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Exploration of geo-mineral compounds in granite mining soils using XRD pattern data analysis

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Abstract. The purpose of the study was to investigate the major minerals present in granite mining waste and agricultural soils near and away from mining areas. The mineral exploration of representative sub-soil samples are identified by X-Ray Diffractometer (XRD) pattern data analysis. The morphological features and quantitative elementary analysis was performed by Scanning Electron Microscopy-Energy Dispersed Spectroscopy (SEM-EDS).The XRD pattern data revealed that the major minerals are identified as Quartz, Albite, Anorthite, K-Feldspars, Muscovite, Annite, Lepidolite, Illite, Enstatite and Ferrosilite in granite waste. However, in case of agricultural farm soils the major minerals are identified as Gypsum, Calcite, Magnetite, Hematite, Muscovite, K-Feldspars and Quartz. Moreover, the agricultural soils neighbouring mining areas, the minerals are found that, the enriched Mica group minerals (Lepidolite and Illite) the enriched Orthopyroxene group minerals (Ferrosilite and Enstatite). It is observed that the Mica and Orthopyroxene group minerals are present in agricultural farm soils neighbouring mining areas and absent in agricultural farm soils away from mining areas. The study demonstrated that the chemical migration takes place at agricultural farm lands in the vicinity of the granite mining areas.

1. Introduction

The granite stones are used in the construction of buildings, allied construction industry for flooring and decorative purposes for past decades. The global stone production has been continuously increasing and estimated as 1.5 billion square meters of processed tile products [1]. The quantity and abundance of minerals of mining areas depends on their mineralogy and geochemistry, and a few raw minerals are occasionally found to have comparatively high concentrations of natural radioactivity [2]. The mining areas may require long-term management and decontamination studies for further use, or mitigate risk to the public health and the environment. It is necessary to carefully evaluate the behaviour and distribution of mineral compounds in mining soils [3]. Therefore, the chemical characterization for such contaminated soils are necessary and useful in the effective removal of toxic trace elements, which is associated with minerals in order to improve environmental quality [4,5]. In recent years, several geochemical characterizations of mining soils have been conducted in order to assess the environmental impact of mining/metallurgy industries [6,7].The sequential and random sampling methodology have been used to collect the soil samples in agricultural farm lands, mine tailing dumps, marine sediments, sewage sludge, soil and industrially contaminated made-up ground [8].

The purpose of the study was to investigate the major minerals present in granite waste soils and agricultural soils near and far away from mining areas via any chemical migration. In the present study, the granite soils are collected and sampled form granite quarries at Rajupalem-Lakshmipuram (RL) village area in India for physiochemical and minerals exploration. The mineral composition of soil samples are identified by X-Ray Diffractometer (XRD) analysis, the morphological features and quantitative elementary analysis using Scanning Electron Microscopy-Energy Dispersed Spectroscopy (SEM-EDS).

2. Materials and methods

2.1. Soil sampling

In the Rajupalem-Lakshmipuram (RL) village, Chimakurthy mandal (Andhra Pradesh, India) a large mining and mineral extraction industry based on the excavation and processing of granite thrived for 30 years [9]. Locally it is called as black galaxy granite, but geologically is *Gabbric anorthosite* [9]. It is originated from igneous rock, name of the mother rock is gabbro and the speck is Bronzite. Hence, it is also called as Bronzite bearing Gabbro [10]. About 50 mining quarries are situated in the industrial area of Chimakurthy village. The industrial area has a surface of 5 km^2 where 25 surface soil samples (5-10cm depth) were collected from three locations of common overburden dump sites, mining sites and mine tailing sites using random sampling method. Figure.1 shows the area map of collection of soil samples. Sequential sampling method was used to collect the agricultural farm soils in the vicinity of granite quarries to find any traces of metals via chemical migration. In the present study, a total of eighteen soil samples have been collected from granite mining dumps and agricultural farm lands (neighbouring and away from mining area) and analysed for mineral compounds.
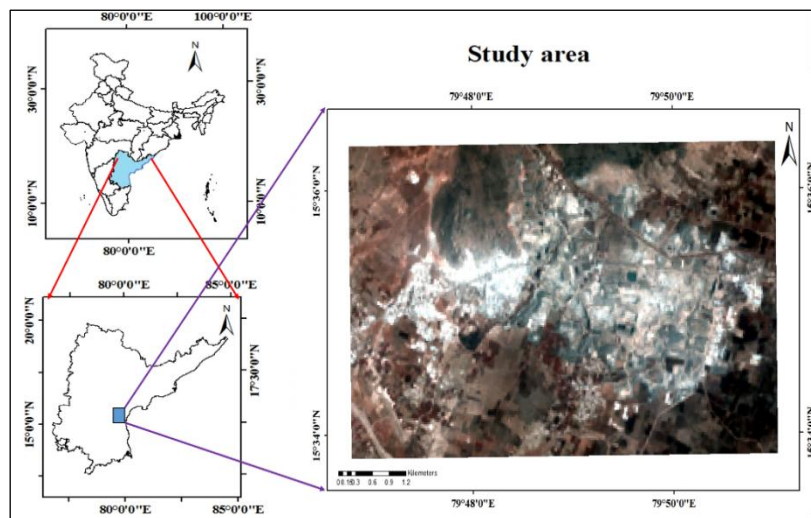

Figure 1. The area map of collection of soil samples

2.2. Sample preparation and physiochemical exploration

The soil samples were passed through a series of 4.75-0.002 mm mesh nylon fibre sieves in situ to remove rock fragments, vegetation and other large material. Finally, samples were homogenized and stored in inert plastic bags at room temperature for further analysis [11,12], (ASTM D- 422-63, Standard Test Method for Particle-Size Analysis of Soils) [13]. In this study, three different areas of mining waste and agriculture soil samples were used for the current chemical characterization. Soil pH, redox potential, electrical conductivity, TDS, moister content, porosity, organic and inorganic content were determined using standard protocols [14–16]. Soil pH and redox potential were measured with a glass electrode in a suspension of soil with deionized distilled water (1:2.5) (Digital P^H meter MK VI) and electrical conductivity at the same time TDS were measured in the same extract (diluted 1:5) (Systronics Conductivity TDS meter 308). Soil moisture content was determined by drying

method in box furnace at 125∘C for overnight. Then organic and inorganic matter were determined by ignition method at 550∘C weight loss in box furnace for 4 hours using INDFURR Superheat furnaces controller [17].

2.3. Zeta potential analysis

The zeta potential of the granite soils and agricultural soils were measured by means of zeta potential analyser (90 Plus Particle Size Analyser, Brookhaven Instruments Corporation, NY, USA, using Zeta Plus software). The measurement of zeta potential is based on the direction and velocity of soil particles dispersed in liquid media under the influence of known electric field. Zeta-potential (ζ) is a dominating factor of a solid surface, which controlling the magnitude and direction of movement in dispersed liquid media under influence of electric field [18,19]. Zeta potential values were used to predict the nature of the electrostatic potential near the surface of the particle by mixing an aliquot of the sample with 10^{-3}M Potassium chloride (KCl) solution prior to analysis [20,21].

2.4. X-Ray diffractometer (XRD)

Qualitative analysis of granite and agricultural clay mineral composition were collected by BRUKER D8 Advance XRD machine and conducted by the X-ray powder method of Debye-Scherrer. The patterns of XRD of all samples have been recorded with DIFFRAC Plus XRD software using the parameters: Cu Kα radiation, Ni filter, 40 kV lamp voltage, lamp current of 40 mA, the registration step by step:0.02° per step, scan speed 1s per step, scanning rate 1^0 per minute.. [22–25]. Total scanning time was 40 minutes per sample and data were collected from 20 to 60^0 for granite clay samples for the present study.

2.5. Scanning Electron Microscopy (SEM) and Energy Dispersed Spectroscopy (EDS)

Quantitative and elementary analysis of granite and agricultural soil samples were characterized by SEM-EDX instrument (X-act, ZEISS, Oxford instruments, United Kingdom). The morphological features of dried soil samples are coated with fine carbon layers to improve the secondary electron signal to enable or improve the images of samples in the SEM. Fine carbon layers are being transparent to the electron beam and conductive in X-ray microanalysis.

The elementary analysis has been examined with Aztec Energy EDS Software. The prepared soil samples with carbon coated were subjected to scanning electron microscopy coupled with energy dispersive X-ray spectrometer (SEM/EDX) at an electrical voltage around 10 keV for characterization of metals, heavy metals, metalloids and any traces elements of radio activity metals [11,26].

3. Results and discussion

3.1. Physiochemical properties

From Table 1, the physiochemical properties of three soils are reported. The mining soil texture of the sub-samples were sandy loam type. The particle size distribution of which is observed high percentage of three types of sand fractions such as coarse with 13-15%, medium with 12-15% and fine with 11-15% respectively, in the sieve analysis process. In case of agricultural soils, are found that sand and silt loam with 41% and 31.5% respectively.

The porosity of the slit and clay soil showed that in the range of 0.36-0.52 for three study areas of granite waste and two agricultural soils, however for agricultural soils has more porosity (0.52) than granite waste and agricultural soil near mining area. The representative soil sub-samples of three study areas showed an alkaline pH (8.3-9.8), the granite waste is more alkaline than agricultural soils, an electrical conductivity and high total dissolved salts (TDS) were in the range of 104-145 µs/ppm and 103-202 ms/ppm respectively. It is observed the redox potential in the range from -78mV to -171mV, it indicates that the soil was highly alkaline in nature due to low redox potential. But in case of agricultural soil, redox potential was -78mV by means of slightly alkaline in nature. It was found that the granite waste soil sub-samples are with high inorganic matter content around 98%, low moister

content (<1%) and less organic matter content (<1.64%). In case of agricultural soils are more organic (5%) and moister content (1.6% than granite dump soils.

Table 1. The physical and chemical properties of granite waste and agricultural soils

Soil texture analysis		Granite Waste*	AG Soil near Granite area*	AG Soil*
Gravel (>4.75 mm)		34.70%	23.25%	12%
Sand	Coarse (4.75-2.00 mm)	15.20%	13.86%	12.97%
	Medium (2.00-0.425 mm)	14.62%	12.11%	14.47%
	Fine (0.425-0.075 mm)	10.97%	11.64%	15.10%
Slit (0.075-0.002 mm)		13.99%	14.75%	31.50%
Clay (<0.002mm)		10.53%	18.10%	12.90%
Porosity		0.36	0.49	0.52
P^H		9.8	9.2	8.3
Electrical Conductivity (µs/ppm)		121.64	145.24	104.56
Redox potential		-171	-149	-78
Zeta Potential		-31.4	-27.2	-25.4
TDS (ms/ppm)		136.41	202.66	103.14
Moisture(%) at 125^0C		0.81	1.22	1.61
Total Inorganic Matter (%) at 500^0C		97.35	95.15	93.45
Total Organic Matter (%)at 500^0C		1.64	2.94	4.58

Data are the average of five replicates

From Figure 2 (a, b & c), shows the zeta potential values of three groups of a) granite waste soil, b) agricultural soil near mining area, and c) agricultural soil away from mining area. The zeta potential values can be used to determine the stability of soil particles by means of interface charged species in liquid dispersed media. In case of granite waste soil solution gave a mean zeta potential value of -31.4 mV, for agricultural soil near mining area soil is -27.2 mV and -25.4 mV for agricultural soil away from mining area, these indicate that colloidal stable dispersion of the particles under influence of electrical potential. It indicated that less colloidal stable dispersion of the particles due to anionic aggregation of particles in agricultural soils.
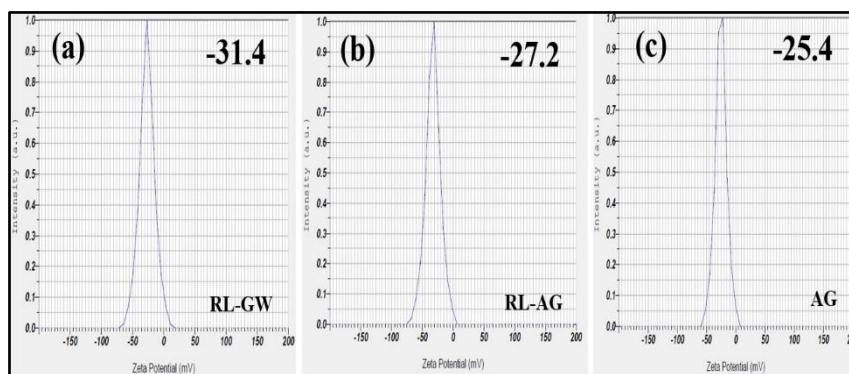
Figure 2. Zeta potential analysis of three soils at Rajupalem-Lakshmipuram (RL) mining zone: a) Granite waste (GW), b) Agricultural soil near mining area (RL-AG), c) Agricultural soil away from mining area (AG)

3.2. Identification of mineral compounds

The dried fine powder samples were subjected to X-ray diffractometer and pattern data were collected from 20 to 60^0 for granite waste soil samples and agricultural clay samples. In the present study, the XRD pattern of granite waste and agricultural soils presented in Figure 3, the XRD pattern analysis of three soils: Granite waste, Agricultural soil at mining area and away from mining area (2Θ range from 20^0 to 37^0) and Figure 4, the XRD pattern analysis of three soils: Granite waste, Agricultural soil at mining area and away from mining area (2Θ range from 37^0 to 55^0). It shows the presence major minerals are Quartz, Albite, Anorthite, K-Feldspars, Muscovite, Annite, Lepidolite, Illite, Enstatite and Ferrosilite in case of granite waste. But in case of agricultural soils the minerals are identified as Gypsum, Calcite, Magnetite, Hematite, Muscovite, K-Feldspars and Quartz.
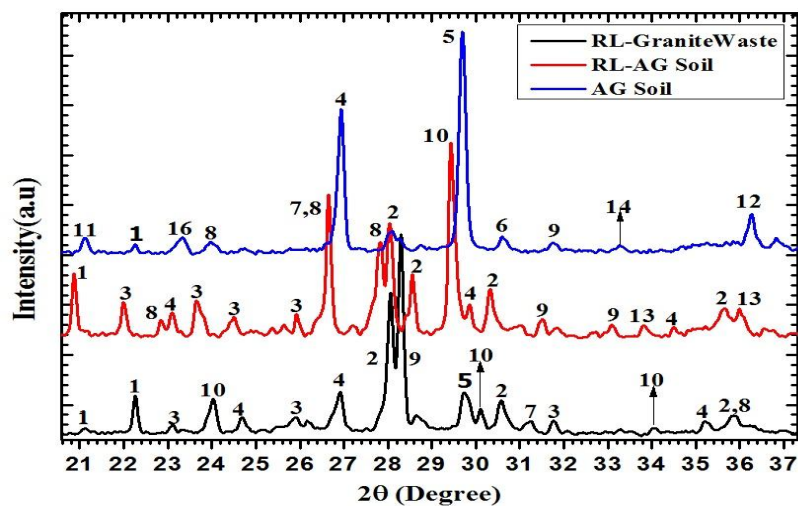

Figure 3. XRD pattern analysis of three soils (Granite waste, Agricultural soil at mining area and away from mining area (2Θ range from 20^0 to 37^0, numbers indicate the corresponding mineral compounds described in Table 2).
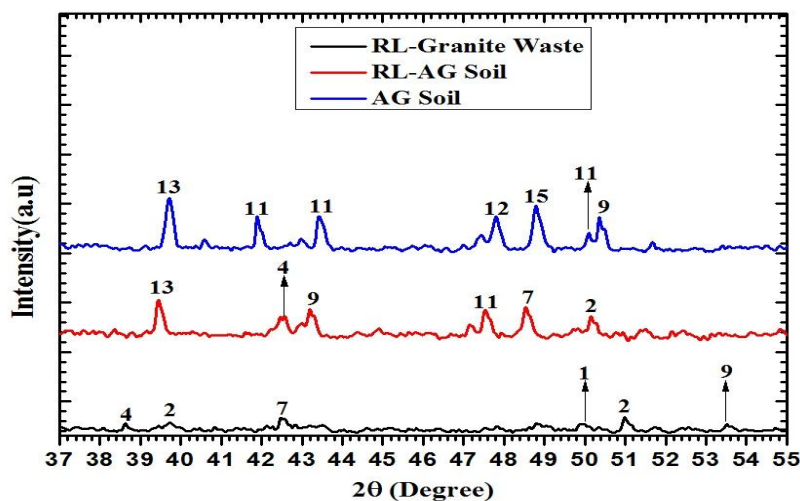
Figure 4. XRD pattern analysis of three soils (Granite waste, Agricultural soil at mining area and away from mining area (2Θ range from 37^0 to 55^0, numbers indicate the corresponding mineral compounds described in Table 2).

Table 2. The list of the Identified mineral compounds in granite waste and agricultural soils using XRD pattern analysis

Pattern No.	Mineral Name	Chemical Formula	JCPDS Card No.	Soil Type
1	Quartz	SiO_2	83-0539	Granite Waste/ AG Soil
2	Albite	$Na\ Al\ Si_3O_8$	10-0393	Granite Waste
3	Anorthite	$CaAl_2Si_2O_8$	89-1461 /89-1462 /89-1470 /89-1471 /85-1472	Granite Waste
4	K-Feldspars	$KAlSi_3O_8$	19-0931	Granite Waste/ AG Soil
5	Muscovite	$K\ Al_2\ Si_3\ Al_{10}\ (OH)_2$	07-0032/ 06-0263	Granite Waste/AG Soil
6	Annite	$K\ (Fe,\ Mg)_3\ AlSi_3\ O_{10}\ (OH)_2$	02-0045	Granite Waste/ AG Soil
7	Lepidolite	$K\ Li_2\ Al\ (Al,\ Si)_3\ O_{10}\ (F,\ OH)_2$	85-0389 /38-0425	Granite Waste
8	Illite	$(K_2H_3O)\ Al_2\ Si_3\ Al\ O_{10}\ (OH)_2$	26-0911	Granite Waste
9	Enstatite	$Mg_2Si_2O_6$	86-0430	Granite Waste/ AG Soil
10	Ferrosilite	$Fe_2Si_2O_6$	76-0891/ 76-0889	Granite Waste
11	Magnetite	Fe_3O_4	89-6466	AG Soil
12	Calcite	$CaCO_3$	87-1863/ 03-0513	AG Soil
13	Gypsum	$CaSO_4\ 2\ H_2O$	76-1746	AG Soil
14	Hematite	Fe_2O_3	89-8104	AG Soil
15	Copper oxide	Cu_2O	78-2076	AG Soil
16	Clintonite	$Ca(Mg,\ Fe,\ Al)_3(Al,\ Si)_4\ O_{10}\ (OH)_2$	20-0321	Granite Waste /AG Soil

Concerning elementary analysis, SEM-EDX instrument was used to estimate the quantity of elements present in the representative soil sub-samples of the three areas of mineral bench at mining waste and agricultural soils near and away from miming site. The presence of the main ten elements and their relative abundances including trace elements of radionuclide found, after energy diffraction X-ray spectrometer determination. The elements are associated with major mica group and Othopyroxene groups as Silicon(Si), Calcium (Ca), Magnesium (Mg), Chromium(Cr), Cobalt(Co), Copper(Cu), Zinc(Zn), Iron(Fe), Manganese (Mn), and Nickel(Ni) are identified based on elementary analysis. Gabbro granites and associated ultramafic rocks contain valuable amounts of chromium, nickel, cobalt, gold, silver, platinum, zinc and copper sulphides [27]. Previous study, Pea Gonzlez et al., 2006 reported the heavy metals for *Rosa Porrino-type* granite in the year 2006 were V, Cr, Ni, Cu, Zn, As, Se, Mo, Cd, Sb, Ba, Hg, Pb, Mn, U, Rb, and Sr respectively [31]. It is observed that the elements in the present study matched with literature for granite soils. However, the quantity of elements and

metals in both granite waste soils and agricultural soils are all most similar but their abundance vary due to different mineralogical and geographical conditions.

From Figure 5, Figure 6 and Figure 7(a, b, c), the SEM images are produced at 20µm, 2µm and corresponding EDX spectrum at EHT10 keV, of three different soil sub-samples. From this analysis, a perceptible variation in the trace element concentration of samples in four different areas are observed. From EDX spectrum analysis, it is found that the major elements present in granite waste and agricultural soils are silicon, calcium and small traces of metals and metalloids.
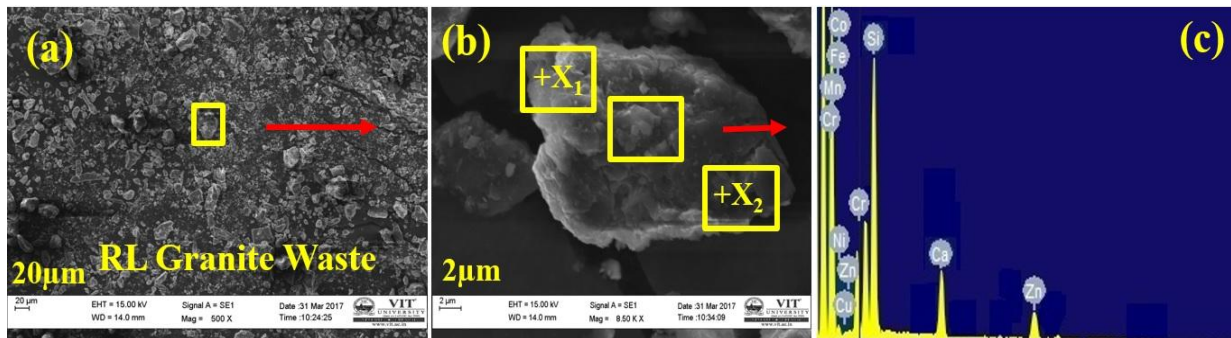

Figure 5. The SEM images (a, b) and corresponding EDX spectrum (c) of granite waste
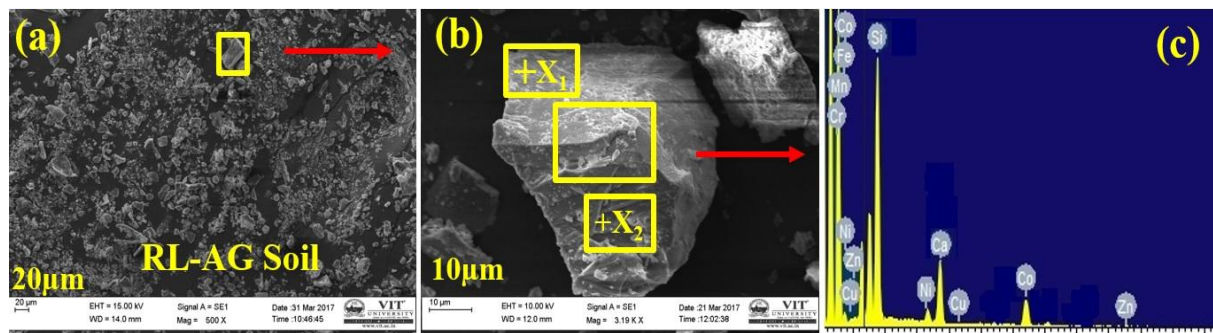($+X_1$: Plagioclase-Albite, $+X_2$: Orthopyroxene-Enstatite)

Figure 6. The SEM images (a, b) and corresponding EDX spectrum (c) of agricultural soil near mining area ($+X_1$: Mica-Lepidolite, $+X_2$: Orthopyroxene-Ferrosilite).
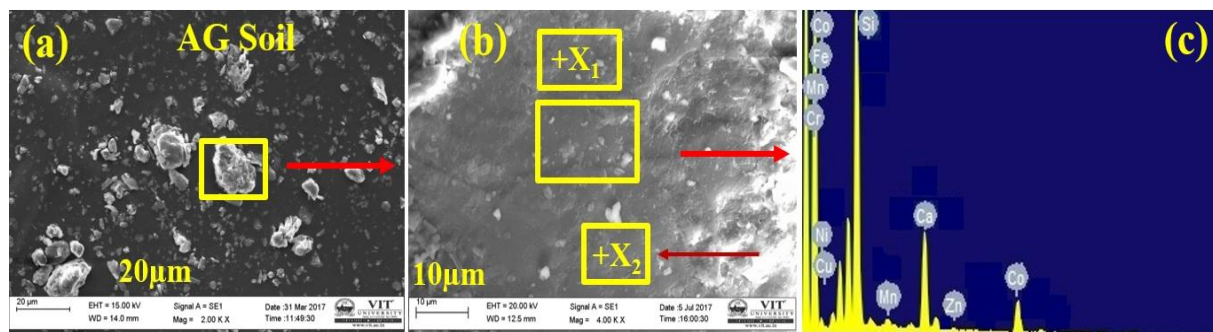

Figure 7. The SEM images (a, b) and corresponding EDX spectrum (c) of agricultural soil away from mining area ($+X_1$: Orthoclase-K-Feldspar, $+X_2$: Mica-Muscovite)

From Figure 5b, the black portion is associated with Orthopyroxene group minerals such as Enstatite and white portion represents Plagioclase mineral group such as Albite mineral. From Figure 6b, the

black portion is associated with Orthopyroxene group minerals such as Ferrosilite and white portion represents Mica mineral group such as Lepidolite and Illite minerals respectively. From Figure 7b, the black portion is associated with Orthoclase group minerals such as K-Feldspars and white portion represents Mica mineral group such as Muscovite minerals respectively.

4. Conclusion

Geochemical mineral data revealed that the major minerals were identified as Quartz, Albite, Anorthite, K-Feldspars, Muscovite, Annite, Lepidolite, Illite, Enstatite and Ferrosilite in granite waste. However, in case of agricultural soils the minerals are identified as Gypsum, Calcite, Magnetite, Hematite, Muscovite, K-Feldspars and Quartz. Moreover, in case of agricultural soils near granite mining area, the minerals are found that, the Mica group minerals such as Lepidolite and Illite, the Orthopyroxene group minerals such as Ferrosilite and Enstatite. The morphological features and quantitative elementary analysis was performed by Scanning Electron Microscopy-Energy Dispersed Spectroscopy (SEM-EDS).The granite minerals such as Mica and Orthopyroxene group minerals observed in agricultural farm soils, where neighbour to the mining areas and absent in agricultural farm soils away from mining areas. It is observed that the chemical migration takes place at agricultural soils in the vicinity of the granite mining areas. The study is useful for the researchers who is interested in a particular geo-chemical exploration and characterization to perform further studies to explore the chemical migration in the environment among contaminated soils, mining dumps, industrial solid waste disposal and sediments. Further studies have to be investigated the migration of environmentally hazardous heavy metals from mining dump sites to environment such as agricultural farm lands and water bodies via chemical migration.

5. Acknowledgement

6. References

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