sub>	d - (A <sup>0</sup> )	2θ	I/I <sub>0</sub>	d - (A <sup>0</sup> )			
20.732	100	4.28	20.870	22	4.25	100	Quartz	85 - 0930
21.889	22	4.06	21.960	23	4.23	100	Quartz	85 - 1780
26.517	88	3.36	26.696	100	3.33	101	Quartz	85 - 0865
36.454	34	2.46	36.551	7	2.45	110	Quartz	85 - 1053
42.353	22	2.13	42.459	5	2.12	200	Quartz	85 - 1053
50.060	15	1.82	50.245	1	1.81	112	Quartz	85 - 0865
59.817	31	1.54	59.958	8	1.54	121	Quartz	85 - 1054
68.192	11	1.37	68.497	4	1.72	231	Aragonite	03 - 1067

**Table: 6 Comparison between observed and standard value of '2θ' for soil samples (A<sub>6</sub>)**

Observed values			Standard values			hkl values	Mineral names	JCPDF number
2θ	I/I <sub>0</sub>	d - (A <sup>0</sup> )	2θ	I/I <sub>0</sub>	d - (A <sup>0</sup> )			
20.894	8	4.25	20.862	22	4.25	100	Quartz	85 - 1053
21.934	11	4.05	21.623	17	4.10	201	Feldsper	70 - 2121
26.665	39	3.34	26.644	100	3.34	011	Quartz	85 - 1053
27.803	6	3.20	27.702	76	3.21	022	Feldsper	70 - 2121
36.527	45	2.45	36.551	7	2.45	110	Quartz	85 - 1053
39.485	9	2.28	39.475	7	2.28	102	Quartz	85 - 1053
42.496	23	2.12	42.666	5	2.12	200	Quartz	85 - 1053
50.141	100	1.81	50.245	1	1.81	161	kaolinite	89-5695
66.431	10	1.40	66.125	3	2.06	125	Hematite	85 - 0599
68.021	4	1.37	68.497	4	1.72	231	Aragonite	03 - 1067
69.487	11	1.35	69.125	1	1.35	114	Aragonite	76 - 0606

**Table: 7 Comparison between observed and standard value of '2 $\theta$ ' for soil samples (A7)**

Observed values			Standard values			hkl values	Mineral names	JCPDF number
2 $\theta$	I/I <sub>0</sub>	d- (A <sup>0</sup> )	2 $\theta$	I/I <sub>0</sub>	d- (A <sup>0</sup> )			
20.987	30	4.23	20.870	22	4.25	100	Quartz	85 - 0930
23.675	7	3.75	23.018	10	3.86	012	Calcite	86 - 2341
26.740	32	3.33	26.696	100	3.33	101	Quartz	85 - 0865
27.937	20	3.19	27.702	76	3.21	002	Feldsper	70 - 2121
39.630	100	2.27	39.475	7	2.28	102	Quartz	85 - 1053
50.253	14	1.81	50.245	1	1.81	112	Quartz	85 - 0865
62.202	4	1.49	62.447	9	1.48	211	Quartz	85 - 0460
67.797	16	1.38	67.766	4	1.37	212	Quartz	89 - 0930

**Table: 8 Comparison between observed and standard value of '2 $\theta$ ' for soil samples (A8)**

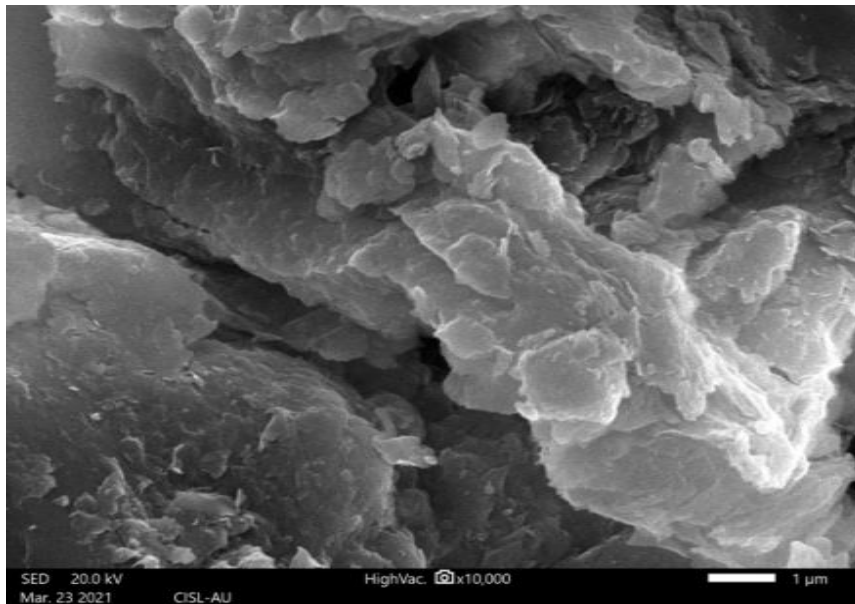
Observed values			Standard values			hkl values	Mineral names	JCPDF number
2 $\theta$	I/I <sub>0</sub>	d- (A <sup>0</sup> )	2 $\theta$	I/I <sub>0</sub>	d- (A <sup>0</sup> )			
20.887	12	4.25	20.862	22	4.25	100	Quartz	85 - 1053
22.010	14	4.03	22.205	7	4.00	201	Calcite	87 - 1780
26.681	100	3.34	26.644	100	3.34	011	Quartz	85 - 1053
27.671	27	3.28	27.702	76	2.97	002	Feldspar	70 - 2121
29.795	27	2.99	29.534	4	3.02	112	Kaolinite	89 - 5695
39.382	6	2.28	39.475	7	2.28	102	Quartz	85 - 1053
50.863	13	1.81	50.245	1	1.81	112	Quartz	85 - 0865
60.007	16	1.54	60.170	7	1.53	211	Quartz	85 - 1780
68.139	24	1.37	68.497	4	1.72	321	Aragonite	03 - 1067

**Table: 9 Comparison between observed and standard value of '2 $\theta$ ' for soil samples (A9)**

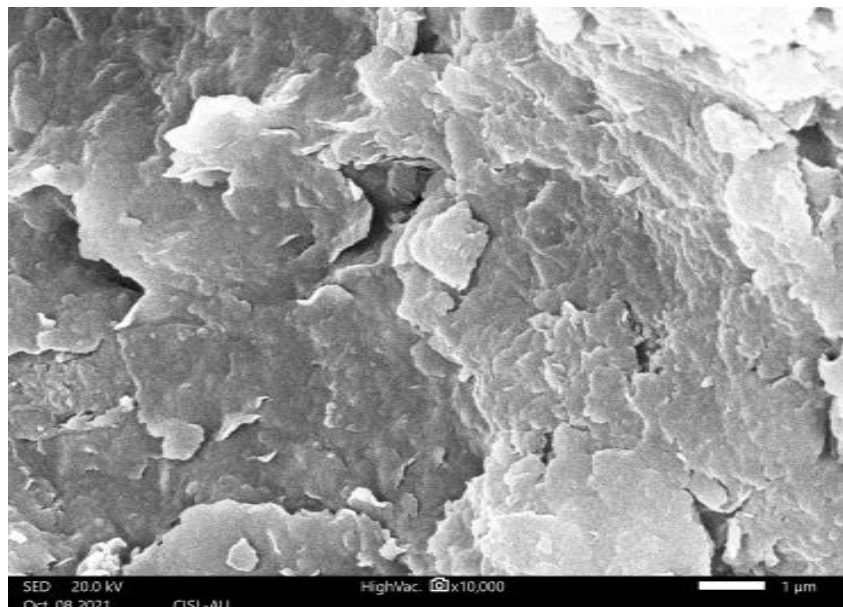
Observed values			Standard values			hkl values	Mineral names	JCPDF number
2 $\theta$	I/I <sub>0</sub>	d- (A <sup>0</sup> )	2 $\theta$	I/I <sub>0</sub>	d- (A <sup>0</sup> )			
20.841	17	4.26	20.960	23	4.23	100	Quartz	85 - 1780
24.013	8	3.70	24.318	4	3.66	112	lillte	29 - 1496
26.679	100	3.34	26.644	100	3.34	011	Quartz	85 - 1053
27.961	15	3.19	27.702	76	3.21	002	Feldspar	70 - 2121
31.510	11	2.83	31.532	6	3.09	022	kaolinite	89 - 6538
39.428	6	2.28	39.475	7	2.28	102	Quartz	85 - 1053
59.919	7	1.54	59.958	8	1.54	121	Quartz	85 - 1054
67.753	18	1.38	67.973	2	1.37	104	Aragonite	76 - 0606

### 3.3. SEM-EDX analysis

soil samples were analysed in this study using Scanning Electron Microscopy Dispersive X-ray (SEM-EDX). The SEM studies help in understanding the morphology and ultra-structure of the soil. EDX spectroscopy was employed to know the mineral element in soil. The observed results indicates that the sunflower like appearance of globular aggregates can be seen in above micrographs and the same aggregate morphology The SEM micrographs of the soils samples are (A<sub>1</sub>- A<sub>9</sub>) are nearly identical to those of soils (Figures 5 -13 (a-i)). Kaolinite particles are typically globular or spherical in shape.

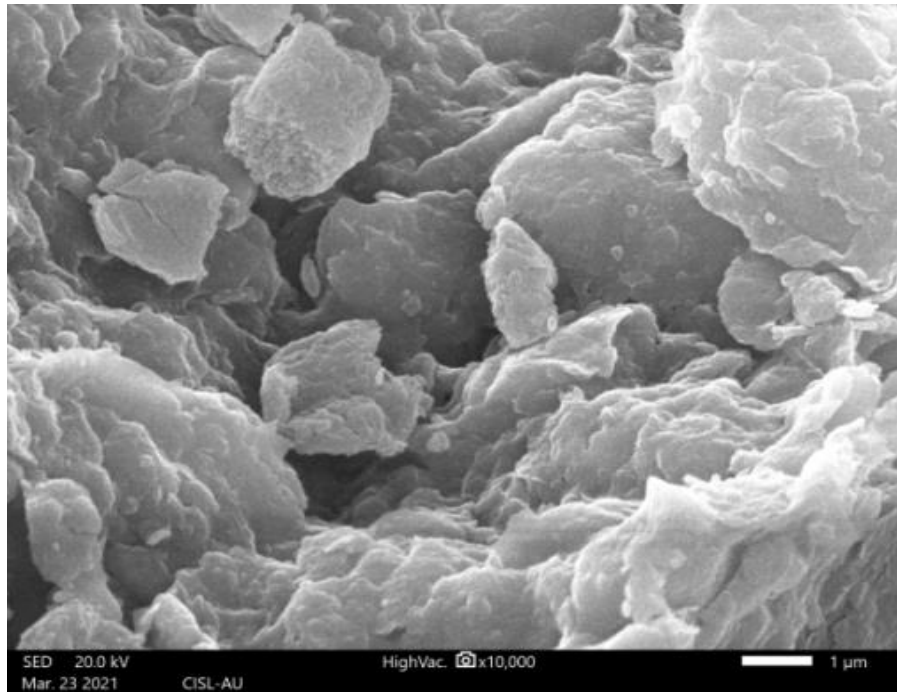


**Figure 5. SEM images of soil in autumn season from site - 1 sample (A<sub>1</sub>)**

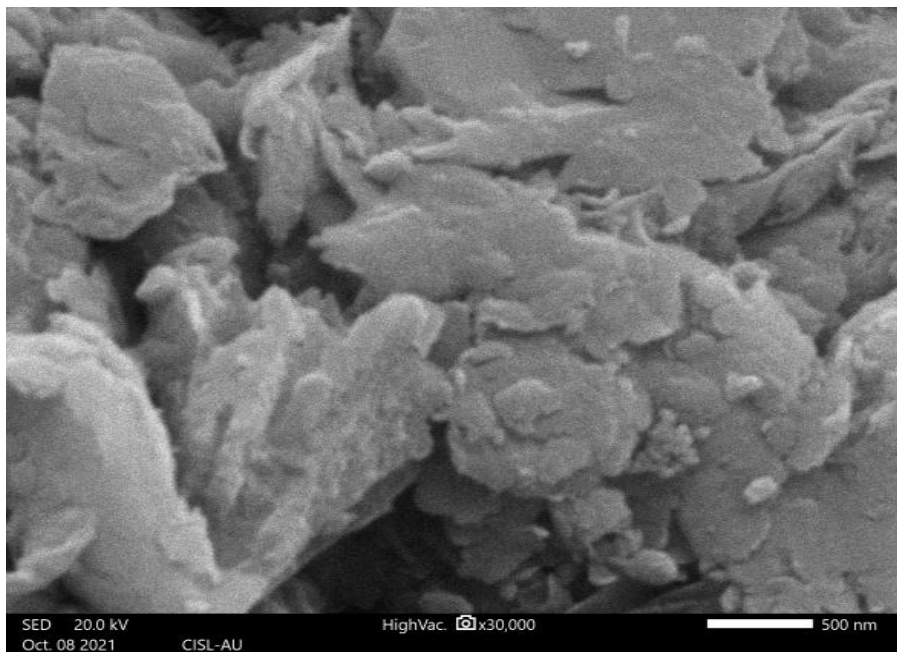


**Figure 6. SEM images of soil in autumn season from site - 1 sample (A<sub>2</sub>)**

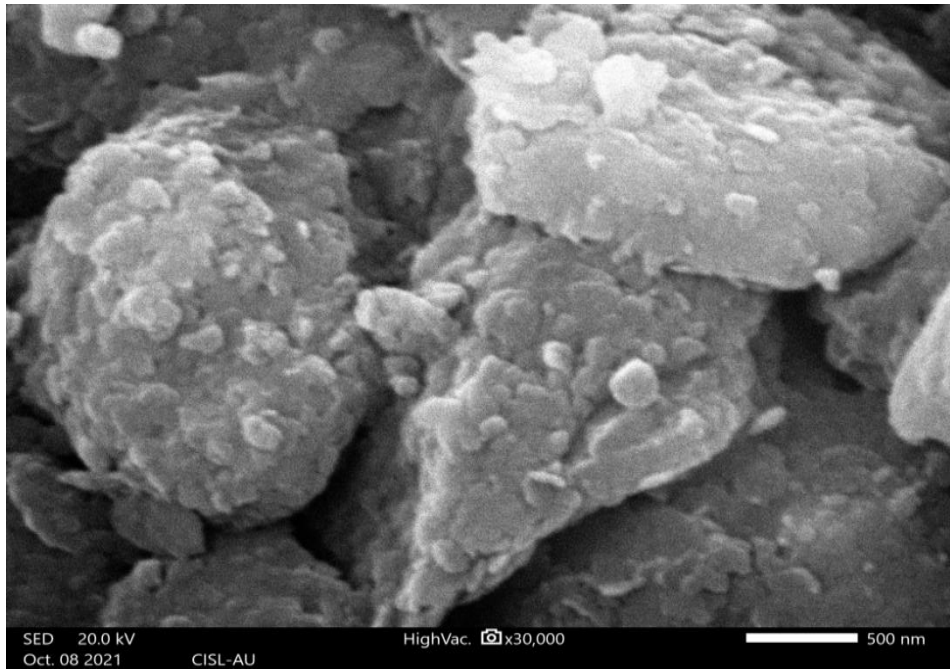




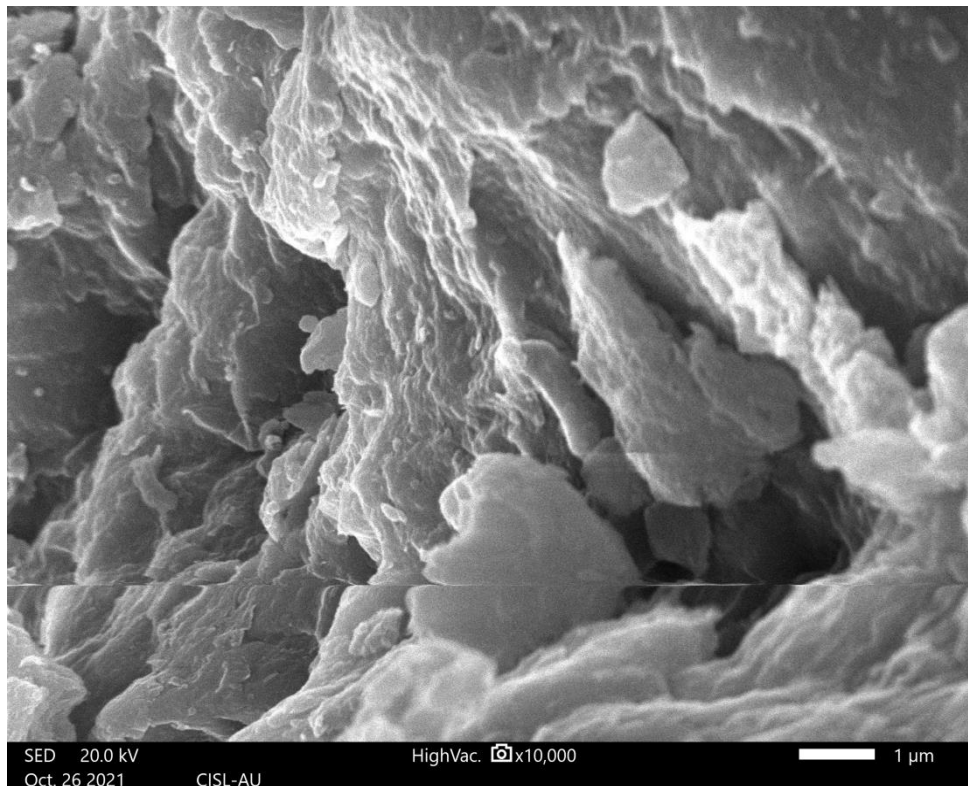
**Figure 7. SEM images of soil in autumn season from site - 1 sample (A3)**



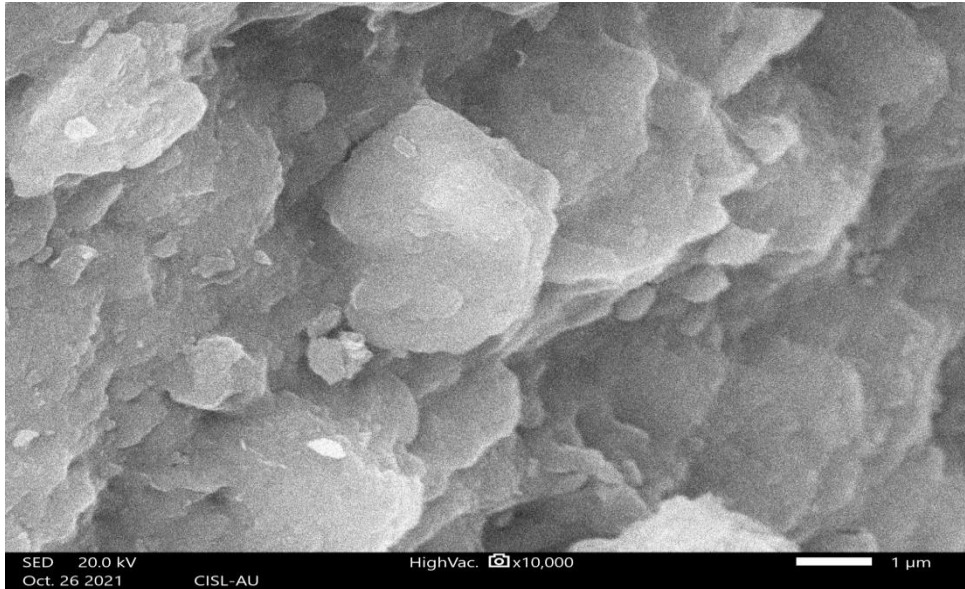
**Figure 8. SEM images of soil in autumn season from site - 2 sample (A4)**



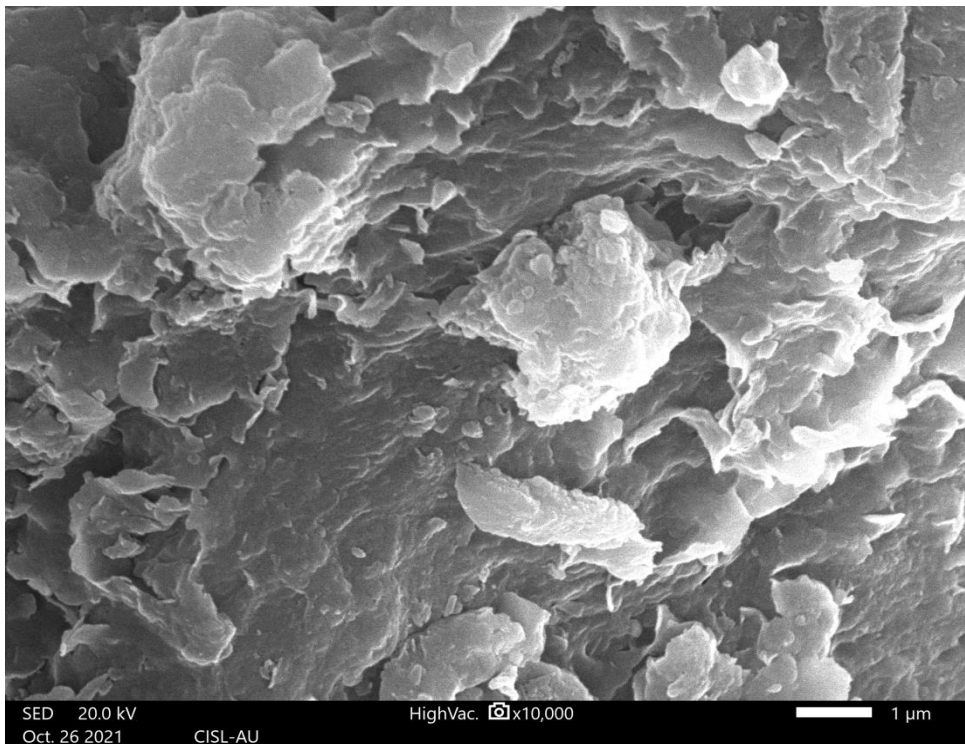
**Figure 9. SEM images of soil in autumn season from site -2 sample (A<sub>5</sub>)**



**Figure 10. SEM images of soil in autumn season from site - 2 sample (A<sub>6</sub>)**



**Figure: 11. SEM images of soil in autumn season from site - 2 sample (A7)**



**Figure 12. SEM images of soil in autumn season from site -3 sample (A8)**

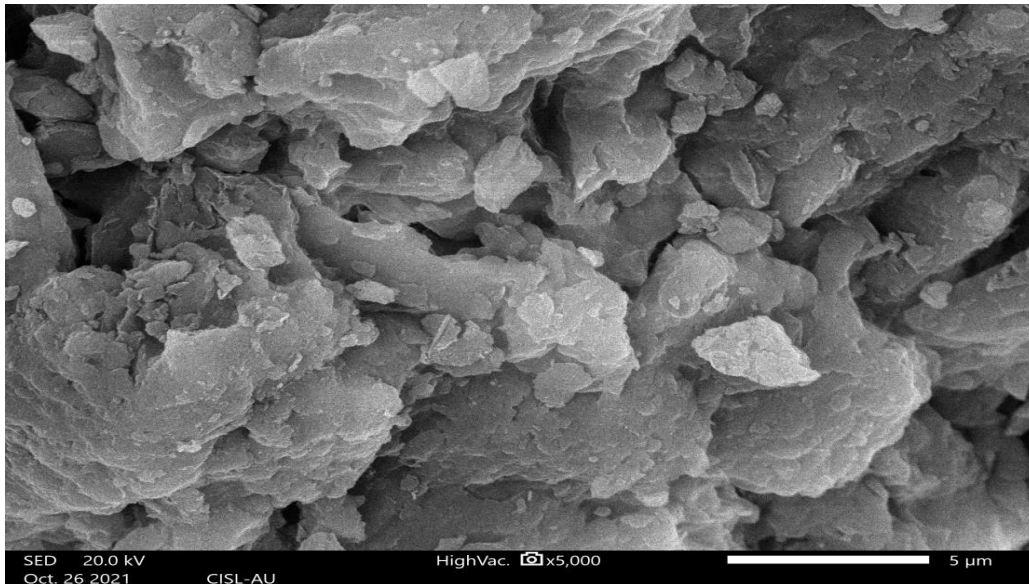


Figure: 13. SEM images of soil in autumn season from site - 3 sample (A9)

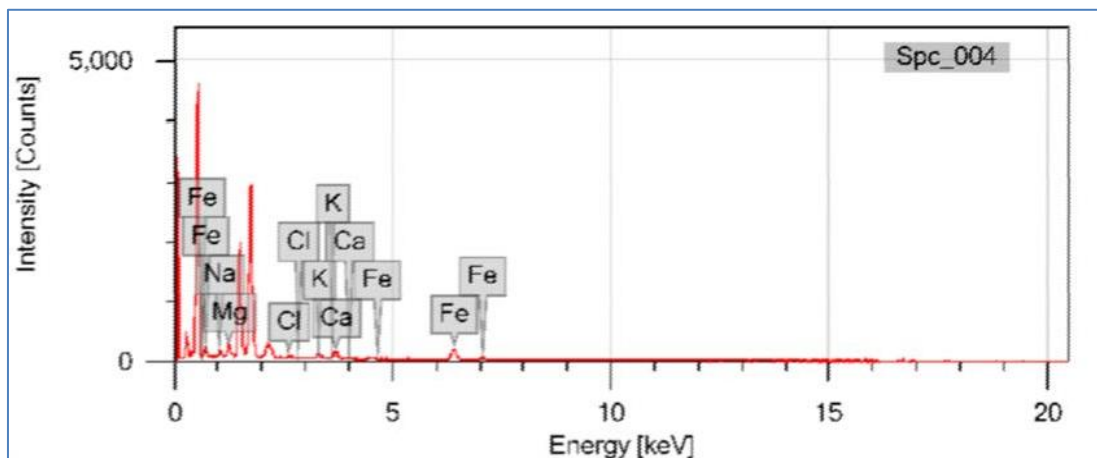


Figure:5a. EDX spectrum of soil samples at site-1 (A1)

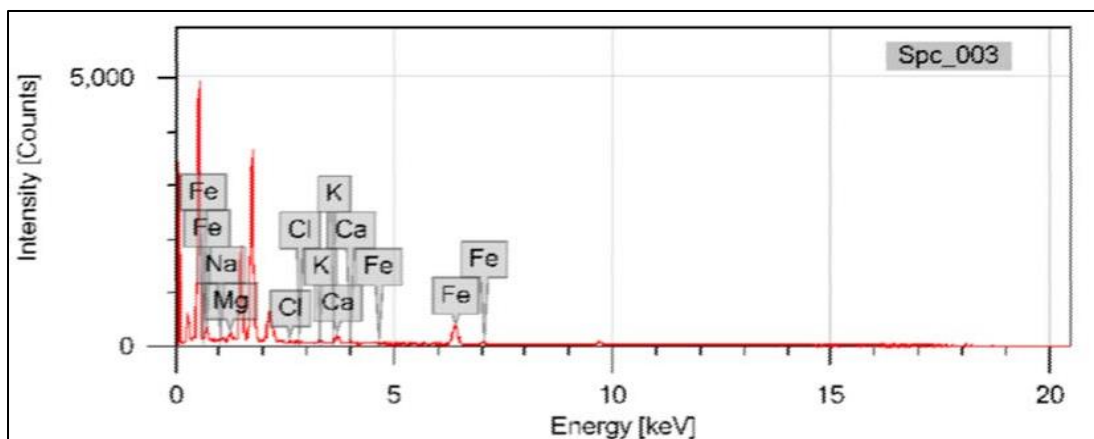


Figure:6a. EDX spectrum of soil samples at site-1 (A2)

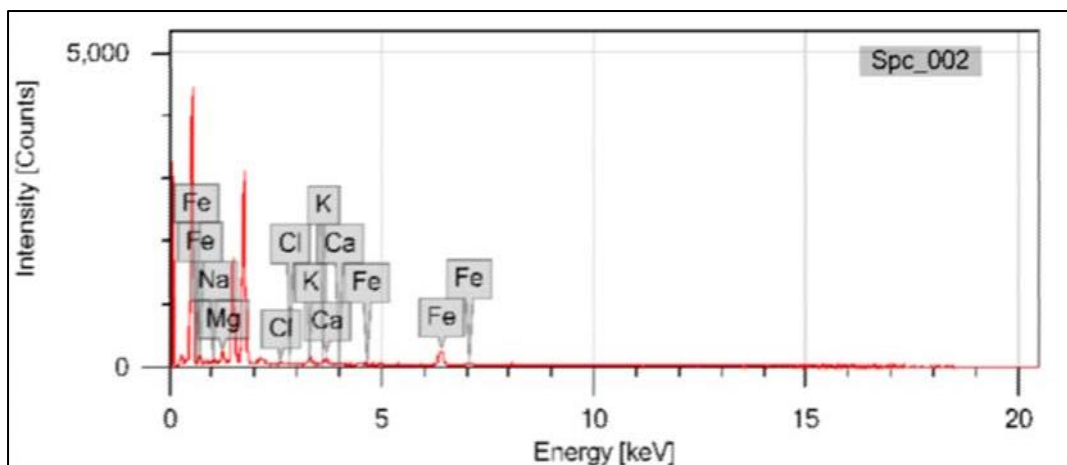


Figure:7a. EDX spectrum of soil samples at site-1 (A3)

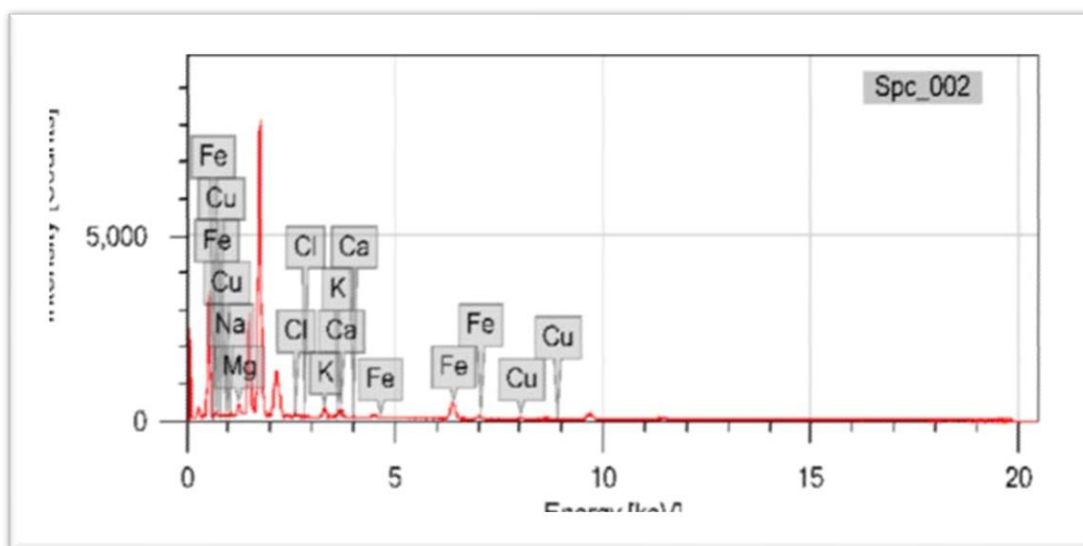


Figure 8a. EDX spectrum of soil samples at site- 2 (A4)

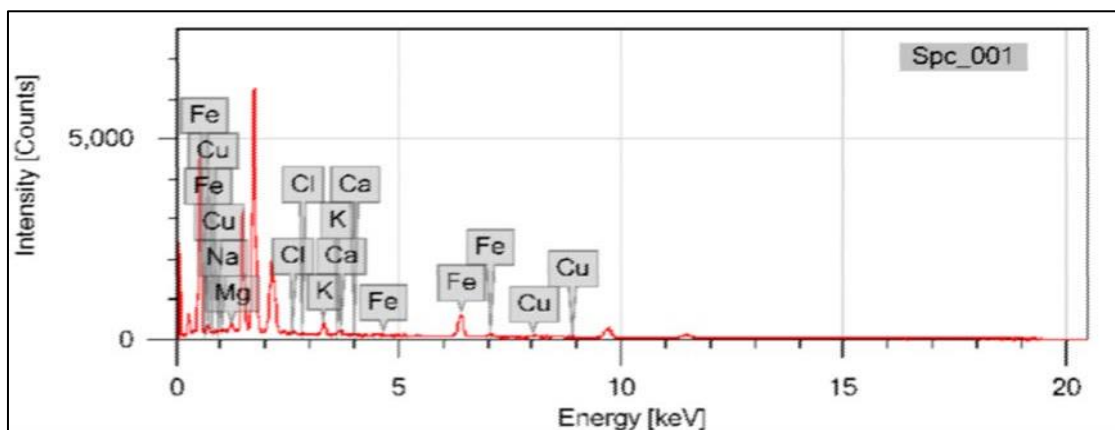


Figure 9a. EDX spectrum of soil samples at site -2 (A5)

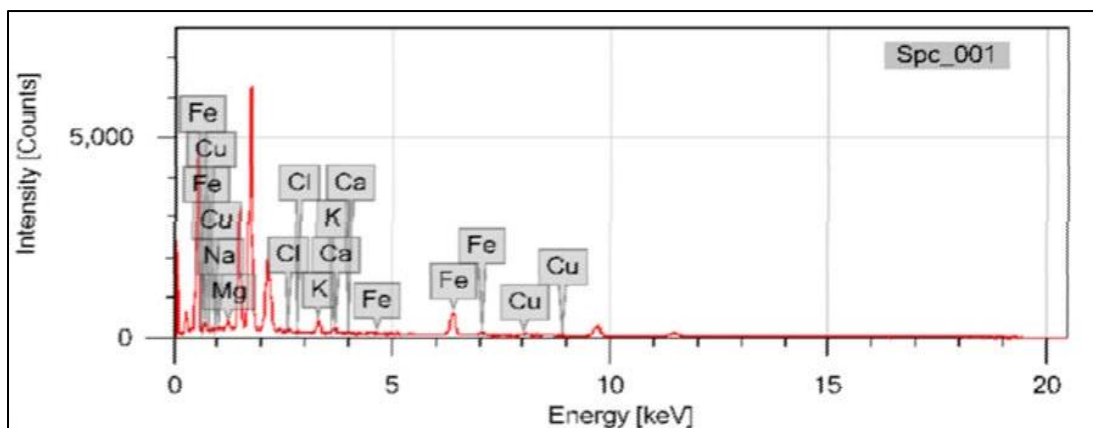


Figure 10a. EDX spectrum of soil samples at site - 2 (A6)

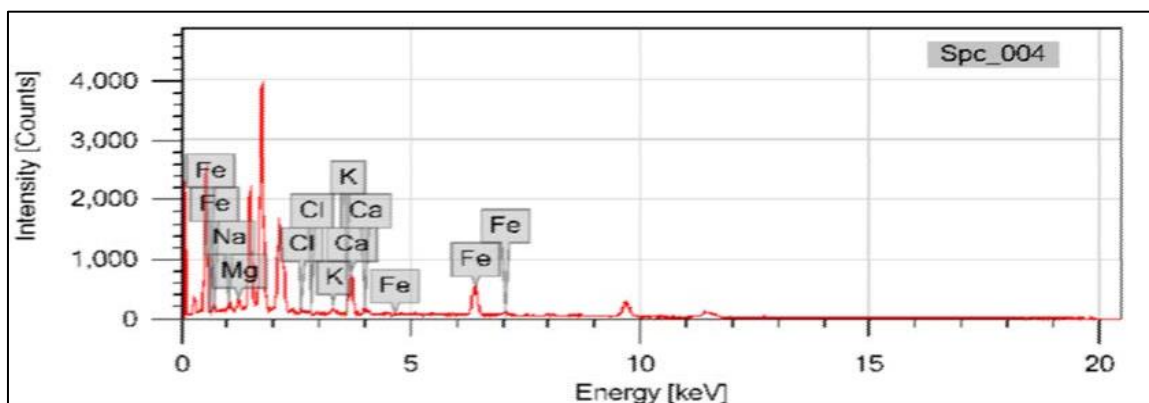


Figure 11a. EDX spectrum of soil samples at site -3 (A7)

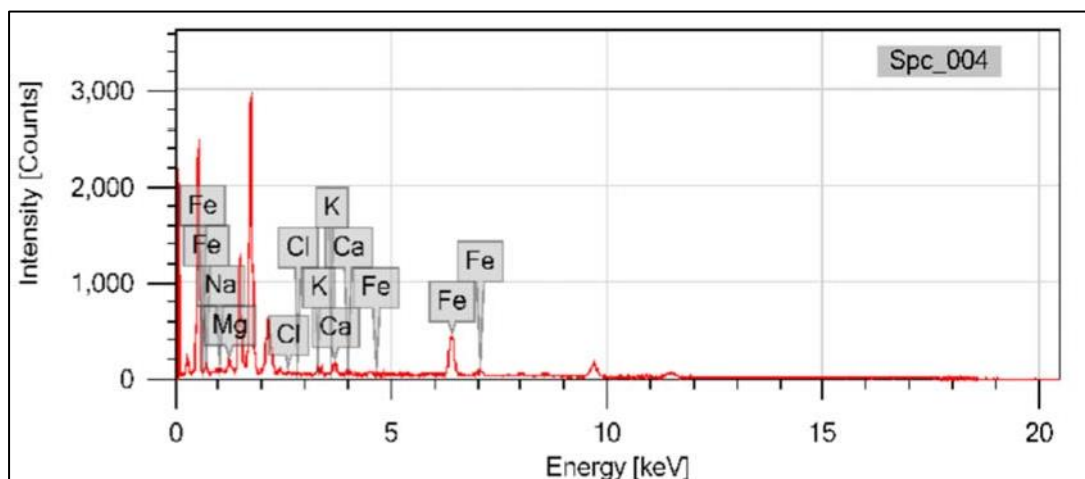
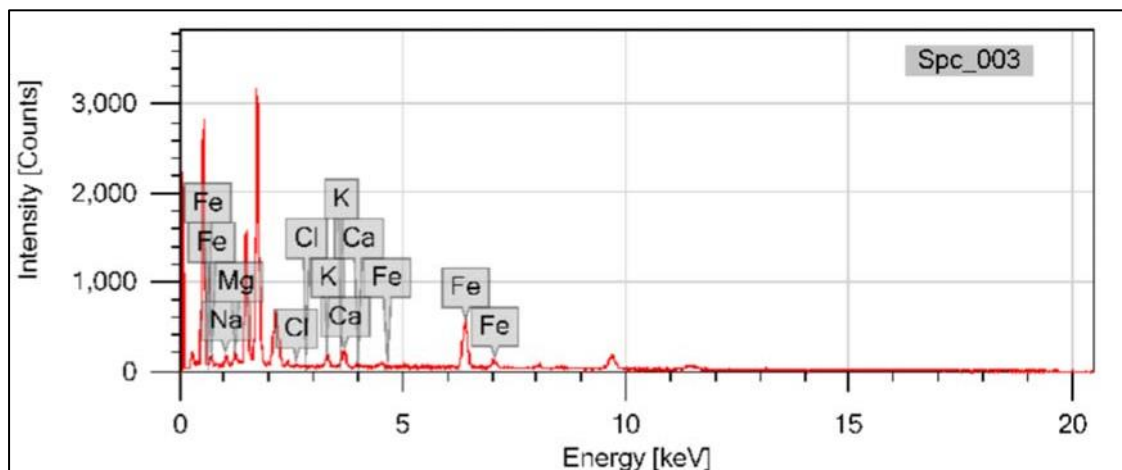


Figure 12a. EDX spectrum of soil samples at site -3 (A8)



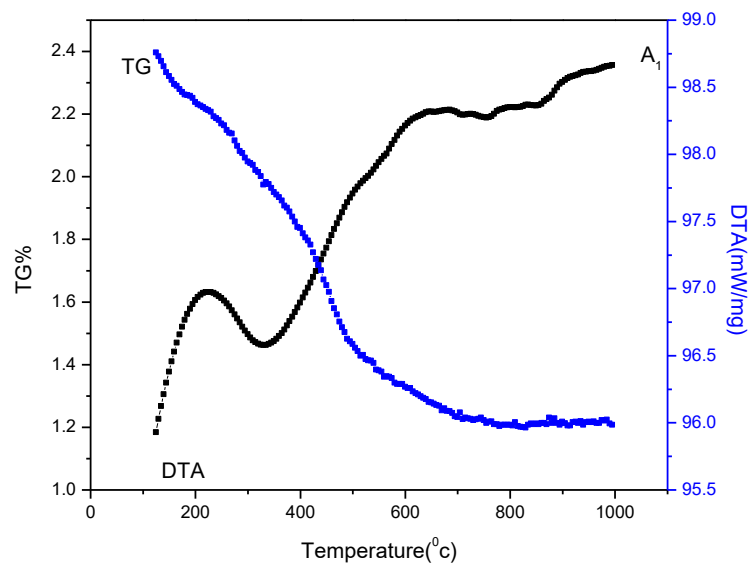
**Figure 13a. EDX spectrum of soil samples at site -3 (A9)**

**Table 10. The elemental concentration of soil samples**

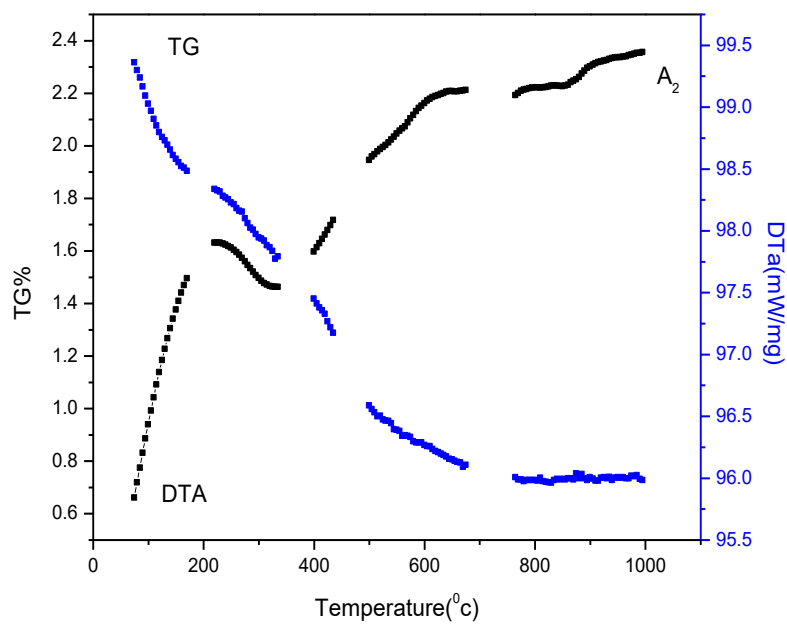
Elements	SEM – EDX method(%)								
	site – 1			site – 2			site – 3		
	A1	A2	A3	A4	A5	A6	A7	A8	A9
<b>Ca</b>	11.30	11.74	13.91	9.91	5.89	21.03	30.38	9.00	9.88
<b>Mg</b>	12.49	13.58	10.62	13.54	6.95	17.95	6.95	8.31	5.32
<b>Na</b>	3.09	3.35	6.06	1.74	3.48	2.67	6.25	2.37	7.64
<b>K</b>	9.38	4.02	3.81	10.00	12.31	4.73	2.71	3.73	5.75
<b>Cl</b>	2.88	2.25	1.48	3.58	2.77	1.93	2.29	1.91	1.51
<b>Fe</b>	56.26	56.14	55.42	52.94	58.60	51.68	51.41	74.67	69.90
<b>Cu</b>	4.60	8.92	8.70	8.30	10.00	-	-	-	-

**3.4 Thermal analysis**

Soil is a mixture of organic materials and the organic component can obscure characteristic mineral endothermic peaks with reaction at 300 – 800 °c. TG-DTA is very useful, clay group characterization. The TG-DTA curves are shown in figure 14 -22 for the samples (A<sub>1</sub> – A<sub>9</sub>).

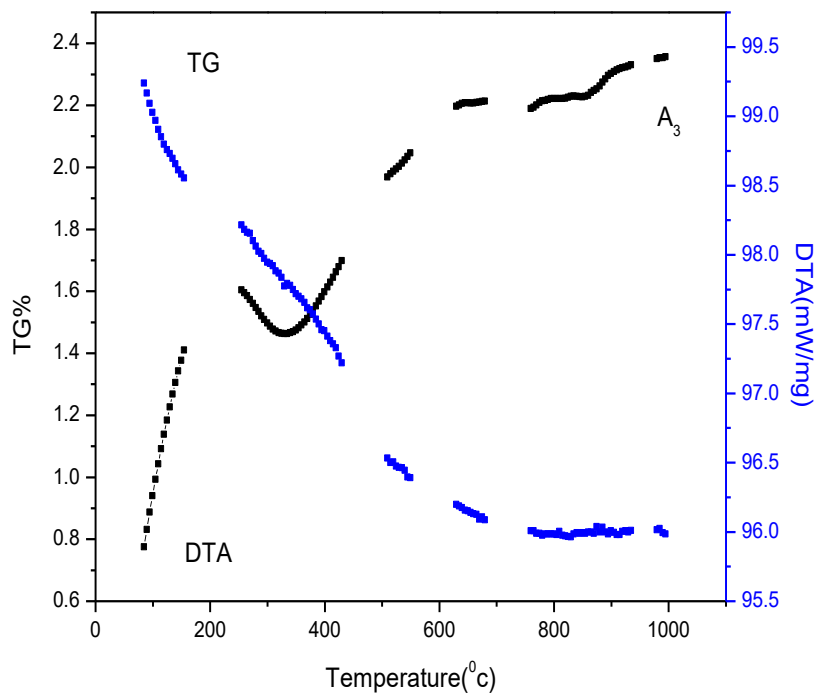


**Figure 14. Thermo gravimetric analysis of soil samples at site-1 Autumn season (A<sub>1</sub>)**

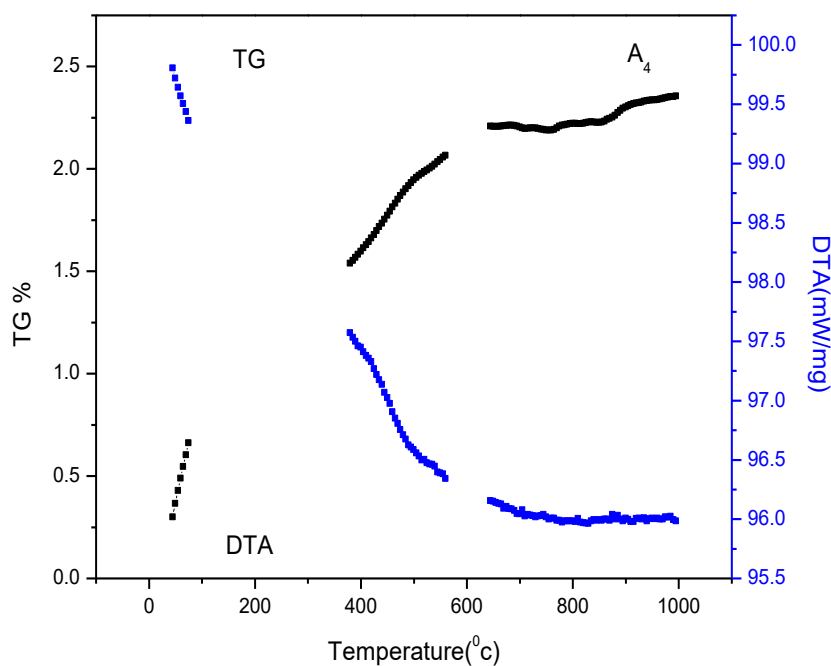


**Figure:15. Thermo gravimetric analysis of soil samples at site-1 Autumn season (A<sub>2</sub>)**

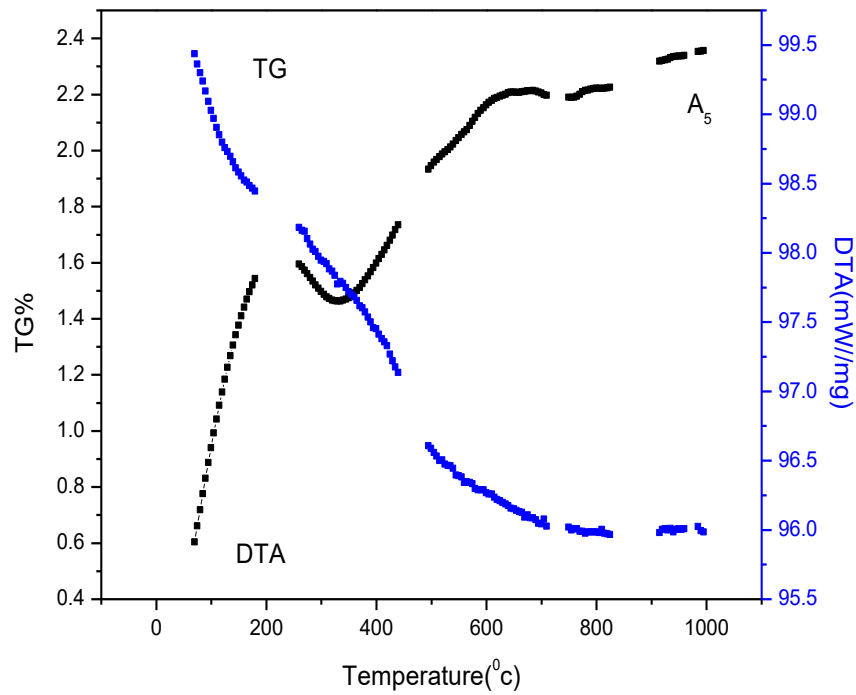




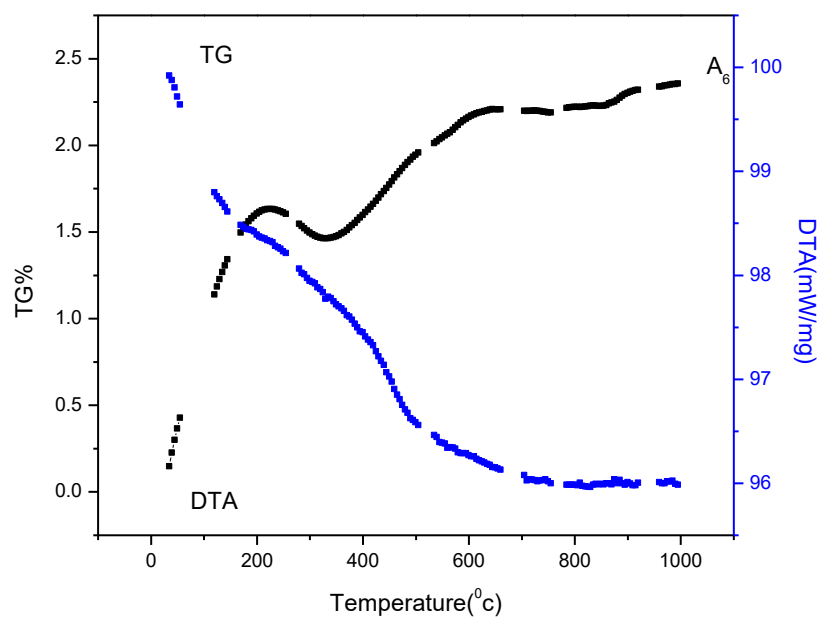
**Figure:16. Thermo gravimetric analysis of soil samples at site-1 Autumn season (A<sub>3</sub>)**



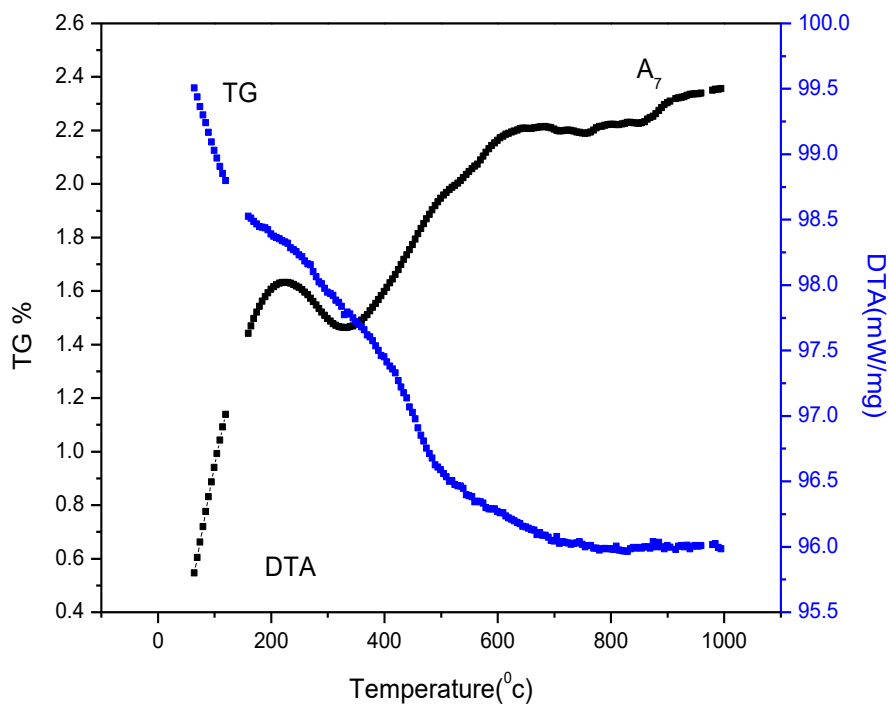
**Figure:17. Thermo gravimetric analysis of soil samples at site-2 Autumn season (A<sub>4</sub>)**



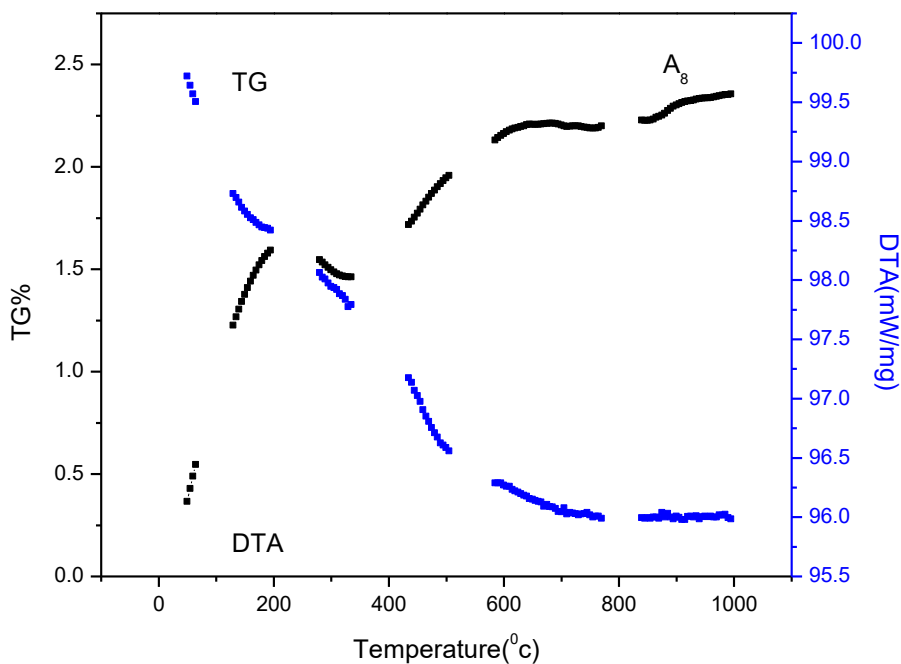
**Figure:18. Thermo gravimetric analysis of soil samples at site-2 Autumn season (A<sub>5</sub>)**



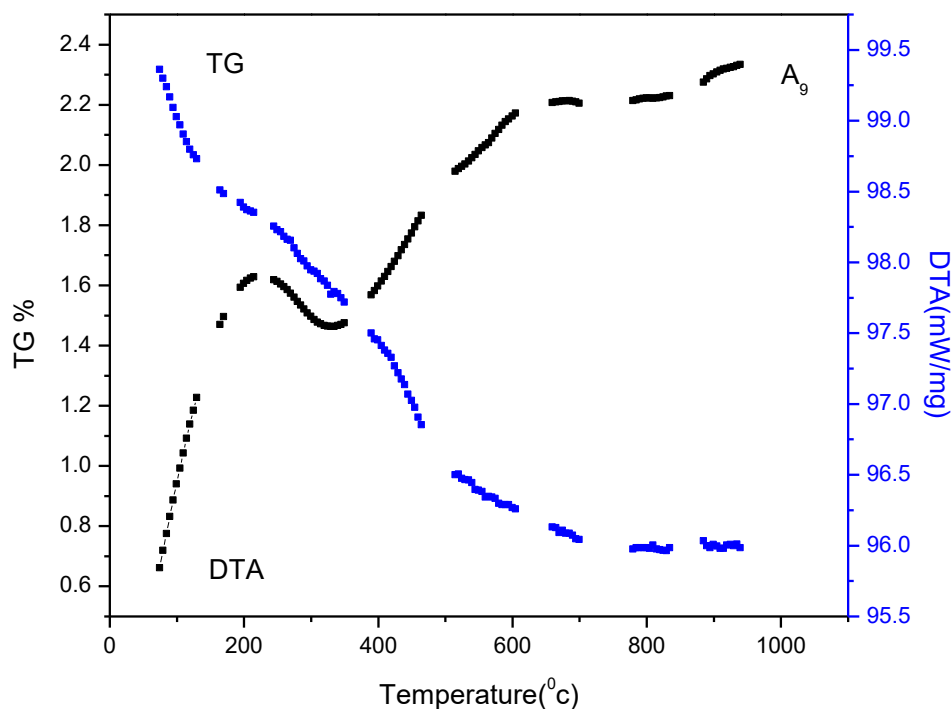
**Figure:19. Thermo gravimetric analysis of soil samples at site-2 Autumn season (A<sub>6</sub>)**



**Figure:20. Thermo gravimetric analysis of soil samples at site-3 Autumn season (A<sub>7</sub>)**



**Figure 21. Thermo gravimetric analysis of soil samples at site-3 Autumn season (A<sub>8</sub>)**



**Figure 22. Thermo gravimetric analysis of soil samples at site-3 Autumn season (A<sub>9</sub>)**

Figure shows the TG-DTA curves, weight losses in one stage may be attributed the decomposition of water and another stage is dehydroxylation. The DTA curves shows endothermic peaks at 199<sup>0</sup>c, exothermic peaks at 258<sup>0</sup>c . The mass losses of first and second stage were 2%, 3% and 4%. They are associated with organic matter decomposition and such transformations.

## Conclusion

The techniques XRD, SEM-EDX and TG-DTA analysis details for mineralogical ,morphological characterizations and thermal behaviours of soil samples. The results are indicate the soils have different minerals are kaolinite, quartz, aragonite, feldspar and calcite. The thermal analysis results peaks for the dehydration and decomposition in soil up to 1200.The results are spectroscopic studies reveal that the industrial area soil is subjected of contamination by minerals. The increase of minerals and heavy metals and the quality of agricultural products.

## References

- [1] R.Selvaraju, N.Oumabady Alias Cannane and M.Rajendran,G.Thirupathi,  
International Research Journal of Engineering and Technology (IRJET),1664-  
1668,(2015).

- [2] Reetu shrma, Khageshwar singh patel, lesia lata and Huber milosh, American journal of Analytical chemistry, (2016).
- [3] C.Manoharan.P. Sutharsan, S.Dhanapandiyan and R.Venatachalapathy, J.Molecular Structure, 99-102,(2012).
- [4] J.Madejova, Analytical Chemistry, 1169-1174,(2003).
- [5] P.S.Nayak and P. Singh, Bull. Material Science, 235-238, (2007).
- [6] R. Ravisanakar, G.Senthilkumar, S Kiruba , A.chandrasekaran and P.P. Jebakumar, Indian journal of science Technology, 774-780, (2010).
- [7] N.Oumabady Alias Cannane, M.Rajendran and R.Selvaraju, International Journal of Chem Tech Research, 5625-5631, 2014.
- [8] N.Oumabady Alias Cannane, M.Rajendran and R.Selvaraju, Journal of Environment.Nanotechnology, 23-29, 2014.
- [9] Patel.K.S, Shukla.A, A.N.Tripathi and Hoffmann.P, Water, air and Soil pollution, 463-468,(2013).
- [10] Chou.C.L, Journal of Coal Geology, 1-13, (2012).
- [11] S.J.Chapman, C.D. Campbell, A.R.Fraser and G.Puri , Soil Biol.Biochem, 1193-1200, (2001).
- [12] N.Oumabady Alias Cannane, M.Rajendran and R.Selvaraju, Journal of Environmental Nanotechnology, 23-29,(2014).
- [13] P.S.Nayak and P. singh, Bull. Material Science, 235-238, (2007).
- [14] D. Prasanthan and T.V.Nayak, Pollut.Research, 475-479,(2000).
- [15] V.Ramasmy, P.Rajkumar and V.ponnusamy, Pure Applied Science, 49-55.
- [16] R. Selvaraju and V. Mahalakshmi, Neuroquantology, 3410 – 3425, (2022).