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Characterization of rare earth ores from Ambatofinandrahana by X-ray diffraction and Zetametry for their bioleaching

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Abstract (< or = 250 words)

Bioleaching is an extraction technique that uses microorganisms as a leaching agent. This method requires several bacteriological, physico-chemical and mineralogical analyzes of the ores, which in this study are the rare earths. The rare earths include the seventeen elements of the periodic table, including fifteen of the lanthanide family with yttrium and scandium. Two important parameters for the experimental process of rare earth bioleaching are studied, the first refers to characterization by X-ray diffraction (XRD). This is a valuable tool to identify the mineralogical composition of the ore, to determine the distribution rare earths in the ore and to follow the evolution of the leaching process. The second is the determination of the surface properties of the samples to be treated. This property is evaluated by the zeta potential technique. In order to choose and know the type of bacteria to be used, we need to know the surface charges and surface properties characterizing each ore to be leached. In this study, the samples come from the Ambatofinandrahana site. Ten samples were characterized by zetameter and XRD. The results show that all ores are negatively charged according to zetametry, hence the need for positively charged bacteria. As for the XRD results, the ores are mainly composed of bastnaesites and monazites. These two parameters make a major contribution to the upstream characterization of our ore samples prior to their actual bioleaching.

Keywords

Rare metals, surface charge, environment, biological leaching, zeta potential, XRD

1- Introduction

Today, the consumption of mineral raw materials required for new technologies is increasing exponentially. Rare earths are one of these indispensable raw materials. According to the International Union of Pure and Applied Chemistry (IUPAC), rare earths include the seventeen elements of the periodic table, fifteen of which belong to the lanthanide family, along with yttrium and scandium **[1].** Obtaining these elements requires a long and complex processing chain, from the mine to the final products used in metallurgy, magnets, catalysts, polishing, glass, lighting and ceramics, mobile phones, flat-screen TVs and more **[2].** In general, current methods used by industrial giants rely on leaching with strong acids to extract these precious metals. When approaching the characterization of rare earth ores and their bioleaching, it is crucial to take into account the global context of **climate change**. This approach is energy-intensive, generating significant pollution for the planet. In this respect, the recent bioleaching method is emerging. It falls into the category of innovative rare earth production technology. It is becoming increasingly important as a green, environmentally-friendly alternative technology. It enables ores such as bastnaesite and monazite to be processed and extracted cleanly. In this process, ore minerals are dissolved by the acids of micro-organisms **[3].** Biological leaching of rare earths requires a number of procedures prior to its implementation, both in academic settings and at laboratory, pilot or even industrial scale. These include the identification and characterization of raw ores by physicochemical means (characterization by X-ray diffraction or XRD, scanning electron microscope or SEM...) and bacteriological means (identification of bacterial strains, measurement of the performance of isolated strains...) **[4].** To successfully carry out biological leaching of rare earths, these multiple phases are essential.

The present research aims to characterize some rare earth ores from Ambatofinandrahana in Madagascar before their bioleaching by previously isolated bacteria using two techniques. The first relies on X-ray diffraction to determine the minerals present in each sample. This technique provides information on the feasibility or otherwise of rare earth bioleaching, as well as on the possibility of assessing the dissolution of minerals by bacteria. It also provides an idea of the evolution of the crystalline structure of each experimental phase. The second technique consists in determining the surface properties of the samples to be processed using the zeta potential technique. To gain a better understanding of the different mineralogical compositions of our samples, and to refine the reliability of the data, inorganic chemical analyses are carried out using inductively coupled plasma atomic emission spectrometry (ICP-AES) at Morocco's National Office of Mines and Hydrocarbons

(ONHYM). The aim is to complete the characterization upstream of the biological leaching of rare earths.

2- Methods

2.1. Materials

The ten samples (Figure 1) used in this study come from rare-earth ores (Bastnaesites) collected at various locations in Ambatodinandrahana Madagascar. The areas concerned are Vohiniariana (samples VOH S1 and VOH S2), Marovoalavo (samples MRV1d and MK_MRV1), Betrandraka (samples BETRAN1 and BETRAN3) and Bagabona (samples BP1P, BP1Pu, BG6 and BG Roche).

2.2. Technical materials

The equipment used depends on the type of characterization involved. In this case, the present research relies on the use of an X-ray Diffractometer (Figure 2) and a Zetameter for physical characterizations (Figure 3). In addition, to complete the data on characterization results, chemical analyses are carried out in the laboratory of Morocco's National Office of Mines and Hydrocarbons (ONHYM). These analyses are based on the Inductively Coupled Plasma Atomic Emission Spectrometry (ICP-AES) technique, with a detection limit of 5 ppm (Figure 4).

2.3. Method

2.3.1. XRD analysis of samples

Ten different ores were characterized by XRD at the Center for Scanning Electron Microscopy and X-Ray Diffraction of the Faculty of Sciences of the University Abdelmaleck Essaâdi, Tetouan of Morocco. For this purpose, we use an X-ray diffractometer (Brucker D8 ADVANCE). Measurement parameters are: 3 to 80° with a step size of 0.02° and a counting time of fifteen seconds (15s) per step. The raw data from the XRD is processed using Highscore Plus Version 3.0.5 software.

2.3.2. Zeta potential measurements

The preparation and determination of the zeta potentials of the ten rare earth ores is carried out in the Chemistry Department of Research Laboratory No. 4 at the Tétouan Faculty of Science.

A Malvern-type zetameter is used for the experiments (Figure 3). The aim here is to determine the surface charge of colloidal particles. Indeed, after preparing the colloidal particles in demineralized water and with a suspension particle concentration of between 1 and 10 mg/ml, we opted for a two-minutes reading per ore. This operation is designed to remove any dirt or debris that might interfere with the measurement.

3- Results

3.1. XRD analysis

With samples previously ground less than 80 µm, the results obtained from the established XRD characterization are presented in Figures 5 to 14.

The results of the chemical analyses confirm the presence of rare-earth elements followed by some mineral heterogeneities in the samples. Tables 1, 2 and 3 and Figures 15 and 16 show the distribution of these elements.

3.2. Zeta potential measurements

The three measurements carried out, together with the average and corresponding charges on each particle surface, are summarized in Table 4 and illustrated in Figure 17.

This shows that all samples have negative charges on their respective surfaces.

4- Discussion

According to S.I.Levy, before choosing a separation technique, it is necessary to understand the composition of the mixture. Generally speaking, the type of mineral used for extraction will provide useful information. Some minerals are also known to be rich in elements from one or more subgroups. The cerium group is known to predominate in some minerals, while the yttrium group predominates in others. **[5]**

4.1. XRD and additional chemical analysis data

Identification by XRD reveals the main mineralogical compositions of the samples. These correspond to bastnaesite (samples VOH S1, BETRAN1, BG R, BP1P, BP1Pu and VOH S2), monazite (sample BG6) and other mineral types such as those present in samples BETRAN3, MRV1d, MK_MRV1. These were confirmed by combining the data obtained from ICP-AES analyses and those received from XRD (Figures 5 to 14, 15 and 16). Indeed, these samples are predominantly composed of elements such as Ce, F, CO3, La, with some cortège elements for bastnaesites where the basic formula is (Ce, La, Th)(CO3)F; Ce, Nd, P, La. Other groups are also observed for monazite, with the basic chemical formula (Ce, La, Th) PO4.

It is important to note that Fluorine elements do not stand out in the chemical analyses carried out. However, they are visible in the XRD identifications. Bastnaesite is therefore present in our samples. Comparing our data with previous mineralogical studies carried out at the same sites in Ambatofinandrahana, notably that of Rasoamalala, (2009), the rocks generally comprise **[6]:**

- syenites and microsyenites,
- granites and microgranites,
- as well as gabbros composed of two textural groups including :
  - the granitic texture, containing feldspars (plagioclase in subautomorphic crystals and potassic feldspars and xenomorphic microclines); altered xenomorphic amphiboles (of the green hornblende type); xenomorphic phlogopite crystals (associated with opaque minerals and green hornblendes) ;
  - the coronite texture of a heteradcumulate formed mainly of olivine (cumulus) cemented by clinopyroxene.

In addition, similar research in this area has highlighted two main deposits at Ambatofinandrahana. One concerns bastnaesite and the other monazite.

According to **Rasoamalala, 2009 ; Rasoamalala et *al*., 2014**, bastnaesites are distributed in locations such as Andakantany, Ankazohambo, Itorendrika, Lesada, Vohininina, Betrandraka and Sahafa **[6] [7]** . Our chemically analyzed samples confirm these rare-earth locations. These samples are VOH S1 and VOH S2 (for Vohinirina), BETRAN1 and BETRAN3 (for Betrandraka).

As for monazites, they are concentrated in Marovoalavo (site of origin of our two samples): MRV1d and MK_MRV1 and Andoharano. The results of our chemical analyses also confirm this.

Thus, knowledge of these different parameters makes it possible to anticipate the rare earth bioleaching experiment. We now need to select the actinomycete micro-organisms best suited to our study. More specifically, we need to focus on the ability of the chosen micro-organisms to leach rare earth ores while delivering an efficient extraction yield of these noble elements.

4.2. Zeta potential

According to these results (see Figure 8 and Table 4), the mean zeta potential is equivalent to -14.37 mV. This means that the mean zeta potential of the samples is negative. Furthermore, the median zeta potential is -14.28 mV. This indicates that 50% of samples have a zeta potential less than or equal to -14.28 mV. The standard deviation of 2.92 mV implies a deviation of 2.92 mV around the mean zeta potential.

The maximum zeta potential of -11.57 mV means that one of the VOH S2 samples (Ambatofinandrahana) has a zeta potential greater than -11.57 mV. Knowing the zeta potential in rare earth bioleaching using bacteria can help improve the efficiency of the process. Zeta potential measures the surface charge of a particle, and can affect how the particle interacts with other particles and with bacteria. So, by determining the zeta potential of the ore particles studied, it becomes possible to select the right bacteria for the bioleaching process. The bacteria then have a greater chance of binding to the particles and leaching the rare earths from the ore **[8].**

In our case, the surfaces of all ten samples turned out to be negatively charged. Indeed, in general, bacteria possess a negative charge and are therefore more likely to bind to particles with a positive zeta potential **[9].** Furthermore, a negative zeta potential means that rare earth ore particles possess a negative charge. In this context, the binding of bacteria to particles and the leaching of rare earths from the ore appear more complicated **[10].**

5- Conclusion

The aim of this study is to identify the rare earth ores at Ambatofinandrahana before carrying out the actual bioleaching experiment. Various physicochemical characterizations were carried out, in particular by X-ray diffraction (XRD), determination of particle surface charges by zeta potential and complementary chemical analysis data by ICP-AES. The results obtained reveal that the different main mineralogical compositions of the samples comprise mainly bastnaesites and monazites, with some mineralogical suites present in the samples. In addition, the zeta potential of the surfaces of the ten samples are all negatively charged. These varied physico-chemical characterization data enable us to determine the prerequisites for our rare earth biological leaching experiment, and to better select the micro-organisms to be used to carry out the extraction. Bacteria are generally negatively charged and are more likely to bind to particles with a positive zeta potential. To this end, zetametry leads to the selection of the right bacteria for the bioleaching process. However, additional upstream parameters may also be required to ensure proper extraction of the desired metals.

6- Acknowledgements

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Table 1: Additional data results-Analysis by ICP-AES (major elements)

Samples	Results in %											
	SiO_2	Al_2O_3	CaO	Fe_2O_3	K_2O	MgO	MnO	Na_2O	P_2O_5	TiO_2	P-Feu	S
BG6	2,70	2,42	0,87	6,27	0,00	0,10	0,00	0,00	10,53	0,01	8,85	0,023
BP1Pu	8,64	1,32	0,43	1,19	0,28	0,24	0,00	0,01	2,43	0,20	17,06	0,129
VOH S1	1,52	0,28	0,12	0,99	0,07	0,13	0,00	0,00	0,57	0,00	18,20	0,035
MK_MRV1	1,49	0,21	0,10	0,41	0,04	0,17	0,00	0,00	1,08	0,39	20,05	0,003
BG R	70,09	2,21	0,06	1,32	0,34	0,08	0,29	0,02	0,49	0,06	5,63	0,091
BETRAN3	61,40	14,56	0,09	1,58	5,34	0,15	0,03	4,83	0,03	0,13	1,47	0,003
BETRAN1	7,24	0,22	0,22	0,20	0,03	0,10	0,00	0,00	2,31	0,00	17,80	0,021
VOH S2	1,75	0,30	0,12	1,13	0,06	0,13	0,00	0,00	0,73	0,00	20,41	0,011
BP1P	6,52	1,02	0,46	2,02	0,28	0,04	0,00	0,00	2,92	0,32	16,62	0,125
MRV1d	49,73	16,82	0,06	6,17	9,08	0,73	0,14	0,75	1,21	0,43	4,95	0,013

(Source: ONHYM Laboratory, 2023)

Table 2: Additional data results - Analysis by ICP-AES (minor elements)

Samples	Results in ppm												
	As	Co	Cu	Nb	Ni	Pb	Ta	Th	U(*)	Zn	Zr	Sc	Y
BG6	1562	47	111	28	41	3,06	<10	226	1103	248	31	12	725
BP1Pu	1588	13	18	125	<5	0,22	<10	830	3091	79	<5	<5	252
VOH S1	782	44	37	41	34	0,47	<10	273	2295	282	10	<5	82
MK_MRV1	47	5	<5	73	<5	0,12	<10	<10	<10	18	<5	<5	276
BG R	396	<5	15	35	17	0,49	10	943	1757	64	27	<5	179
BETRAN3	30	<5	<5	<10	8	0,03	<10	<10	20	20	22	<5	<5
BETRAN1	2969	<5	9	92	<5	0,64	<10	<10	<10	24	<5	<5	112
VOH S2	264	26	24	102	<5	0,17	<10	<10	<10	48	<5	<5	211
BP1P	1744	7	9	160	<5	0,22	<10	830	3091	79	<5	<5	280
MRV1d	123	13	15	48	17	0,26	<10	207	36	112	73	<5	87

(Source: ONHYM Laboratory, 2023)

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Table 3: Supplementary Data Results - Analysis by ICP-AES (Rare Earth Elements)

Samples	**Ce**	**Dy**	**Er**	**Eu**	**Gd**	**Ho**	**La**	**Lu**	**Nd**	**Pr**	**Sm**	**Tb**	**Tm**	**Yb**
	%	ppm	ppm	ppm	ppm	ppm	%	ppm	%	%	%	ppm	ppm	ppm
BG6	17,03	194	108	438	2040	44	13,28	5	4,69	1,62	0,62	27	11	32
BP1Pu	27,06	97	33	244	1947	9	20,52	<5	5,59	2,03	0,58	<5	<5	14
VOH S1	19,45	52	20	163	1494	6	15,25	6	4,17	1,52	0,41	<5	<5	5
MK_MRV1	29,40	149	62	518	2373	18	20,92	<5	6,90	2,38	0,70	<5	<5	7
BG R	4,46	64	22	149	443	8	3,33	<5	1,04	0,38	0,14	<5	<5	6
BETRAN3	0,05	<5	<5	<5	<5	<5	0,04	<5	0,02	0,00	0,04	<5	<5	<5
BETRAN1	30,47	59	15	200	1647	<5	26,18	<5	5,04	2,04	0,38	<5	<5	5
VOH S2	31,97	121	44	427	2406	11	23,95	<5	6,63	2,39	0,60	<5	<5	<5
BP1P	26,82	112	39	290	1996	11	20,79	<5	5,23	2,04	0,47	<5	<5	15
MRV1d	1,53	23	9	30	157	<5	1,07	<5	0,36	0,13	0,10	<5	<5	5

(Source: ONHYM Laboratory, 2023)

Table 4: Results of the mean value of the zeta potential of each rare earth ore

Samples	**Zeta potential** ZP (mV)
BG6	-16,53
BP1Pu	-14,30
VOH S1	-14,57
MK_MRV1	-11,77
BG R	-14,73
BETRAN3	-11,90
BETRAN1	-12,03
VOH S2	-11,57
BP1P	-14,40
MRV1d	-14,03

(Source: Chemistry Department Laboratory, Tetouan, 2023)

Figure 1: The different rare earth ores of Ambatofinandrahana

Figure 2: X-Ray Diffraction device used (Tétouan University, 2023)

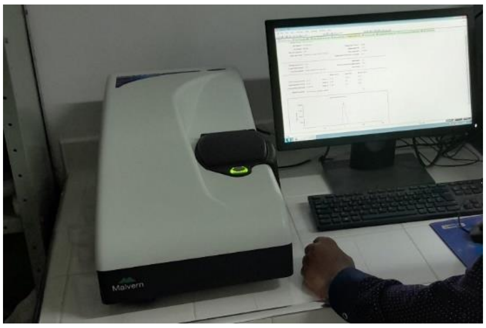

Figure 3: Malvern-type zetameter used (Tétouan University, 2023)

Figure 4: ICP-AES (ONHYM, 2023)

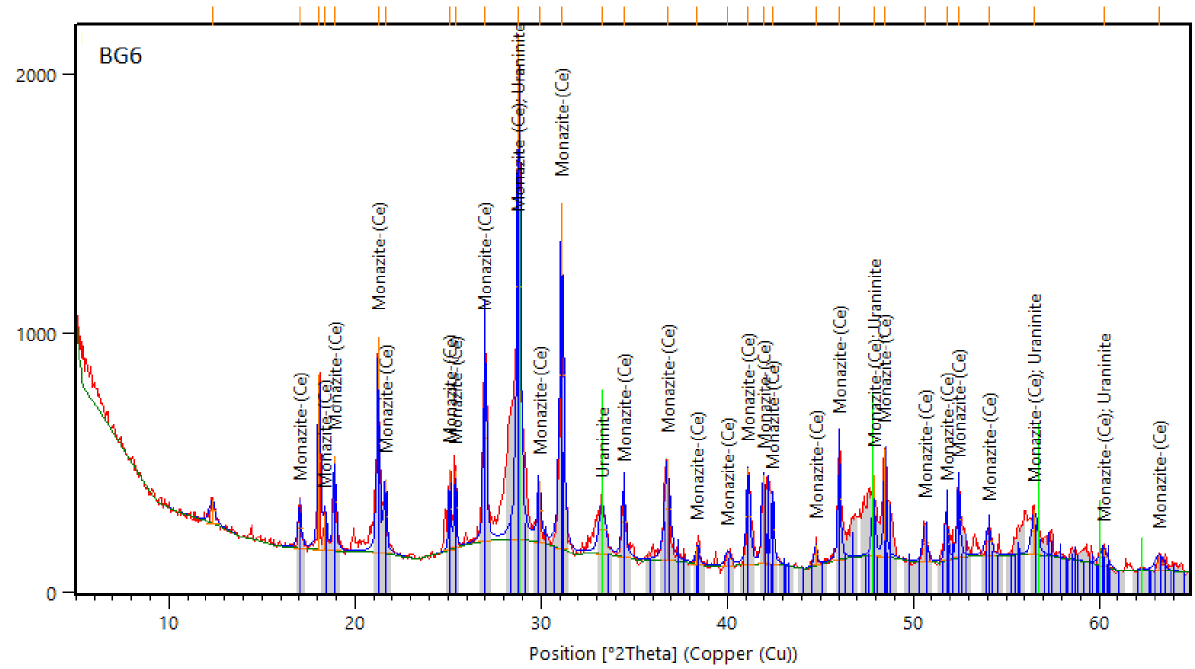

Figure 5: Highscore Plus results from XRD data (Sample BG6)

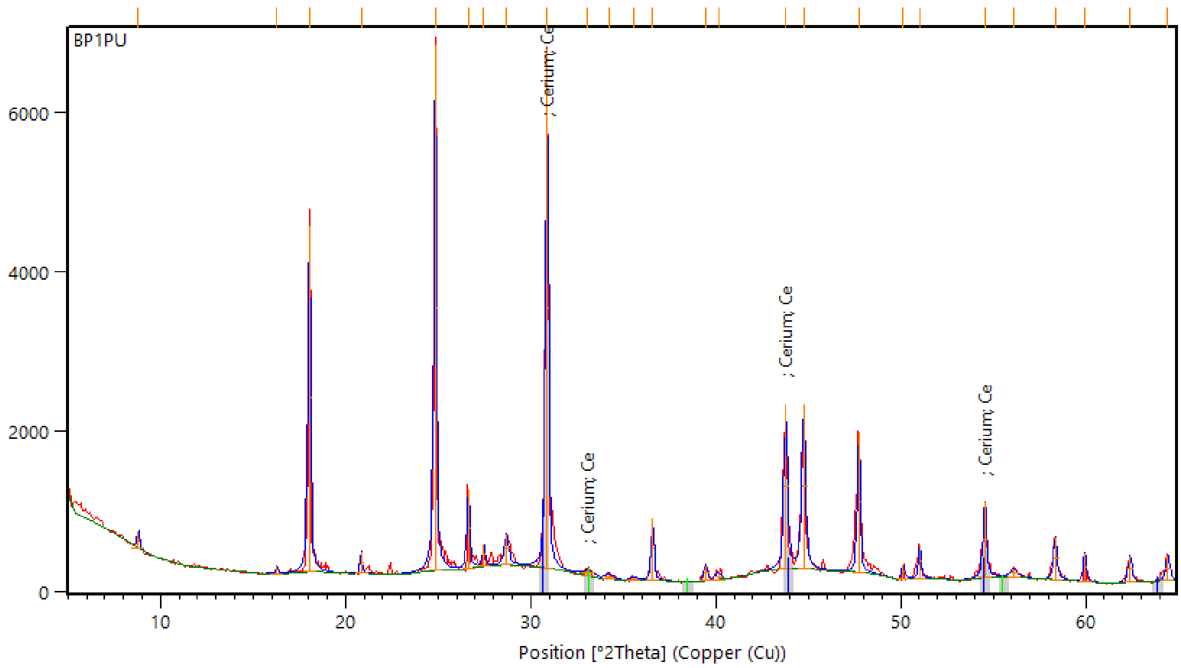

Figure 6: Highscore Plus results from XRD data (BP1Pu sample)

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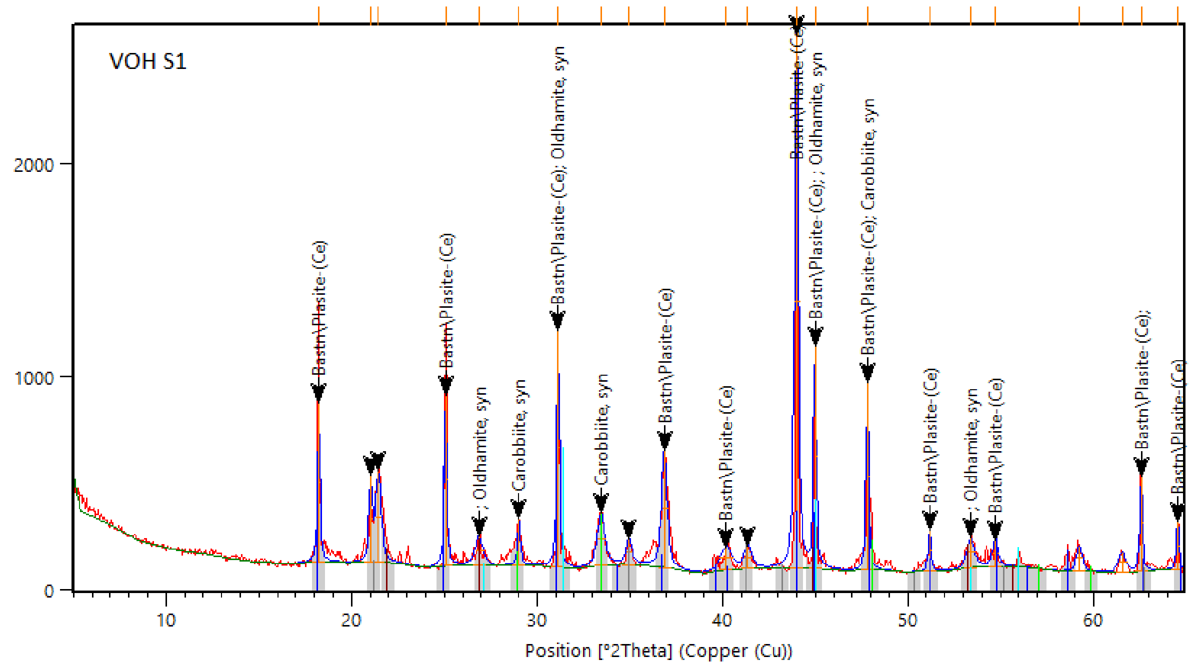

Figure 7: Highscore Plus results from XRD data (VOH S1 sample)

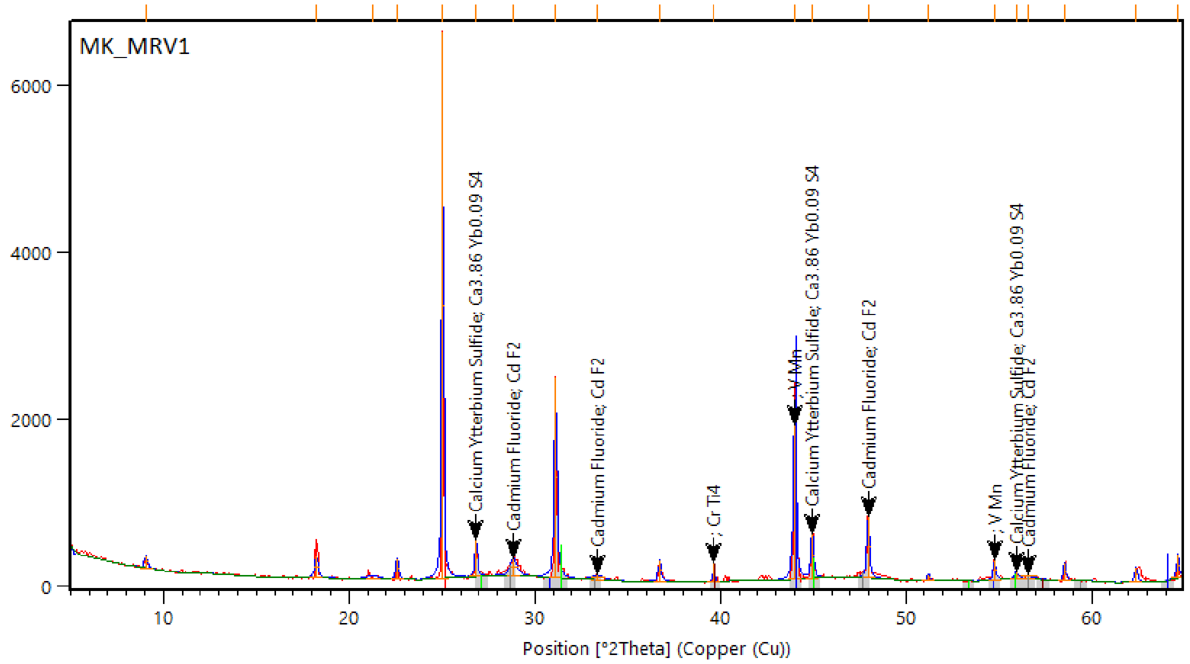

Figure 8: Highscore Plus results for XRD data (Sample MK_MRV1)

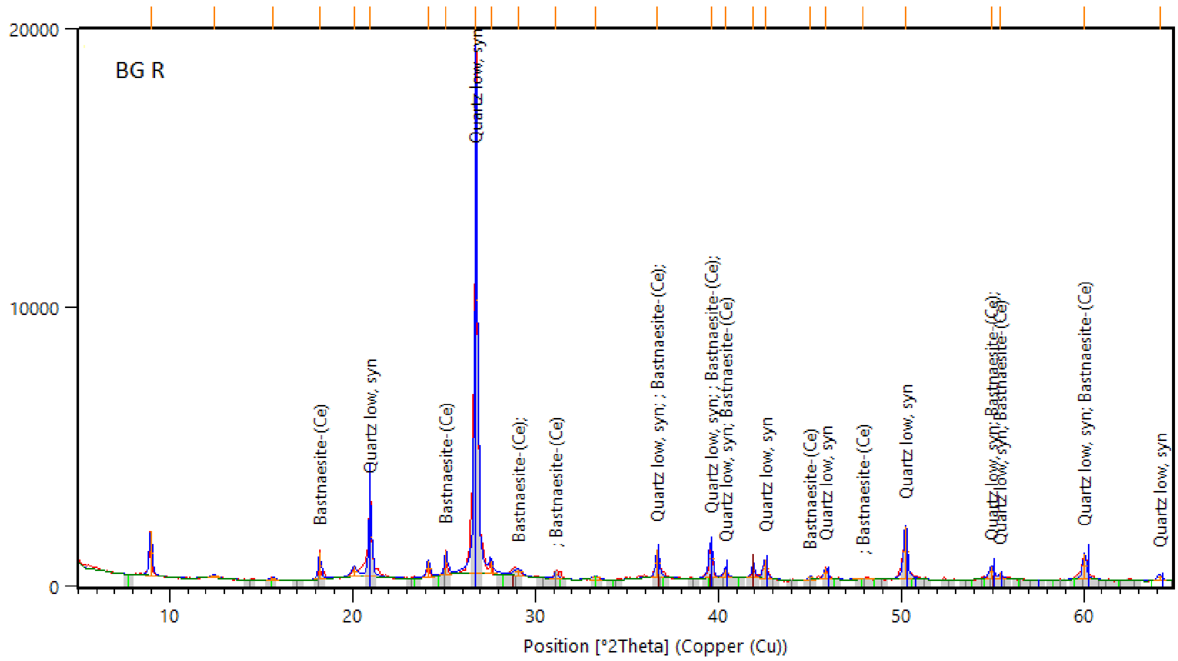

Figure 9: Highscore Plus results for XRD data (BG R sample)

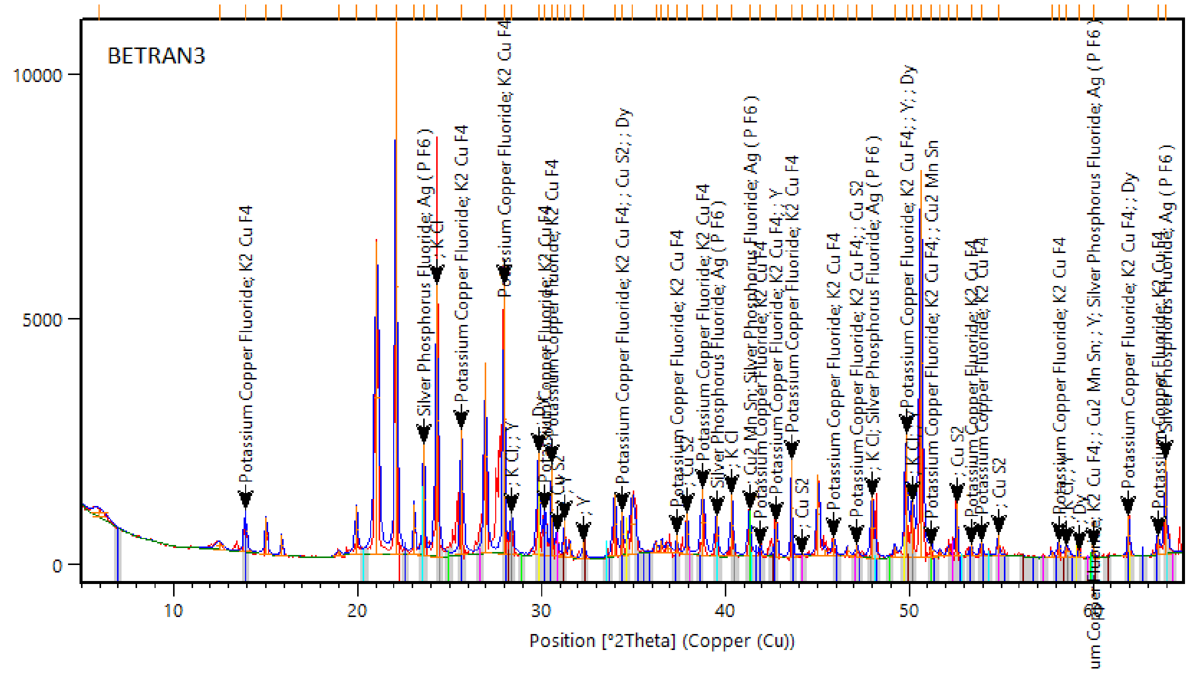

Figure 10: Highscore Plus results from XRD data (BETRAN3 sample)

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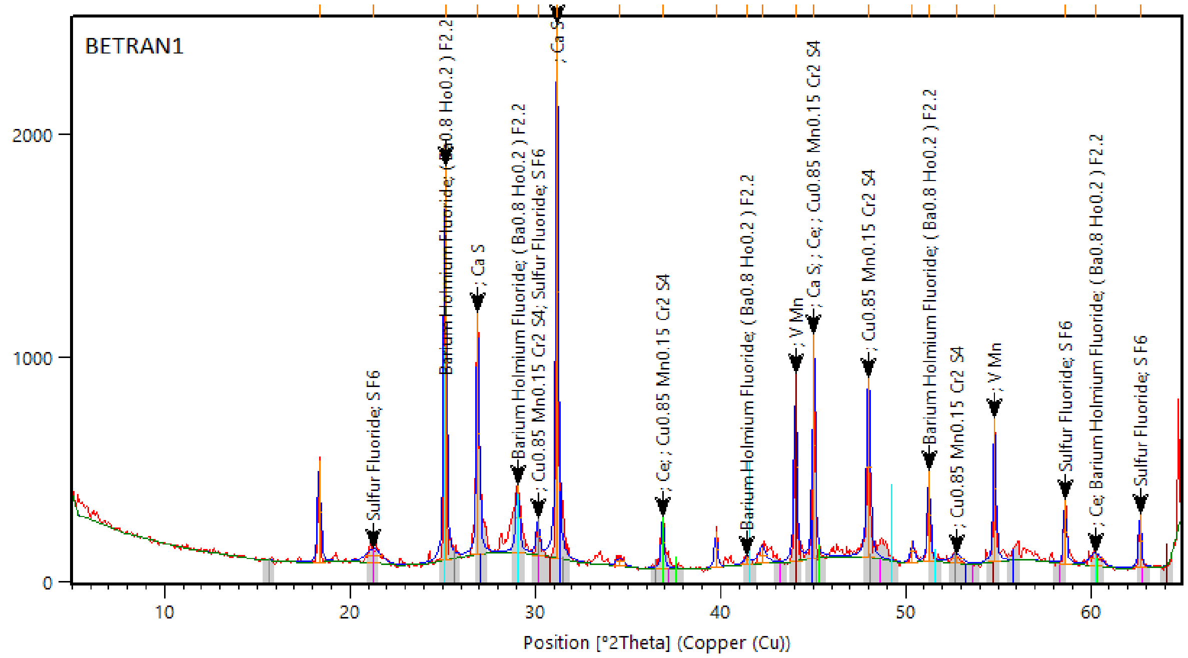

Figure 11: Highscore Plus results from XRD data (BETRAN1 sample)

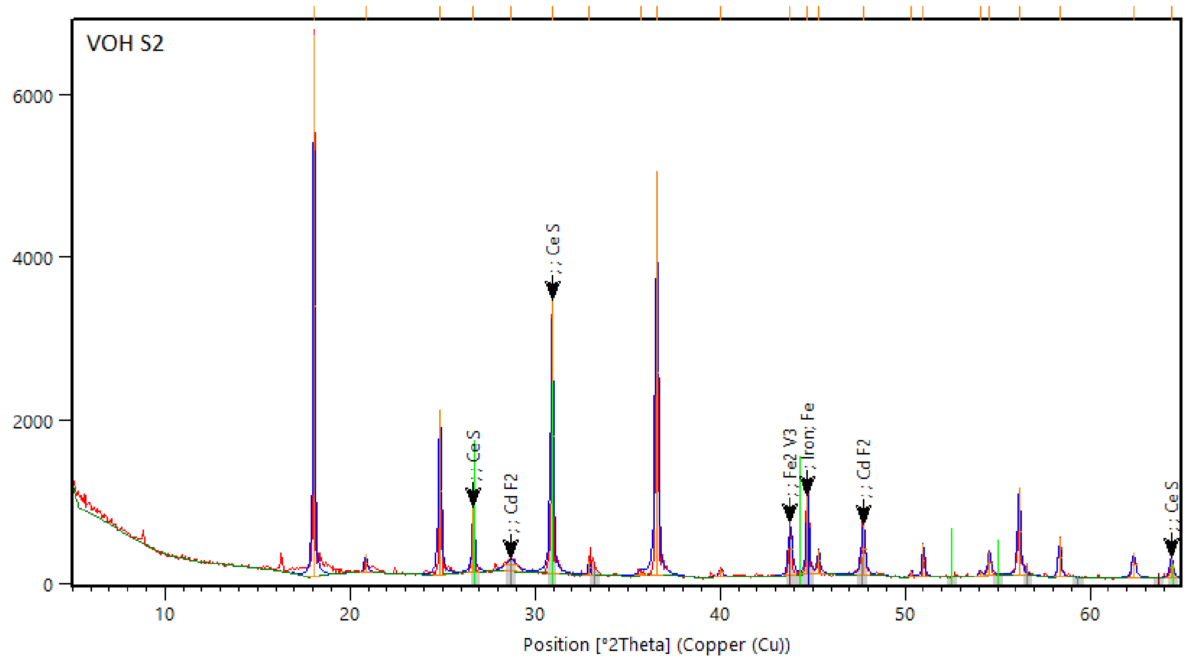

Figure 12: Highscore Plus results for XRD data (VOH S2 sample)

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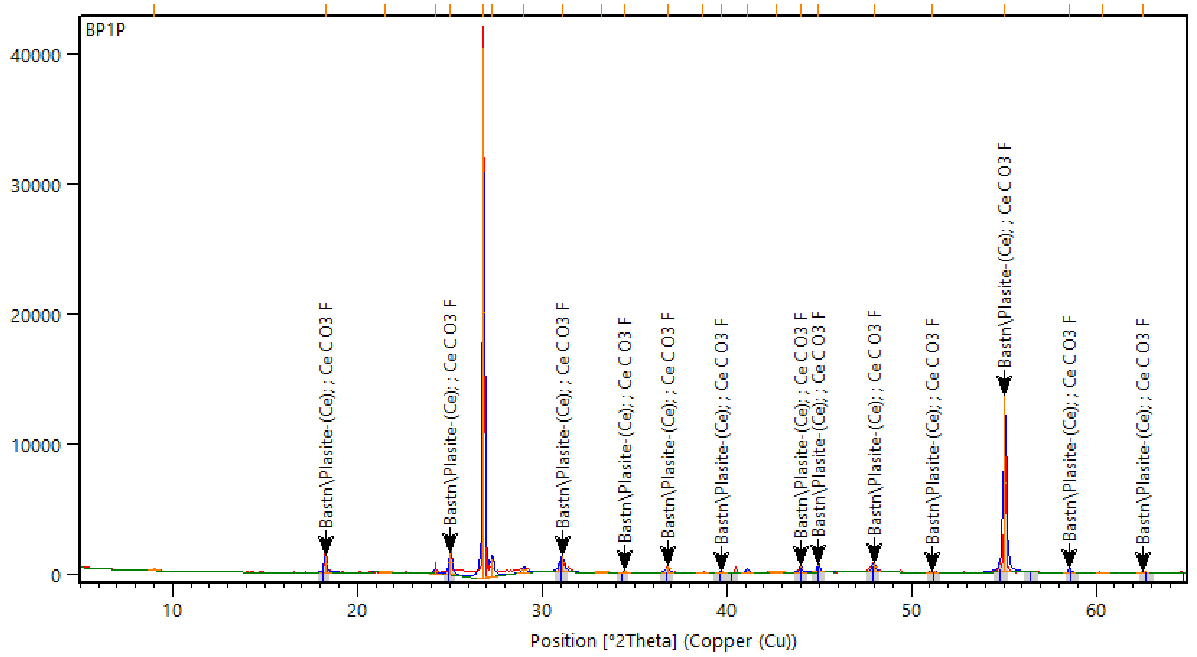

Figure 13: Highscore Plus results for XRD data (Sample BP1P)

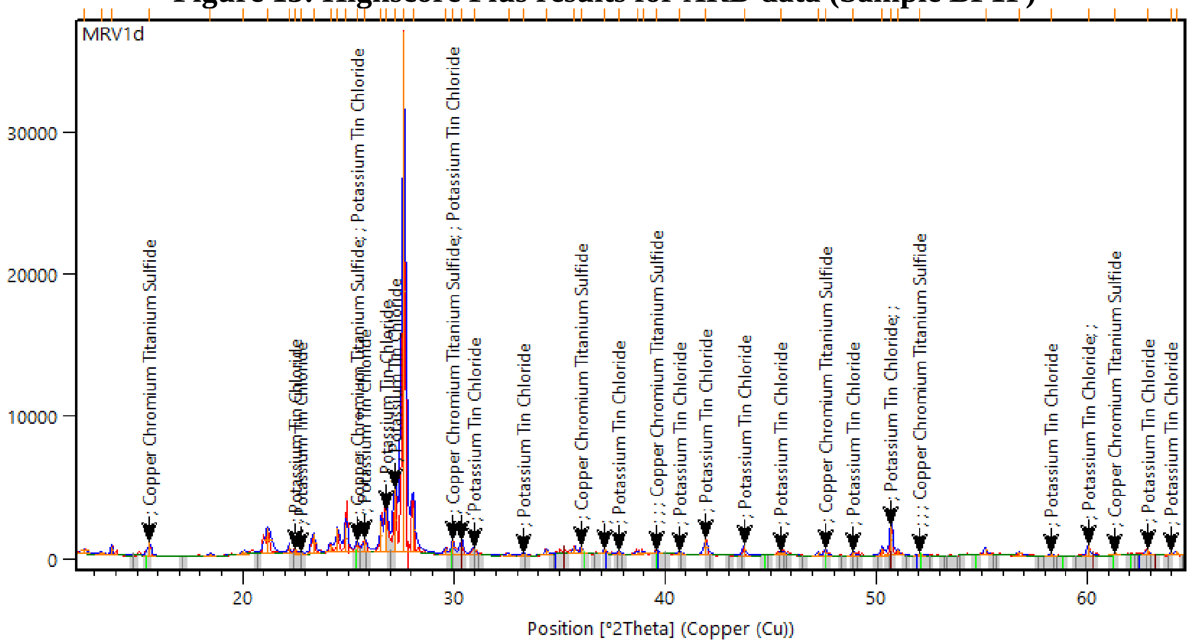

Figure 14: Highscore Plus results for XRD data (MRV1d sample)

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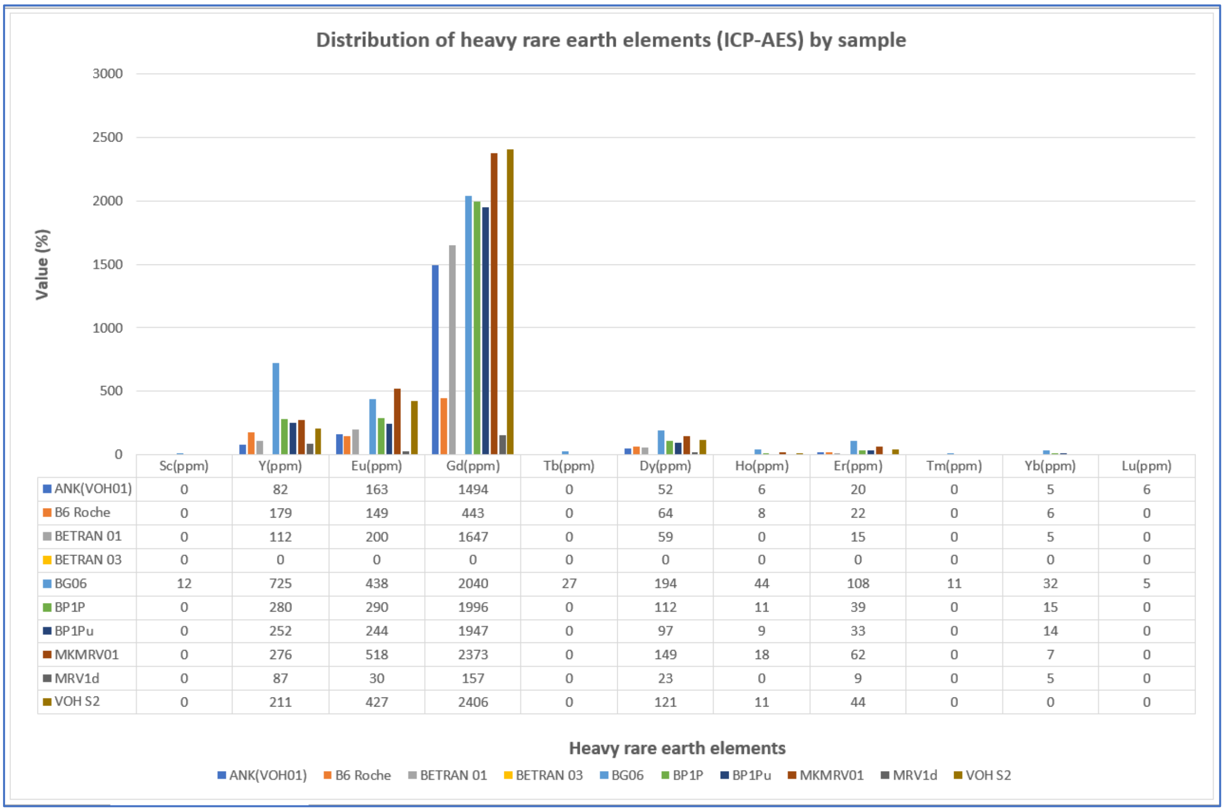
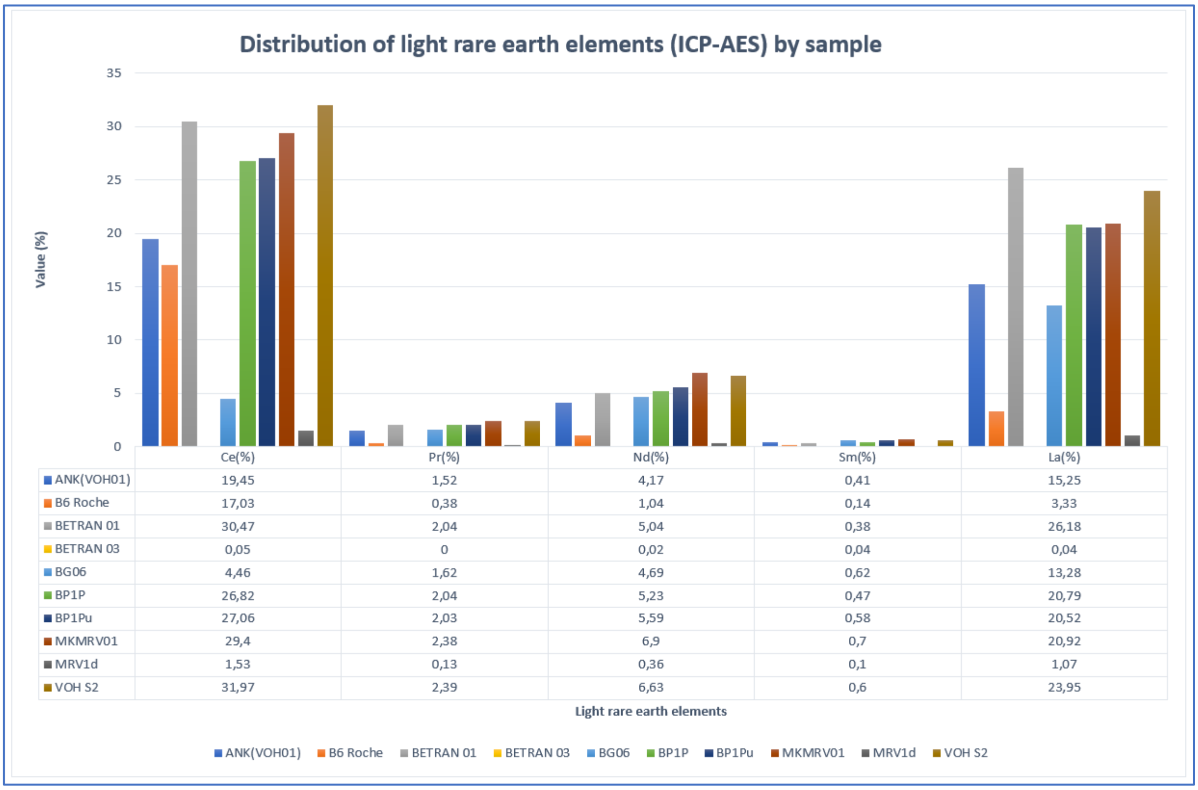


	Sc(ppm)	Y(ppm)	Eu(ppm)	Gd(ppm)	Tb(ppm)	Dy(ppm)	Ho(ppm)	Er(ppm)	Tm(ppm)	Yb(ppm)	Lu(ppm)
ANK(VOH01)	0	82	163	1494	0	52	6	20	0	5	6
B6 Roche	0	179	149	443	0	64	8	22	0	6	0
BETRAN 01	0	112	200	1647	0	59	0	15	0	5	0
BETRAN 03	0	0	0	0	0	0	0	0	0	0	0
BG06	12	725	438	2040	27	194	44	108	11	32	5
BP1P	0	280	290	1996	0	112	11	39	0	15	0
BP1Pu	0	252	244	1947	0	97	9	33	0	14	0
MKMRV01	0	276	518	2373	0	149	18	62	0	7	0
MRV1d	0	87	30	157	0	23	0	9	0	5	0
VOH S2	0	211	427	2406	0	121	11	44	0	0	0

Figure 15: Heavy rare earths in the samples studied



	Ce(%)	Pr(%)	Nd(%)	Sm(%)	La(%)
ANK(VOH01)	19,45	1,52	4,17	0,41	15,25
B6 Roche	17,03	0,38	1,04	0,14	3,33
BETRAN 01	30,47	2,04	5,04	0,38	26,18
BETRAN 03	0,05	0	0,02	0,04	0,04
BG06	4,46	1,62	4,69	0,62	13,28
BP1P	26,82	2,04	5,23	0,47	20,79
BP1Pu	27,06	2,03	5,59	0,58	20,52
MKMRV01	29,4	2,38	6,9	0,7	20,92
MRV1d	1,53	0,13	0,36	0,1	1,07
VOH S2	31,97	2,39	6,63	0,6	23,95

Figure 16 : Light rare earths in the studied samples

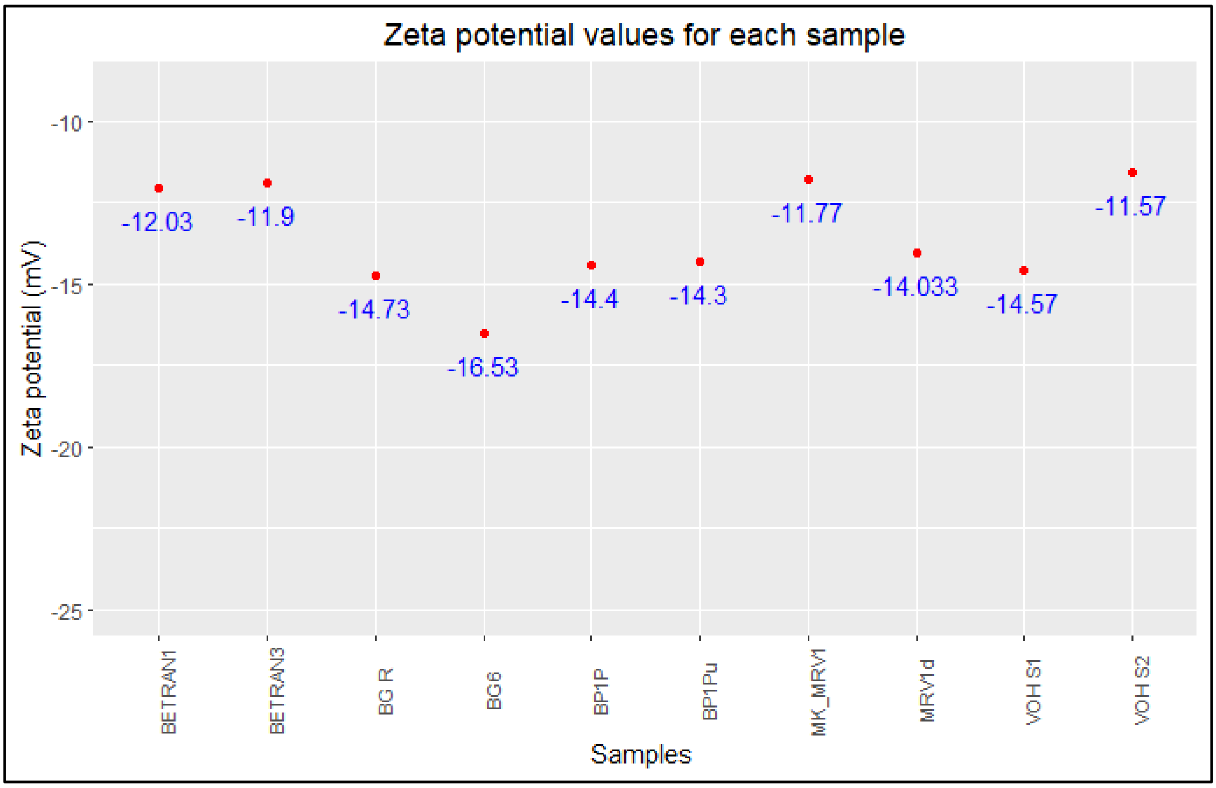


Figure 17: Results of sample zetametry measurements

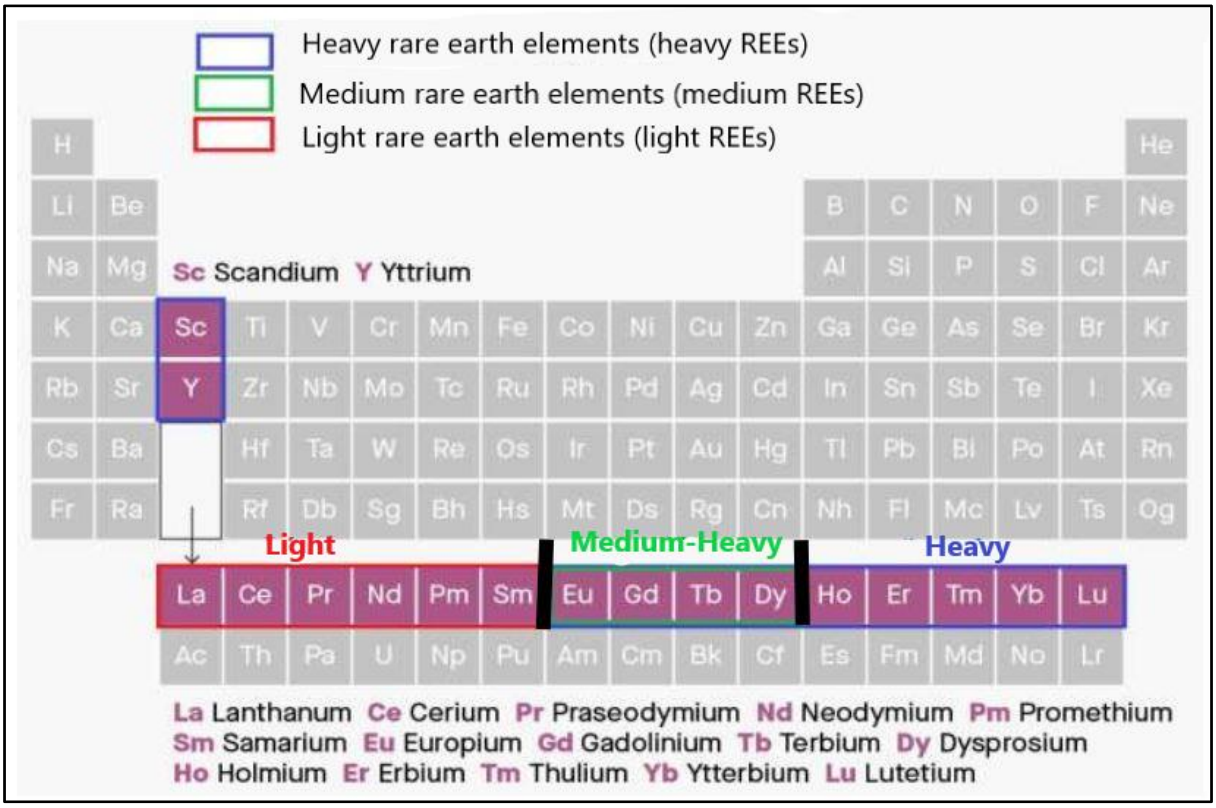


Figure 18: Rare earth elements in the periodic table of elements [11]

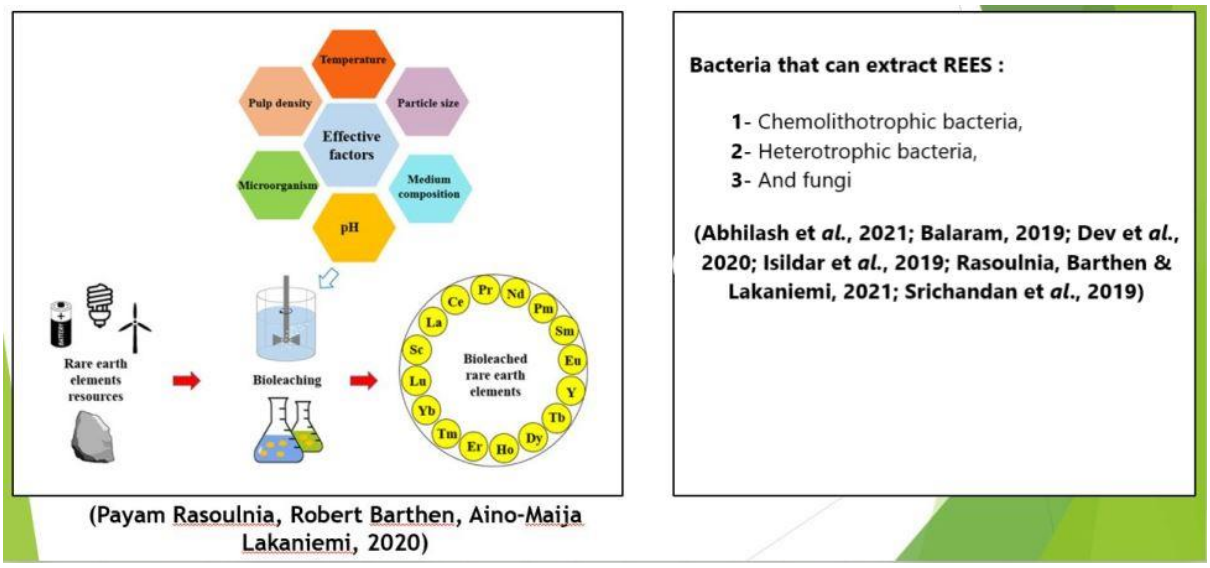


Figure 19: Brief biochemical descriptions of the bioleaching process of rare earths by bacteriological agents

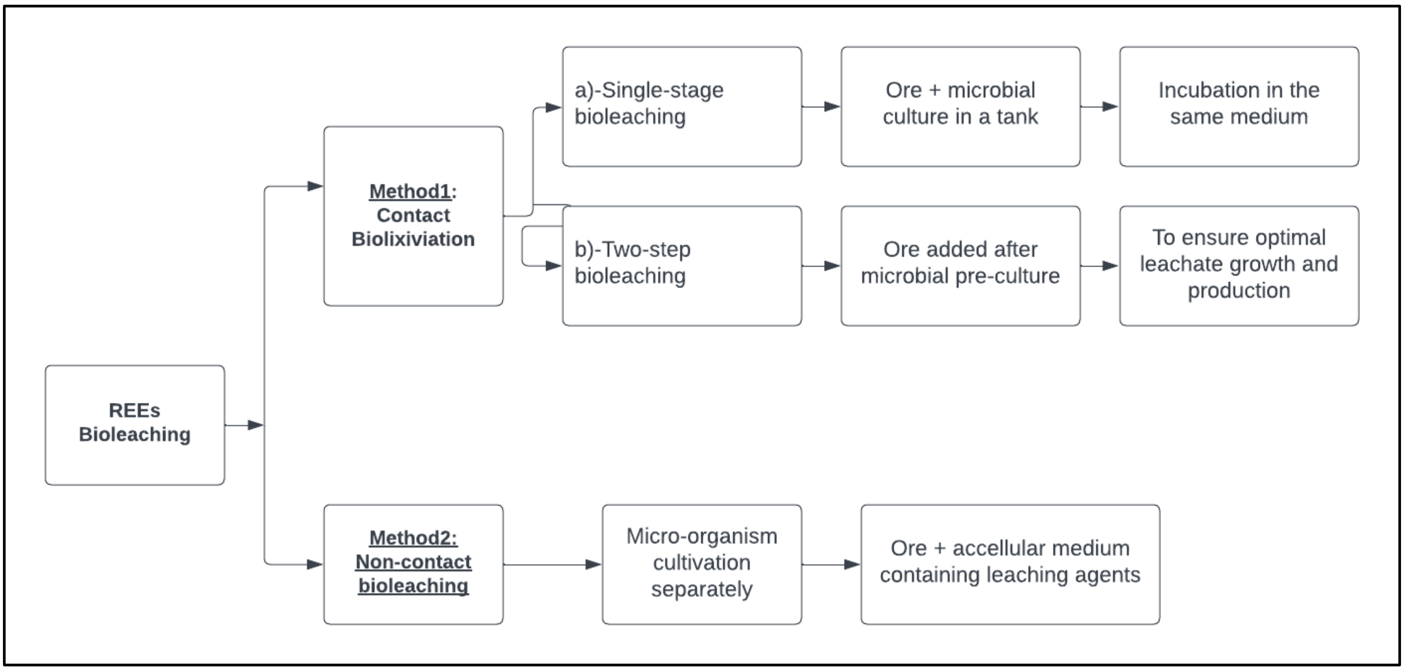


Figure 20: Flowsheet of different ways to bioleach rare earth ores [3]