XRF and Normative Analysis of Clay Samples

By: Ryan Manning

Introduction

In order to aid the finding and developing of critical minerals for many of the crucial devices and technologies that make daily life possible, the Department of Critical Minerals was established at Penn State. The goal of this department to be a leading group in the discovery, extraction and application of critical minerals in the country. This starting with many of the Mercer clay, and other deposits found right here in Pennsylvania.

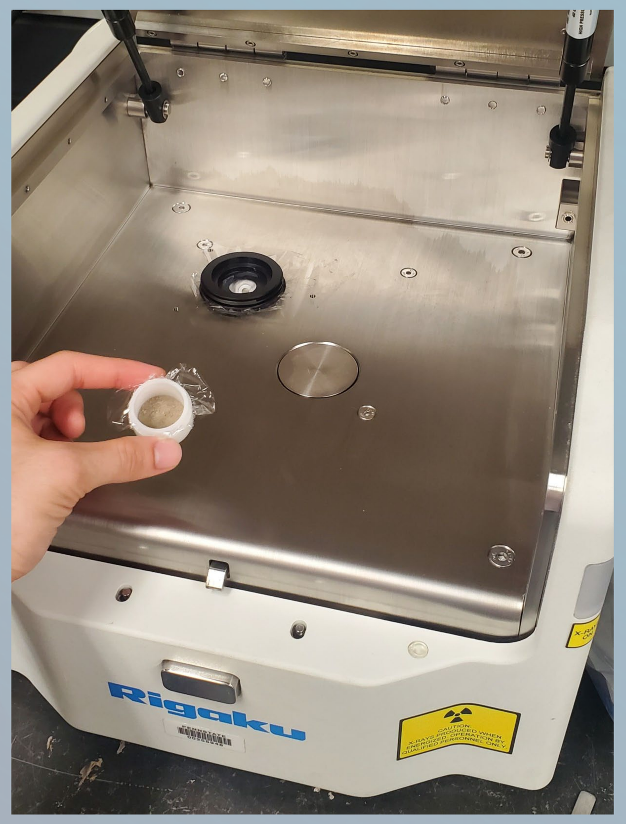
Supporting Plant Design With Lab Results

In the lab we received multiple different samples, these samples ranging from extracts from coal seams to mercer clay.

We have a few machines in the lab that we are using to replicate a factory workplace.

Our overall goals are trying to find methods to extract critical minerals from these clay samples, and ways to identify which samples from what area may contain desired minerals.

One important goal was finding ways to crush up the clay samples we have received. We are also using the data we gather to evaluate the grinding behavior of Mercer Clay Lithotypes



(XRF Machine)

Sample Breakdown and XRF Analysis Process

-The first step upon receiving a sample is to place the desired amount in a heat dryer.

-The next step is to crush up the media, the initial step would be to use the Chipmunk -Crushing machine to grind up the large samples into essentially large pebbles. This can then be placed in in a ball mill to grind that up farther into more small rocks and sand type material. Depending on the hardness of the sample we would have to use different size media in the ball mill.

-After the sample has been crushed in the ball mill, We then place it in the RoTap separator. This machine stirs the sample and screens it with different mesh sizes. One other method we may use in the future for separation would be the magnetic separator.

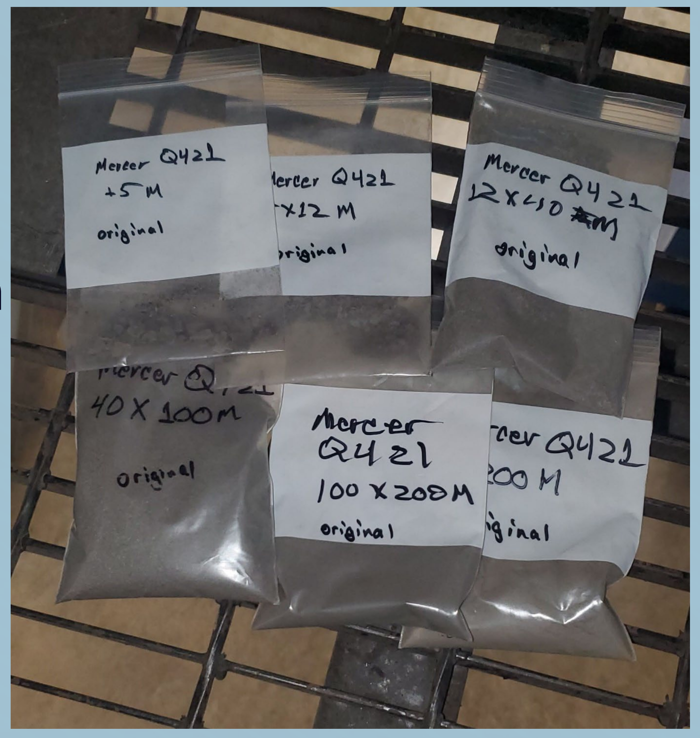
-Once we have a few different size fractions, we can then weigh each size to get an idea of how much constitutes each size.

-After this the different sizes can then be run through the or XRF machine.

-This machine uses X-Rays to knock out electrons from the orbitals of the atoms in the sample, depending on the distance the electrons need to travel to return, you can determine its composition

-Using the XRF you are able to have it select the specific elements and oxides you wish for it to display, and a few known compounds built into the system

-We can then do a normative analysis to estimate mineral composition Based on the data we receive from the XRF. This is shown in the following results



XRF and Analysis Results

The Following normative analysis technique was developed by Erickson for the Mercer Clay (Anatase, Goethite, Kaolinite, Muscovite, Quartz, and Diaspore). It has been modified for Chlorite and Illite instead of Muscovite.

You take advantage of the XRF ability to select certain oxides and

1. Allot XRF TiO_2 Result to Anatase
2. Allot XRF Fe_2O_3 to Goethite
3. Calculate Chlorite (Clinochlore) Content from MgO Analysis
4. Convert Al_2O_3 and SiO_2 Analyses to Al and Si and Allot Stoichiometric Portions to Clinochlore
5. Allot XRF TiO_2 Result to Anatase
6. Allot XRF Fe_2O_3 to Goethite (May be Modified to Include Siderite)
7. Calculate Chlorite (Clinochlore) Content from MgO Analysis
8. Convert Al_2O_3 and SiO_2 Analyses to Al and Si and Allot Stoichiometric Portions to Clinochlore

10. If R > 0.96 (Stoichiometric Kaolinite), Calculate Kaolinite Content from all Unallotted Si
11. Allot Al to Kaolinite and Use the Remaining Unallotted Al to Calculate Diaspore Content.

This Graph shows the Element and Oxide composition of 11 XRF runs of a sample. It was run numerous times in order to increase accuracy

Run	1	2	3	4	5	6	7	8	9	10	11
Al_2O_3	43.3%	41.1%	39.7%	39.4%	39.4%	39.2%	39.2%	39.2%	39.2%	39.2%	39.2%
SiO_2	56.7%	50.8%	48.1%	47.5%	47.5%	47.3%	47.3%	47.3%	47.2%	47.2%	47.2%
Fe_2O_3		8.1%	8.4%	8.4%	8.4%	8.5%	8.4%	8.4%	8.4%	8.4%	8.4%
TiO_2			3.8%	3.8%	3.8%	3.8%	3.8%	3.8%	3.8%	3.8%	3.8%
K_2O				0.9%	0.8%	0.8%	0.8%	0.8%	0.8%	0.8%	0.8%
MgO					0.0%	0.0%	0.0%	0.0%	0.0%	0.0%	0.0%
Na_2O					0.0%	0.0%	0.0%	0.0%	0.0%	0.0%	0.0%
CaO					0.0%	0.0%	0.0%	0.0%	0.0%	0.0%	0.0%
S						0.2%	0.2%	0.2%	0.2%	0.2%	0.2%
P						0.1%	0.1%	0.1%	0.1%	0.1%	0.1%
Cl						0.1%	0.1%	0.1%	0.1%	0.1%	0.1%
Zr							0.1%	0.1%	0.1%	0.1%	0.1%
Ba								0.0%	0.0%	0.0%	0.0%
Cu, Br, Sn, Sc, Cr									0.0%	0.1%	0.1%
Mn, Co, Ni, Ce, Y, Hf										0.0%	0.0%
Rb, W, Ta											0.0%
Total	100.0%	100.0%	99.9%	100.0%	100.0%	100.1%	100.0%	100.0%	99.9%	99.9%	99.9%

11 Runs Processing the Same Test, Each Run had Elements Added Beyond the Previous Run

Normative Analysis Results

Here we display the results of the previously shown Normative Analysis technique with the additional light elements beyond the Stoichiometric oxides calculated and included in the chart.

Data Set	1b	1b	1b	1b	2b	1b
Material	Mercer SF	Mercer SF	Mercer SF	Mercer SF	Mercer SF	Mercer SF
Type	Mill Product	Mill Product	Mill Product	Mill Product	Mill Product	Mill Product
Topsize	3/8"	5 Mesh	12 Mesh	40 Mesh	100 Mesh	200 Mesh
Bottom Size	5 Mesh	12 Mesh	40 Mesh	100 Mesh	200 Mesh	0
Measured						
Wt% TiO_2	2.42%		2.22%	2.48%	2.23%	
Wt% Fe_2O_3	5.06%		6.25%	5.18%	4.93%	
Wt% MgO	0.00%		0.68%	0.73%	0.74%	
Wt% K_2O	5.61%		4.72%	5.12%	4.71%	
Wt% Al_2O_3	31.40%		32.40%	33.10%	32.80%	
Wt% SiO_2	54.60%		52.90%	52.20%	53.50%	
LOI						
Calculated						
Wt% Anatase	2.18%		2.00%	2.23%	2.01%	
Wt% Goethite	5.07%		6.26%	5.19%	4.94%	
Wt% Kaolinite	51.50%		56.17%	56.29%	57.06%	
Wt% Illite	22.57%		18.99%	20.60%	18.95%	
Wt% Chlorite	0.00%		1.70%	1.81%	1.83%	
Wt% Quartz	13.37%		10.99%	9.43%	11.09%	
Wt% Diaspore	0.00%		0.00%	0.00%	0.00%	
Total	94.68%		96.10%	95.54%	95.87%	

Summary and Conclusion

In conclusion we found the process of Using a large breaking machine, such as a chipmunk after drying the sample, then using a ball mill and a RoTap was the most efficient process. We can then use the XRF to analyse samples and due to its ability to quickly process many different samples it is able to efficiently give us results accurate enough to determine if the sample being tested can be used farther, ie: The site where it was discovered is rich in critical minerals, and more exploration can be done.

References

XRF Function and Use:
https://www.bruker.com/en/products-and-solutions/elemental-analyzers/xrf-spectrometers/how-does-xrf-work.html

The Team

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