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Pixe Analysis of South Indian Natural Chromite Ore and Rocks

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The complementary and non-destructive PIXE (Proton Induced X-ray Emission) technique has been used to study a variety of Indian chromitites. Thirteen elements, including Cobalt, Nickel, Copper, Zinc, Gallium, Zirconium, Molybdenum, Niobium, and Lead, were established in these chromitites, which could aid in the interpretation of different geochemical conditions and the likely cause of the chromitite matrix development. An effective tool for quickly determining changeable multi-elemental studies is the PIXE technique, which has the benefit of allowing one to collect all the major as well as minor elements occuring simultaneously in pure concentrations. The chemical compositions of ultrabasic rocks and chromite ores of Tekuru, West Godavari district of Andhra Pradesh, a southern state in India, which also belongs to the Eastern Ghats, were analyzed by using PIXE technique. The use and versatality of the PIXE technology for geoscientific methodology research are thus established by the current work.

Keywords: Chromite ores; Proton Induced X-ray Emission; Peninsular India; Nuclear Analytical Technique; Geological Samples.

1. Introduction

Mineral prospecting and geological research both heavily rely on multi-elemental examination of geological samples. In geological research, elements like Al, Si, Fe, Ti, Mn, Ca, K, Mg, P and Na are studied in order to classify ore and rock samples. Analytical techniques will also be used to gather similar information on trace elements. In the current investigation, an element that is present in a sample at values below 1000 parts per million is referred to as a trace element. Obtaining information on trace elements is challenging due to their low concentrations unless an appropriate technique is modified. Numerous techniques are used to describe the geological regions in accordance with potentially intriguing discoveries.

Potentially interesting locations are identified by a traditional geological survey. These identified areas are then investigated in more detail by drilling. It is crucial to get as much preliminary data as possible because final drilling is quite expensive. Accurate elemental analysis is carried out on small samples taken from the drilled core at regular depth levels. Therefore, it's critical to modify practical analytical methods in order to obtain data that is more reliable. Below is a brief overview of several analytical methods used in georesearch to determine the elemental concentrations.

A number of analytical methods like Particle Induced X-ray Emission (PIXE), X-ray Fluorescence (XRF), Instrumental Neutron Activation Analysis (INAA), Electron Probe Micro Analysis (EPMA), Inductively Coupled Plasma Mass Spectroscopy (ICP-MS), and Atomic Absorption Spectrometry (AAS),can be utilized to infer the necessary information about the elemental composition of the geological material. [1-11] Some of these procedures are destructive as they need sample preparation before analysis, or they provide information on only one element, making the samples unsuitable for use in subsequent research. On the other hand, PIXE is unique since the technique requires little to no sample preparation, is non-destructive, thereby allowing for multi-elemental determination.

Johansson et al. (1970) were the first to establish PIXE technique. [12] Trace components in any sample can be identified and quantified using this analytical method, which is comparatively straightforward but effective. PIXE is a unique approach to carry out non-destructive analysis that makes use of characteristic X-rays generated by a proton beam of a few MeV directed at the specimen's surface. Numerous sectors, including environmental, ecological, archeological, biomedical, archeometrical, geological, and nanotechnology, have effectively employed this technique for analysis. [13-22] Numerous applications and examples have been provided that show the PIXE approach favors geological applications because it is among the most appealing techniques for multi-elemental analysis developed recently. [23-27] Since they offer a key to the history of the minerals, it is crucial to understand the major and trace elements as well as the relationships between them in geological samples.

Since archeological and geological materials are valuable and challenging to recover once damaged, PIXE is essentially required for non-destructive investigation. With its well-known characteristics, such as its ability to analyze many elements, sample mass being small, with sensitivity being high, dynamic ranges, along with minimal or no sample preparation, this approach is the preferred method for sample analysis.[28-31]

With ppm sensitivity, PIXE is a non-destructive method to analyze a variety of trace elements quickly. Its sensitivity being high, it is employed in geophysics and geology to examine trace elements in geological specimens. [27, 32-37] Since the Bremsstrahlung noise produced by protons is much smaller than that of electron excitation, using protons provides better sensitivity at lower atomic numbers also. The increasing need for element analysis of extremely smaller sample sizes, such as in geologic materials, has led to the quick acceptance of the PIXE approach as a useful analytical tool. In the current study, we used the PIXE technique to assess this trace element concentration in ultrabasic rocks and chromite ores of the Indian Peninsula.

The trivalent form of the hard, grey metal chromium (Cr) is the most prevalent in nature. Small amounts of hexavalent (chromium (VI)) compounds are also present. Only chromite (FeOCr2O3) has a substantial amount of Cr in it. The ore's highest grade includes roughly

55% chromic oxide; it has never been found in its pure form. The amount of chromium in soil varies by region and the extent of anthropogenic chromium contamination. Chromium concentrations in soils have been shown to range from 1 mg/kg to 1000 mg/kg, with a mean concentration of 14 mg/kg to around 70 mg/kg. Organic matter in the soil can quickly convert chromium (VI) to chromium (III). Given the prevalence of Cr in nature, wind erosion of clay, shale, and several other types of soils may act as a source for chromium in air. Production procedures may be a major source of airborne chromium in nations where chromite is mined. The most significant source of chromium in the air in Europe is most likely the end-point synthesis of chromium compounds.

South Africa (44%), followed by India (18%), Kazakhstan (16%), Zimbabwe (5%), Iran (4%), Finland (4%), and Brazil (2%) are the top producers of chromium ore, with several other nations contributing less than 10% of global production. [38] Dunite, pyroxenite, amphibolite, and other basic and ultrabasic rocks, along with their altered masses, are discovered in India alongside chromite deposits. The Peninsular Indian chromites, found in Karnataka, Tamil Nadu, Orissa, and Andhra Pradesh states, make up the entirety of India's chromium production. Geologically speaking, the Iron Ore Series, Green Schists, and Eastern Ghats are the only places in Peninsular India where chromite deposits may be found. Tekuru, the current study location, is located in the Eastern Ghats, where pyroxenites are linked to chromium-enriched chromite.

With a mean concentration of about 100 ppm, chromium (Cr) ranks the 21st most prevalent component in the crust of the Earth.[39] Cr elements are found in the environment because chromium-containing rocks when break down might get dispersed by volcanic eruptions. The amounts vary from 1 mg/kg to 3000 mg/kg in soil, 5 µg/liter to 800 µg/liter in seawater, and 26 µg/liter to 5.2 mg/liter in lakes and rivers.[40] The relationship between Cr (III) and Cr (VI) highly depends on the location's oxidative characteristics and pH. But in most cases, Cr (III) is the dominating species, [40] though 30 µg of total chromium, of which 39 µg are Cr (VI), can be found in some places in groundwater. [41] Chromium is extracted as the mineral chromite (FeCr2O4). [42] South Africa produces about two-fifths of the world's chromite ores, with significant production also coming from Kazakhstan, Russia, India, and Turkey. Although there are many unexplored deposits of chromite, they are primarily located in Southern Africa and Kazakhstan. [43]

The use of chromium, an essential component along with iron and manganese, is largely responsible for global industrial progress. The main mineral used in the creation of chromium metal, the main ingredient in high-tech machinery and stainless steel, is chromium. Depending on specific ore specifications, it is helpful in the chemical and metallurgical refractory sectors. The study of chromite mineralogy is highly intriguing due of the isomorphous spinal group combinations. Given its significance, it is more crucial to estimate the exact amounts of chromium present in rock samples and chromite ore, including the minor and trace elements. This serves as the study paper's backdrop.

2. Materials and Methods and Methodology:

2.1. Sample collection and Preparation:

2.1.1. Study Area:

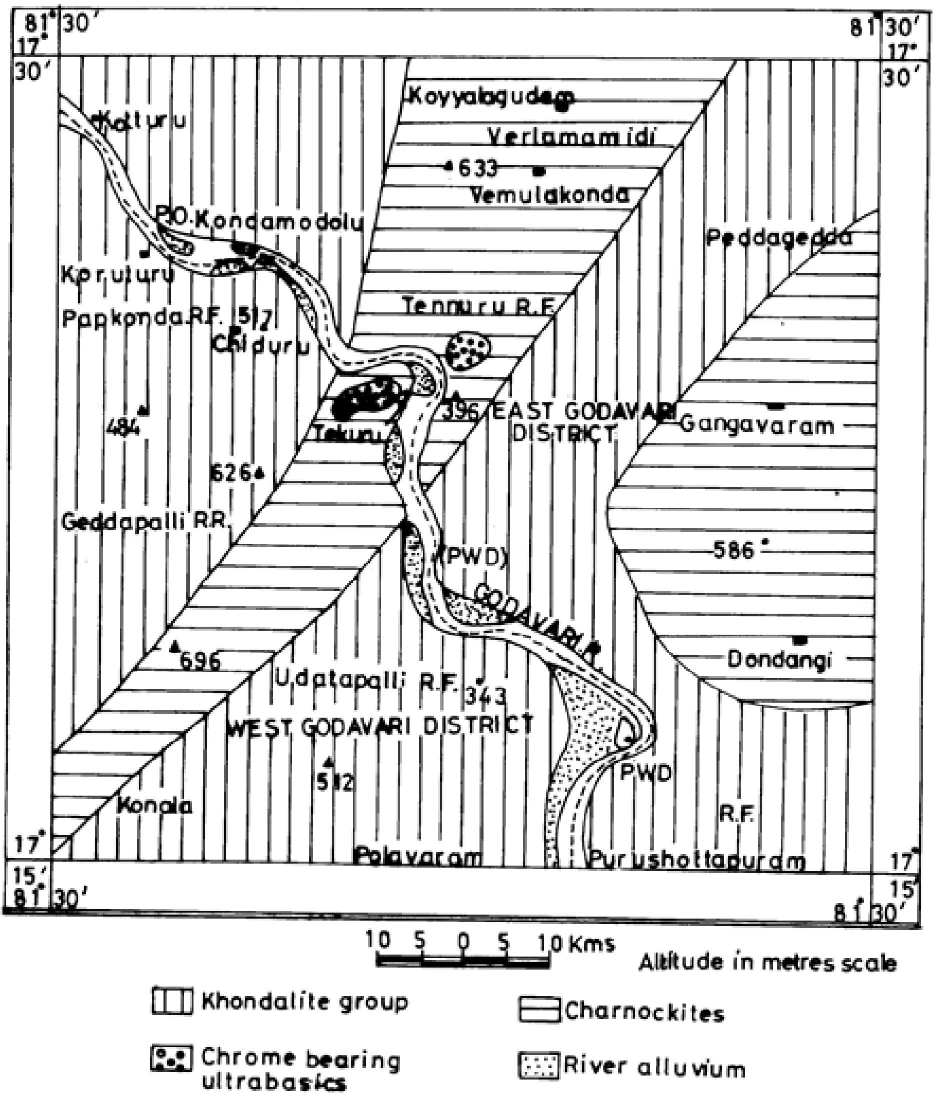

FIG. I. GEOLOGICAL MAP OF TEKURU AND SURROUNDINGS

The rock samples and chromite ore were obtained from a number of Tekuru chromite mines in the West Godavari region of Andhra Pradesh, Eastern India (Fig.1). Broad rivers like the Godavari, which flow southeast, divide the Tekuru region, which is primarily a hilly terrain composed of low ridges and foothills with northeastern spurs. The highest point is Phyljora

Huli, at Δ1343. All of the other rivulets, with the exception of the Tammileru and Godavari rivers, have prolonged dry spells for significant portion the year.

2.1.2. Ore sample:

In a stainless-steel mortar, roughly 200g of chromite ore is first crushed to fine grains using strong strokes. The material that is highly magnetic is then parted as magnetite. The size, shape, form, and color of chromium granules are examined under a microscope in order to separate them. Heavy solvents, such as tetrabromethane, are employed to separate gangue from the ore mass more clearly. After being rinsed with water, the granules are decanted, allowed to dry at room temperature, and made into a powder minus 230 microns. Using an agate mortar, the obtained powder is then ground to a mesh size of -400 microns for roughly 45 minutes each.

2.1.3. Rock samples:

A fresh rock sample of 200g was collected, properly cleaned with water, and then oven-dried. Using a mesh of -170 microns, the sample was broken up into tiny pieces. Coning and quartering were applied to the resulting powder until it was processed in an agate mortar for easy digestion. The analytical work that followed made use of this powder.

In geochemical analysis, contamination during sample preparation is a major cause of inaccuracy. It commonly happens when crushing and grinding, and it can lead to cross-contamination. Cross contamination is avoided in this process by thoroughly cleaning the equipment and pre-contaminating it with the given sample that will be crushed. Utilizing agate for grinding eliminates contamination during the operation.

After the ore and rock samples were cleaned, processed, and separated, the following method was used to turn them into pellets. The binding substance (such as boric acid, etc.) was completely mixed with a small quantity (~150 mg) of rock and ore from each of the sample, which had been finely crushed into powder in a 1:1 by weight ratio. To track the beam current, the samples were combined with 150 mg of high-purity (99.9997%) powdered graphite. These solid mixes were homogenized, compressed into pellets, and then finely powdered. Using a hydraulic press, the resulting mixture was compacted to create a roughly 1.5 mm thick and 13 mm diameter pellet. Then, the prepared pellets that were employed as targets were placed on the target ladder. To collect the characteristic X-rays generated when the collimated beam fell on the samples, a Si (Li) detector kept at liquid nitrogen temperature was employed.

2.2. The details of PIXE Experiment:

A 3 MV pelletron accelerator was employed to conduct the current PIXE experiment at the Institute of Physics, Bhubaneswar, Orissa state, India. The PIXE method has been frequently employed to material analysis utilizing this accelerator. [24, 44-48] A 3 MeV proton beam was used to stimulate the provided samples, and then suspended on a sample holder made of aluminum. Thereafter, the irradiation was conducted under vacuum after placing the target holder placed inside the PIXE chamber. A sample holder made of aluminum held the samples in suspension. After the target holder has been loaded inside the PIXE chamber, the irradiation was conducted under vacuum. In the scattering chamber, the targets on the aluminum holder were angled 45 degrees with regard to the proton beam. The typical X-ray spectra at 90° were collected by an energy dispersive Si (Li) detector (with 160 eV FWHM resolution at 5.9 KeV) corresponding to that beam. For each sample, the average beam current and the total charge collected were noted. To get better statistics, the spectra were collected over a prolonged period of time. A Canberra series MCA was used to record the spectra. Chromium ore and rock samples from Tekuru, in the West Godavari district of Andhra Pradesh State, India—a region that forms a portion of the Eastern Ghats—was analyzed in this study by employing an adaptation of the PIXE technique. The GUPIX software tool was used to compute the amounts of various elements present in each sample. Tables 1, 2, and 3 provide the estimates of concentrations of chromite and other trace elements.

2.3 Data Analysis:

The GUPIX software was utilized for examining the gathered dataset. [49-52] For a particular element of a given atomic number Z, the relation between its characteristic X-ray yield Y (Z) and its concentration Cz in a target matrix is

$$Y\ (Z) = HY1\ (Z)\ C_z Q Є_z\ t_z,$$

Where Y1 (Z) represents the calculated Yield from the database per steradian per unit concentration and unit integrated beam charge; tz is the X-ray transmission through an absorber that is in between the specimen and the detector; Єz the intrinsic efficiency of the Si (Li) X-ray detector; and Q is the beam charge. The instrumental constant H is obtained by multiplying the geometric solid angle of the X-ray detector by any systematic normalizing factor in the charge integration system. A variety of pure single-element standards that emit both K and L X-rays in the energy range of 3–26 KeV and 3 MeV protons were used to measure the instrumental constant H. Therefore, a specified standardization technique that involves the H value, theoretical yield, detection efficiency, and filter transmission value is used to convert the X-ray yield (Y) to elemental concentration.

3. Results and Discussion:

The tandem type pelletron accelerator's 3 MeV proton beam at the Institute of Physics (IOP) in Bhubaneswar, the capital city of Orissa state, India, was utilized to irradiate the chromite ore and ultra basic rock samples that were gathered from several locations in Tekuru. The characteristic x-rays released were employed for determining the elemental concentration of samples. Table 1 lists the estimated primary elements concentrations, with the concentration values expressed as a percentage. Tables 2 and 3 show the minor and trace elemental concentration of chromite ore and ultrabasic rock measured, along with the standard deviation of each. Tables 2 and 3 provide elemental concentrations in µg/g (ppm), with the exception of Ti, V, Mn, Co, and Ni, which have concentrations in percentage.

Together with the concentrations, the experimental error for every measured concentration are shown in the tables above. The number of countings and the peak areas' least-squares fitting are takes into account, the uncertainty in the background subtraction—are the two main factors that determine the errors in PIXE results. Tables 1, 2, and 3 demonstrate that the findings are fully consistent and are within the permissible error limits.

Table.1: Major elements (values in %) determined in chromitite by PIXE.

S.No	CR1	CR2	CR3	CR4	CR5	CR6	CR7	CR8	CR9	CR10
Iron	8.45	12.32	10.42	21.38	14.36	19.28	24.45	15.35	6.40	11.31
Chromium	19.96	14.95	23.88	16.51	12.78	13.72	17.92	31.88	15.90	18.32

Table .2: Minor and trace elements concentrations distribution in chromitite ores – by PIXE method (values in ppm).

S.No.	Ni *	Mn *	V *	Ti *	Zn	Mo	Nb	Pb	Ga	Zr	Cu
CR1	0.41	0.36	0.45	0.96	919 ± 39.51	16 ± 1.40	38 ± 3.11	73 ± 5.69	778 ± 32.67	48 ± 3.79	46 ± 3.86
CR2	1.53	0.32	0.32	0.95	940 ± 40.42	11 ± 0.96	10 ± 0.82	48 ± 3.74	724 ± 30.40	34 ± 2.68	18 ± 1.51
CR3	0.62	0.24	0.42	0.88	382 ± 16.42	08 ± 0.70	6 ± 0.49	21 ± 1.63	516 ± 21.67	41 ± 3.23	34 ± 2.85
CR4	1.43	0.18	0.38	0.51	906 ± 38.95	12 ± 1.05	18 ± 1.47	33 ± 2.57	638 ± 26.79	26 ± 2.05	28 ± 2.35
CR5	0.96	0.21	0.36	0.78	328 ± 14.10	07 ± 0.61	22 ± 1.80	70 ± 5.46	580 ± 24.36	18 ± 1.42	31 ± 2.60
CR6	0.52	0.26	0.28	0.72	248 ± 10.66	05 ± 0.52	28 ± 2.29	54 ± 4.21	732 ± 30.74	16 ± 1.26	22 ± 1.84
CR7	0.56	0.33	0.45	0.92	882 ± 37.92	09 ± 0.79	9 ± 1.38	18 ± 1.40	542 ± 22.68	8 ± 0.63	40 ± 3.36
CR8	0.63	0.31	0.35	0.88	661 ± 28.42	08 ± 0.70	14 ± 1.14	46 ± 3.58	328 ± 13.77	30 ± 2.37	37 ± 3.10
CR9	0.48	0.22	0.40	0.90	761 ± 32.72	08 ± 0.70	11 ± 0.9	51 ± 3.97	676 ± 28.39	19 ± 1.50	25 ± 2.10
CR10	1.76	0.13	0.31	0.32	309 ± 13.28	09 ± 0.79	17 ± 1.39	14 ± 1.09	346 ± 14.53	21 ± 1.65	15 ± 1.26

(* = concentrations are in percentage)

Table .3: Minor and trace elements distribution in ultra-basic rocks – by PIXE method (Values in ppm).

S.No.	Ni*	Mn*	V*	Ti*	Co*	Zn	Ga	Zr	Cu
CR1	1.79	0.27	0.080	0.18	0.01518	77.8 ± 5.83	40.7 ± 2.8	240 ± 16.56	69.3 ± 3.95
CR2	1.53	0.014	0.024	0.34	0.0088	41 ± 3.07	21 ± 1.44	18 ± 1.24	34 ± 1.93
CR3	0.86	0.018	0.029	0.41	0.0096	24 ± 1.80	28 ± 1.93	32 ± 2.20	40 ± 2.28

CR4	1.43	0.031	0.004	0.017	0.0036	15 ± 1.12	11 ± 0.7	16 ± 1.60	27 ± 1.53
CR5	1.96	0.01612	0.030	0.30	0.0132	28 ± 2.10	30 ± 2.07	22 ± 1.51	73 ± 4.16
CR6	0.52	0.0986	0.014	0.14	0.0086	14 ± 1.05	24 ± 1.65	11 ± 0.75	28 ± 1.59
CR7	2.56	0.0094	0.0014	0.013	0.0025	24 ± 1.80	8 ± 0.55	9 ± 0.62	16 ± 0.92
CR8	0.63	0.10	0.0229	0.48	0.0071	8 ± 0.60	18 ± 1.24	14 ± 0.92	31 ± 1.77
CR9	2.04	0.015	0.0026	0.011	0.0122	6 ± 0.45	10 ± 0.6	19 ± 1.31	19 ± 1.08
CR10	1.76	0.0078	0.0014	0.44	0.0092	10 ± 0.75	6 ± 0.41	4 ± 0.27	12 ± 0.68

(* = concentrations in percentage)

Ti concentrations in the samples ranged from 0.013% to 0.96%, while Mn concentrations ranged from 0.0078% to 0.36% (tables 2 and 3). The element Ni, on the other hand, has a rather wide concentration range, ranging from 0.41% to 2.56%. Except for the elements Pb and Cu, that are found in these samples in the tens of ppm range (14-73 ppm and 12-78 ppm, respectively), the concentration of trace elements (tables 2 and 3) exhibit intriguing behavior along with significant variations.

The mean concentrations of nickel (Ni) in Proterozoic-era igneous rock are stated to be higher than 200 parts per million in the literature. [53-54] It has been observed that our samples' nickel (Ni) concentrations are higher than those reported in earlier study on igneous rocks. It shows that the samples we have are igneous and from the Proterozoic era. One other significant geochemical parameter is the zirconium (Zr) concentration, used as a reliable indicator of a province that has been found to be less susceptible to weathering. It is believed that transportation occurring during weathering and any other particular later geochemical event of lesser importance have no effect on Zr concentration. [30, 36, 55-56]. In addition, it is extremely stable, immobile, and seldom breaks down in the environment. Therefore, the Zr content in ore might indicate the component of the parent rock or the solution that it originates from. The Zr content in these samples are very high with a significant variation, which suggests that numerous geological events may have contributed to their origin, formation and subsequent evolution.

We also like to draw readers' attention to the fact that many geologists believe, i.e., when basalt melts, magmatic differentiation produces chromite deposits,[57] that are separated either in the beginning because of the fractional crystallization of the dry magmas or, at the completion, and separated from residual melt that has been enriched with volatile matter, which lowers the chromite crystallization temperature. To comprehend the likelihood of the differentiation process, which might result in the association among different trace metals with important elements, a number of inter-element correlations have been tried based on the findings of the concentration of different elements. For additional comprehension and debate, the extraordinary and extra-ordinary correlations have only been displayed and taken into account. Fe-Co (Fig. 2) and Cr-Ni (Fig. 4) exhibit very good correlations and a little similar behavior patterns, while Fe-Ni (Fig. 3) and Cr-Co (Fig. 5) do not exhibit any particular relationship. In other words, geologically speaking, iron and chromium elements have a sympathetic relationship with cobalt and nickel, while they have an antipathetic relationship with nickel and cobalt, respectively. The latter set of components and the source solution may be differentiated at the same time, as well as simultaneous precipitation for the first two group of elements.

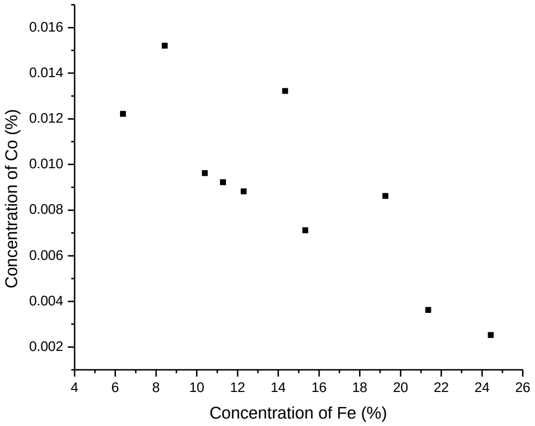

Fig.2: Variation of cobalt (Co) with respect to iron (Fe)

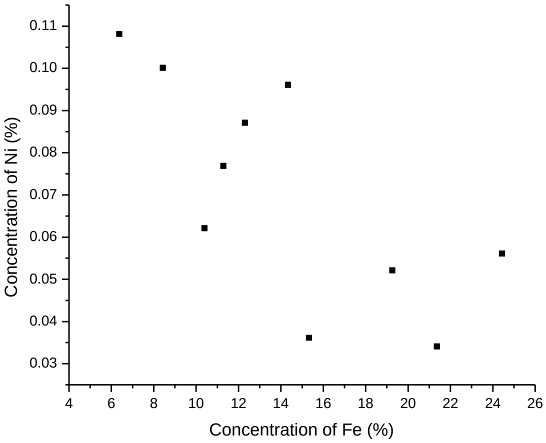

Fig.3: Variation of nickel (Ni) with respect to iron (Fe)

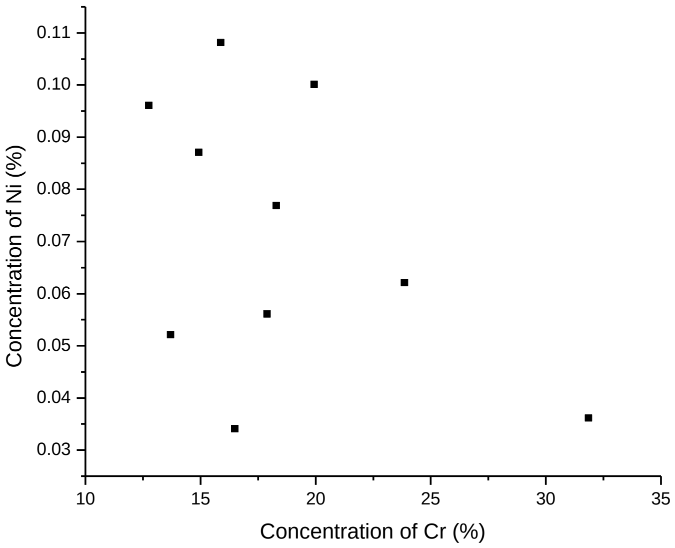

Fig 4: Variation of nickel (Ni) with respect to chromium (Cr)

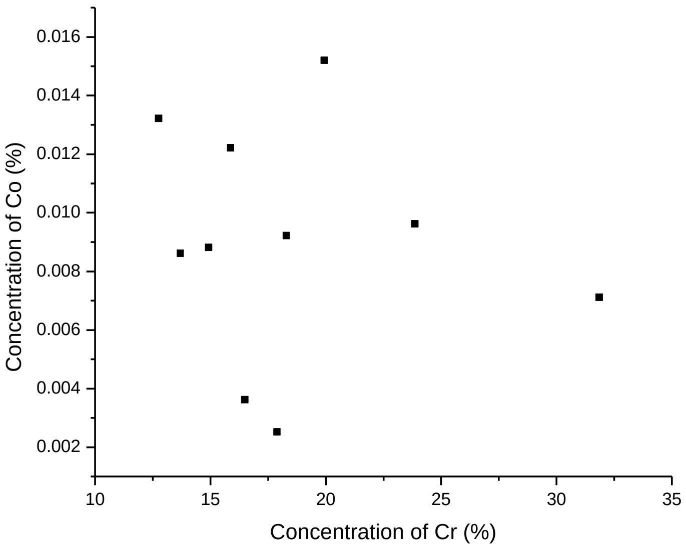

Fig.5: Variation of cobalt (Co) with respect to chromium (Cr)

The mean element concentrations and the ranges from each of these samples have been calculated for comparing the elemental concentration profiles between rock samples and chromite ore. Fig. 6 summarizes the mean and minor trace element concentration of the rock and chromite ore samples. With the exception of Zn and Ga, the minor element concentrations in the rock samples and chromite ore were found to be quite similar. While Co is low in chromite ore samples, Ga and Zn concentrations are relatively higher in chromite ore samples than in rock samples. Table 2 and Figure 6 show that the concentrations of Nb, Mo and Pb that were found in ore samples of chromite, but not in host rocks, viz. gabbro, dunite, and pyroxenite. The findings mentioned above may be caused by variations in various factors, namely temperature, pressure, energy, and gravity, during precipitation or formation of the rock samples and chromite ore. However, the main root cause and the involved mechanism behind the absence or presence of all these elements in a particular type of chromite ore and a rock sample has not been established yet. Therefore, these components are crucial for identifying different kinds of rock and mineral samples.

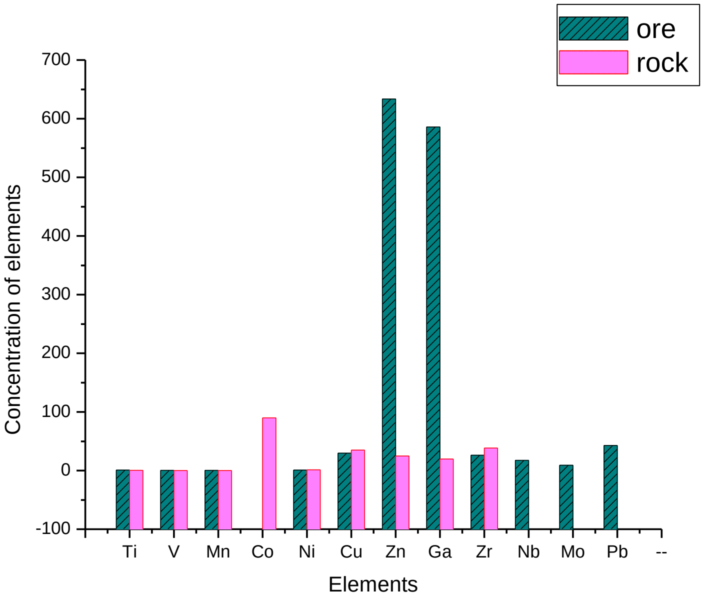

Fig.6: Mean minor and trace elemental concentration in chromite ore and rock samples

4. Conclusion:

Thus, the complementary and non-destructive PIXE approach has been used to study a selected number of Indian Peninsular chromitites. These chromitites were found to include thirteen elements: Ni, Co, Cu, Zr, Ga, Zn, Nb, Mo, and Pb. These elements might be utilized for interpreting various conditions geochemically and the likely causes for the inceptions in the chromitite matrix. It was noticed that Mn, Ni, and Ti are available in large amounts than other trace elements. It's intriguing that the nickel concentration suggests that these chromitites originated during the Proterozoic era. Furthermore, it is important to note that the amount of zirconium in this particular geological belt reflects the impact of numerous geological processes. In order to quantify trace elements in high-grade ores, the study recommends using PIXE. However, as this work is still in its early stages, more thorough research could help us better comprehend the role of trace elements throughout the Proterozoic period.

Certain natural chromitites were characterized using the accelerator-based nuclear method of PIXE analysis. The primary uses of these minerals are in science and technology. From these samples, the concentrations of a number of tracer, major, and minor elements were found. The chromitites are then obtained from their elemental concentrations. As a non-destructive method of analysis that facilitates complete characterization, the PIXE approach thus aids in figuring out the chemistry and mineralogy of the chromitites. In order to characterize geological materials, particularly when a number of the elements are of specific interest, the current study recommends the use of the PIXE technique.

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