

Trace Elemental Characterization of Forest Soil Samples from Mulugu Region, Telangana, India Using Energy Dispersive X-Ray Fluorescence Technique

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Abstract: Soil is an important component of forest ecosystems and serves as a reservoir for essential and trace elements that influence nutrient cycling, plant growth, and environmental quality. The present study demonstrates the elemental composition of soil from the Mulugu forest region, Mulugu District, Telangana, India, by using energy-dispersive X-ray fluorescence (EDXRF) analytical technique. Three soil samples (SS1, SS2, and SS3) collected within approximately a 1 km radius were analyzed for seven elements, namely V, Cr, Mn, Fe, Ni, Cu, and Zn. The measured concentrations showed clear spatial variations among the sampling locations. Iron (Fe) exhibited the highest concentration, ranging from 29,486 to 38,590 ppm, followed by Manganese (Mn) (599.1–735.18 ppm), Zinc (Zn) (79.5–93.2 ppm), Vanadium (V) (75.6–96.5 ppm), Chromium (Cr) (69.2–83.36 ppm), Copper (Cu) (40.1–49.8 ppm), and Nickel (Ni) (34.5–43.5 ppm). Among the three sampling locations, SS1 consistently showed the highest concentration of all investigated elements, whereas SS3 exhibited the lowest concentrations. Comparison with standard certified reference values of NIST SRM 2587 showed that the mean concentrations of V, Mn, Fe, and Ni were approximately comparable to or higher than the corresponding reference values, whereas Cr, Cu, and Zn were lower. The results demonstrate measurable spatial variability in the elemental composition of the forest soils over a relatively small geographical area. The study provides baseline elemental information for soils of the Mulugu forest region and demonstrates the applicability of EDXRF for rapid multi-element soil characterization. The observed differences from the reference material should be interpreted as differences in elemental abundance rather than direct evidence of contamination, since NIST SRM 2587 is a reference material and not a site-specific environmental.

Keywords: Forest soil; Trace elements; EDXRF; Mulugu; Telangana; Soil elemental composition; nEXT software.

1. Introduction

Soil is one of the most important natural resources supporting terrestrial ecosystems and plays a fundamental role in vegetation growth, nutrient cycling and environmental sustainability. Forest soils, in particular, represent complex systems in which minerals, organic matter, microorganisms and plant-derived materials interact continuously [1]. The elemental composition of soil is therefore an important parameter for understanding the geochemical characteristics and ecological condition of forest ecosystems [2].

Trace elements in soils originate from both natural and anthropogenic sources. Weathering of parent rocks, atmospheric deposition, biological activity and decomposition of organic matter contribute to the natural elemental composition of soil, while agricultural activities, transportation, industrial emissions and other human activities can introduce additional elements into the soil environment [3]. The distribution of trace elements can vary considerably even over relatively short geographical distances because of differences in geological substrate, soil formation, vegetation and local environmental conditions [4].

Several analytical techniques, including Atomic Absorption Spectroscopy (AAS), Inductively Coupled Plasma Mass Spectrometry (ICP-MS), Particle-Induced X-ray Emission (PIXE), Neutron Activation Analysis (NAA) and X-Ray Fluorescence (XRF), have been employed for elemental characterization of environmental samples [5]. Among these techniques, Energy Dispersive X-Ray Fluorescence

(EDXRF) is particularly useful for multi-elemental analysis because it provides simultaneous qualitative and quantitative determination with relatively simple sample preparation and without extensive chemical digestion [6].

The EDXRF approach was previously applied for the elemental characterization of soils from a coal-mining environment, where V, Cr, Mn, Fe, Ni, Cu and Zn were quantified in soil samples using a Xenometrix EX-3600 EDXRF system and nEXT software. The same study demonstrated considerable variation in elemental concentrations among sampling locations.

Mulugu District contains extensive forest ecosystems and represents an environmentally significant region of Telangana. However, baseline information concerning the trace elemental composition of soils from selected forest locations is comparatively limited. Therefore, the present study focuses specifically on the elemental characterization of three soil samples collected within a 1 km radius of the selected Mulugu forest region using EDXRF spectroscopy.

The main objective of the present investigation is to determine and compare the concentrations of selected trace elements in the three forest soil samples and to establish a preliminary elemental profile that can serve as baseline information for future environmental studies.

2. Experimental Methods

2.1 Study Area and Collection of Soil Samples

The soil samples were collected from the Mulugu forest region, Mulugu District, Telangana, India. Three sampling locations were selected within a 1 km radius of the designated study area.

The samples were named as a coding of:

- SS-1 –Soil Sample 1
- SS-2 –Soil Sample 2
- SS-3 –Soil Sample 3

The three locations were selected to represent spatial variation within the selected forest region. The original reference paper also used geographically separated soil sampling locations and presented their positions on a sampling map. For the present study, a new sampling-location map showing SS-1, SS-2 and SS-3 should be prepared

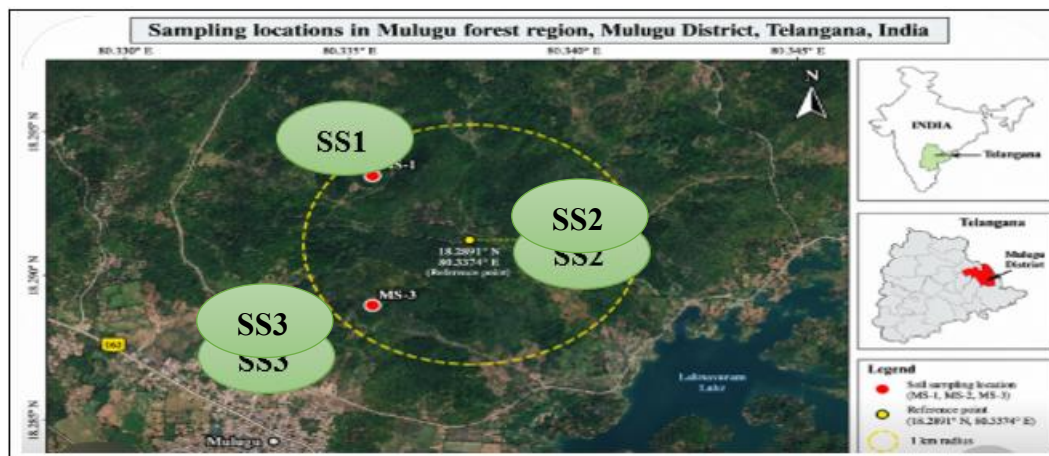


Figure 1: Geographical Satellite image of the Forest location in Mulugu district

2.2 Sample Preparation

The collected soil samples were initially air-dried to remove moisture (shown in figure 2). Visible stones, plant residues and other extraneous materials were removed manually. The dried samples were subsequently ground using an agate mortar and pestle to obtain a homogeneous fine powder.

Approximately 150 mg of each powdered sample was used for pellet preparation. The powder was compressed into

pellets of approximately 13 mm diameter and 2 mm thickness using a hydraulic press. Triplicate pellets were prepared for each sample to improve the reliability of the measurements. This sample-preparation approach follows the pellet preparation procedure described in the reference study, in which dried soil was ground to a fine powder and approximately 150 mg was compressed into 13-mm pellets [7].



Figure 2: Images of 3 soil samples collected from Mulugu forest region

2.3 EDXRF Experimental Procedure

The elemental analysis was carried out using an Energy Dispersive X-Ray Fluorescence spectrometer (shown in figure 3). The EDXRF technique enables simultaneous detection of several elements present in the soil matrix. The elemental concentrations were determined using the nEXT software following calibration with an appropriate certified reference material. In the reference study, NIST 2587 was

used for calibration/validation and the instrument was capable of detecting elements from Na to U, with reported detection limits in the range of approximately 1–10 ppm [8]. For the selected elemental analysis, the experimental parameters should be reported according to your actual instrument conditions. If the same conditions were used as in the previous study, they may be reported as:



Figure 3: EDXRF analytical Spectrometer and their specifications

X-ray filter: Ti
Filter thickness: 0.05 mm
Operating voltage: 14 kV
Measurement time: 400 s
Detector: Si (Li)
Reference material: NIST 2587
Quantification software: nEXT

3. Results and Discussion

3.1 Elemental Composition of Mulugu Forest Soil

The EDXRF analysis of the three forest-soil samples collected from the selected Mulugu forest region revealed the presence of V, Cr, Mn, Fe, Ni, Cu and Zn at measurable concentrations (Figure 4 shows the bar graph of the elemental content in soil sample). The elemental concentrations showed noticeable spatial variation among SS1, SS2 and SS3, although all three samples were collected within a relatively restricted geographical area. Such short-range variations can arise from differences in parent material, mineral weathering, soil-forming processes and organic-matter interactions. Studies of forest soils have similarly demonstrated that geological background can strongly influence trace-element concentrations and that interpretation should account for natural geochemical variability [9].

The elemental concentrations are expected to exhibit some degree of spatial variation among the three sampling locations. Such variation can arise from differences in parent material, mineral composition, weathering intensity, organic matter accumulation and localized deposition. Because the three samples were collected within a relatively restricted geographical area, the comparison provides an opportunity to examine short-range variations in the elemental composition of the forest soil.

Table 1: Mean Elemental concentration of three soil samples compared with NIST 2587 certified standard reference values

Elements	SS1 (ppm)	SS2 (ppm)	SS3(ppm)	NIST 2587
V	96.5	85.2	75.6	78
Cr	83.36	76.4	69.2	92
Mn	735.18	682	599.1	651
Fe	38590	32714	29486	28130
Ni	43.5	39	34.5	36
Cu	49.8	45.4	40.1	160
Zn	93.2	87.9	79.5	335.8

SS1: Soil sample 1; SS2: Soil sample 2; SS3: Soil sample 3

3.1 Vanadium (V)

The concentration of V varied from 75.6 to 96.5 ppm, with an average concentration of 85.77 ppm. The highest value was observed in SS1 (96.5 ppm), followed by SS2 (85.2 ppm), while SS3 exhibited the lowest concentration (75.6 ppm). The average concentration was approximately 1.10 times the NIST 2587 reference value of 78 ppm.

The gradual decrease from SS1 to SS3 suggests localized spatial variation in V distribution. Because the samples were collected within a limited forest area, this variation may be associated with differences in mineralogical composition and weathering of the underlying parent material. Therefore, the observed V distribution should be interpreted as a geochemical variation rather than directly as evidence of anthropogenic contamination [10].

3.2 Chromium (Cr)

Cr concentrations ranged from 69.2 to 83.36 ppm, with an average value of 76.32 ppm. SS1 showed the maximum Cr concentration (83.36 ppm), whereas SS3 showed the minimum concentration (69.2 ppm).

All three samples exhibited Cr concentrations lower than the NIST 2587 reference value of 92 ppm. The concentrations correspond to approximately 75–91% of the NIST reference value. The relatively consistent decrease from SS1 to SS3 indicates spatial heterogeneity but does not suggest a substantial enrichment relative to the reference material. Chromium in forest soils can be controlled strongly by the geological composition of the soil-forming material. Previous work on forest soils has emphasized that geological background must be considered when interpreting Cr and other trace-element concentrations [11].

3.3 Manganese (Mn)

Mn exhibited concentrations between 599.1 and 735.18 ppm, with an average value of 672.09 ppm. SS1 showed the highest concentration (735.18 ppm), followed by SS2 (682.0 ppm), while SS3 showed the lowest concentration (599.1 ppm). The average Mn concentration was approximately 1.03 times the NIST 2587 reference value of 651 ppm. SS1 and SS2 were above the reference value, whereas SS3 was below it.

Manganese is an important micronutrient in forest ecosystems, and its concentration and availability are influenced by mineral weathering, soil redox conditions, organic matter and biological cycling. Research on forest

ecosystems has demonstrated that Mn can undergo substantial changes through interactions among vegetation, soil minerals and organic matter [12].

3.4 Iron (Fe)

Among the investigated elements, Fe exhibited the highest concentration in all three soil samples. The concentration varied from 29,486 to 38,590 ppm, with an average value of 33,596.67 ppm. SS1 recorded the highest Fe concentration of 38,590 ppm, followed by SS2 with 32,714 ppm, while SS3 exhibited 29,486 ppm. The average Fe concentration was approximately 1.19 times the NIST 2587 reference value of 28,130 ppm.

The relatively high Fe concentration is consistent with the major contribution of iron-bearing minerals to soil elemental composition. The decreasing concentration from SS1 to SS3 indicates spatial heterogeneity within the selected sampling area. Fe is also strongly involved in forest-soil biogeochemical cycling; changes in organic matter and soil processes can influence Fe retention and transformation [13]. Importantly, the higher Fe concentration relative to NIST 2587 should not by itself be interpreted as contamination, because NIST 2587 is a reference soil material and comparison with a reference material does not constitute a site-specific pollution assessment.

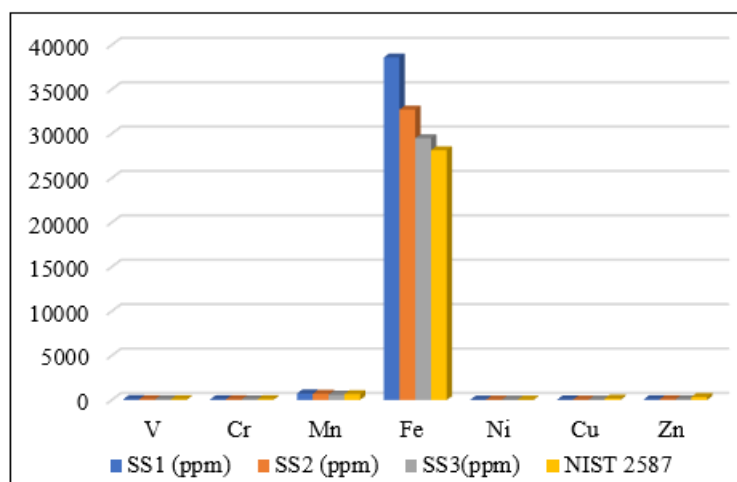


Figure 4: Bar graph of the Elemental content in Soil samples compared with NIST

3.5 Nickel (Ni)

Ni concentrations ranged from 34.5 to 43.5 ppm, with an average concentration of 39.0 ppm. The highest concentration was observed in SS1 (43.5 ppm), followed by SS2 (39.0 ppm), while SS3 recorded 34.5 ppm. The average concentration was approximately 8.3% higher than the NIST 2587 reference value of 36 ppm. SS1 and SS2 were above the reference value, whereas SS3 was slightly below it.

The relatively small variation in Ni among the three locations suggests a comparatively consistent distribution across the selected study region. Natural geological sources are particularly important when interpreting Ni concentrations in forest soils because parent material can produce substantial differences in background levels [14].

3.6 Copper (Cu)

Cu concentrations varied from 40.1 to 49.8 ppm, with an average value of 45.1 ppm. The maximum concentration was observed in SS1 (49.8 ppm), while SS3 recorded the minimum value (40.1 ppm).

All three samples exhibited Cu concentrations substantially below the NIST 2587 reference value of 160 ppm, corresponding to approximately 25–31% of the reference value. The comparatively lower Cu concentration may reflect the natural geochemical characteristics of the selected forest soils. Copper is an essential micronutrient involved in plant

physiological processes, and its distribution in forest soils can be influenced by organic matter and biological cycling [15].

3.7 Zinc (Zn)

Zn concentrations ranged from 79.5 to 93.2 ppm, with an average concentration of 86.87 ppm. SS1 exhibited the highest concentration (93.2 ppm), followed by SS2 (87.9 ppm), whereas SS3 showed the lowest value (79.5 ppm). The average Zn concentration was approximately 25.9% of the NIST 2587 reference value of 335.8 ppm. Thus, all three Mulugu forest-soil samples showed substantially lower Zn concentrations than the reference material.

Zinc is an essential micronutrient for plants, and its concentration in forest soils can be influenced by mineral weathering, organic matter interactions and biological cycling. Long-term forest studies have shown that Zn and Cu can behave differently from Fe and Mn because of differences in their retention and biological cycling [16].

3.8 Overall Comparison of the Three Soil Samples

A clear pattern can be observed across the three sampling locations. SS1 consistently exhibited the highest concentration for all seven investigated elements, whereas SS3 consistently exhibited the lowest concentration. SS2 generally showed intermediate concentrations.

This systematic trend is particularly interesting because the three samples were collected within a relatively small

geographical area. The results therefore indicate that even within a limited forest region, measurable spatial heterogeneity can occur in soil elemental composition [17]. The descending order of the average concentrations obtained for the investigated elements was: Fe > Mn > V > Cr > Ni > Zn > Cu

Overall, the EDXRF results demonstrate that the three Mulugu forest-soil samples possess a measurable and systematically varying elemental profile. Fe and Mn were the predominant elements, while Cu and Zn showed considerably lower concentrations relative to the NIST 2587 reference material. The consistent SS1 > SS2 > SS3 trend observed for all seven elements suggests localized geochemical variability within the selected 1-km study area. The results are consistent with the broader understanding that forest-soil trace-element concentrations can be controlled by geological parent material and ecosystem processes, rather than being determined solely by anthropogenic inputs [18].

4. Conclusion

The present study successfully characterized seven elements (V, Cr, Mn, Fe, Ni, Cu, and Zn) in three soil samples from the Mulugu forest region using EDXRF. Among the analyzed elements, Fe was the most abundant element, followed by Mn, Zn, V, Cr, Cu, and Ni based on the calculated mean concentrations. The mean concentrations of V, Mn, Fe, and Ni were close to or above the corresponding values reported for NIST SRM 2587, while Cr, Cu, and Zn were below the reference values. This consistent variation indicated that even within a restricted forest area, differences in soil composition can be observed. The results provide baseline information on the elemental composition of Mulugu forest soils and demonstrate the suitability of EDXRF for rapid multi-element analysis. Further studies involving a larger number of sampling locations, seasonal sampling, soil physicochemical parameters, and appropriate local/regional geochemical background values would strengthen the assessment of elemental distribution and help distinguish natural spatial variability from possible anthropogenic influences.

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