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# Comparation of Pressed Powder Pellet and Fused Glass Bead Preparation Techniques for Mayor Elements Analysis of Rock Samples using X-Ray Fluorescence (XRF)

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## Abstract

The composition of chemical elements in rock samples can be used as a tool for classifying rock types, predicting the depositional environment, rock age, and the tectonic environment in which the rock is formed. X-Ray Fluorescence (XRF) is one of the instruments used to analyze rock samples. The advantage of using XRF analysis is that the analysis process can be done more quickly, easily, accurately, and does not damage the sample. However, before measurements are made using the XRF instrument it needs to be supported by an appropriate sample preparation process. In this study, optimal conditions in the standard (CRM) rock sample preparation of GBW 07105 and JR-1 have been studied. The parameters studied were variations in the composition ratio of Cellulose Mycro Crystalin (CMC) with standard (CRM) samples, namely 1: 4 and 1: 3. The technique of mixing the sample is done by using a mortar grinder and a shaker. Optimization of sample preparation was carried out using pressed powder pellet and fused glass bead techniques. Based on the data obtained from the analysis results the best ratio of binders to standard samples is 1: 3. The pressed powder pellet preparation technique is the best technique in analyzing samples using the X-Ray Fluorescence method. Further studies also need to be carried out to analyze minor elements and traces (REEs).

*Keywords*: mayor element, pellet, glass bead, XRF, rock sample, CMC

## INTRODUCTION

The chemical composition of rock from X-Ray Fluorescence (XRF) analysis is needed to increase geological confidence in determining rock unit boundaries in geological mapping [1]. While in Volcanology, XRF analysis is used to determine the type of rock, depositional environment, and geochronology of several geological phenomena that have occurred several years ago [2]. XRF analysis is also needed in other fields of science like mining exploration, mineral separation, etc [3,4,5,6,7].

Therefore, valid, accurate and precise XRF analysis results are very important and vital. Furthermore, in the world of mining exploration, the XRF data have a big role in the strategic policy decision for the assessment of mining working areas.

In this study, the optimal preparation technique of rock sample for XRF analysis using Certified Reference Material (CRM) was studied. The preparation techniques used in this study are Pressed Pellet Technique and Fused Glass Bead. CRM was used as a benchmark for the accuracy of the analysis. In this experiment, an analysis was performed to determine the chemical composition of rock samples. The data from the XRF analysis was compared with the CRM certificate value for determining the optimal technique in the preparation of rock samples.

## EXPERIMENTAL METHOD

### Materials

There are 3 rock samples used for this research, CRM GBW 07105, CRM JR-1, and indent

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samples. Additional materials used are Boric acid, Cellulose Mycro Crystalin (CMC) as a binder, Lithium Tetraborat ($LiB_4O_7$), Lithium Metaborat ($LiBO_2$), Lithium Bromide (LiBr) 10%, and Lithium Nitrate (LiNO3) 10%. The chemical composition of the sample was determined using sequential X-ray Fluoresence (XRF) ADVANT XP Thermo ARL. The data from the XRF analysis was compared with the CRM certificate value [13,15].

**Lost On Ignation (LOI) Analysis**

A gravimetric method was used for LOI analysis. The empty porcelain cup was put into the electric furnace with a temperature of 300 °C for 1 hour. After cooled in a desiccator, 1 grams of rock sample was added to the porcelain cup. Next, the porcelain cup containing the sample is put back into the electric furnace to for 2 hours at a temperature of 1000 °C. After that, the porcelain cup containing the sample was cooled in a desiccator and then weighed again with an analytical balance.

**Pressed Powder Pellet Preparation**

A 200 mesh rock sample was weighed as much as 5 grams using a weighing glass in an analytical balance then 1 gram of Cellulose Mycro Crystalin (CMC) was added [8,9,10,11,12]. After that, the mixture was homogenized in two different methods. The first method used mortar to make the mixture of rock samples and CMC become homogenous and smooth. The second method used shaker for 15 minutes to mix the rock sample and CMC. The homogeneous mixture is then pressed using a Herzog press machine. The pellet ring is placed on the instrument and then covered with 2 grams of boric acid and the sample is homogeneously added. Then the crossbar is closed and the pellet ring is removed from the sample and placed in a container. The equipment is cleaned with a vacuum cleaner and 90% alcohol. After that, the ring pellet containing the sample is labeled and dried in an oven at 52 oC for 15 minutes. In this study, the ratio of rock sample and CMC was also varied between 1: 4 and 1: 3.

**Fused Glass Bead Preparation**

The standard rock sample was weighed as much as 0.300 grams using analytics balance then 7,300 grams of lithium tetraborate was added and 2,700 grams of lithium metaborate were added. The rock sample mixture was homogenized with a spatula. After that, add 10% LiBr and 10% LiNO3 each 1 drop then the mixture is put into the oven for 2 minutes. Then, the mixture is fused with the glass disk making machine. First, the platinum-Au cup containing the mixture is placed on the glass disk making device then the flame is set. Next, the Reset and Start buttons are pressed, the flame is ignited with a lighter, the appliance door is closed then the blower is turned on and it waits until the tool stops. Then the tool door is opened and the glass platinum-Au cup is taken and the two are separated. Platinum platter cleaned with ultrasonic cleaner and HCL. After that, glass is labeled to facilitate the analysis [13,14, 15,16].

**RESULTS AND DISCUSSION**

Before an XRF analysis is performed, the Loss On Ignitions (LOI) of rock sample must be analyzed first. The result of LOI analysis from a rock sample used in this study is in table 1.

Table 1. Result of Loss On Ignitions (LOI) Analysis

| Sample ID | LOI (%) |
|---|---|
| GBW 07105 | 2.93 |
| Indent Sample | 0.74 |
| JR-1 Press Pellet | 1.83 |

LOI is needed for the calculation of chemical compound concentrations in XRF analysis. LOI reflects the water content, organic content and carbonate content contained in the rock sample. Organic matter lost and water content of GBW 07105 is 0.0293 grams, indent sample is 0.0074 grams, and JR-1 is 0.0183 grams. The difference in LOI values is due to different rock types, the influence of the duration of geological processes and activities. If the LOI value obtained is large enough, the levels of organic substances contained in the sample are high enough.

The preparation technique in this experiment was carried out by two methods, the pressed powder pellet and the fused glass bead. For pressed powder pellet, preparation is done by mixing CMC and indent sample or GBW 07105. CMC functions as a binder to bind the indent sample or GBW 07105 when pressing is carried out so that the indent or GBW 07105 sample becomes a pellet that has a smooth surface and not cracked. In addition, when making pellets boric acid is needed as a base layer. The boric acid is needed to hold firmly the sample in the pellet ring and during the X-ray testing process. Boric acid is often used for the pellet ring base layer because the price is relatively cheap and easy to obtain. The resulting pellets are inserted into the cassette contained in the XRF instrument. Cassette serves as a container for placing samples.

The fused glass bead or glass disk preparation method is done by mixing Lithium Tetraborate and Lithium Metaborat with JR-1. Both chemicals are needed as binders and to reduce the melting point of the sample when the fused glass bead process is

carried out. In addition, a 10% LiBr is needed to prevent cracks in the glass disks and 10% LiNO3 is added to reduce the viscosity of the hot liquid sample so that no mixture is left in the platinum - Au cup. The resulting glass disk was analyzed using the XRF instrument.

This experiment used a ratio of binders and indent samples with a ratio of 1: 5 and the ratio of binders to GBW 07105 with variations of ratios of 1: 4, 1: 3. The ratio is used because the ratio of 1: 5 is an optional ratio. When using a ratio of 1: 6 the resulting pellet cracks during pressing. This is because the composition of the binder volume is much less than the indent sample or GBW 07105, so the binder power is not strong enough to bind the indent sample or GBW 07105. Whereas when using a 1: 2 ratio, the number of binder volumes and indent samples or GBW 07105 is not enough to make pellet.

Tabel 2. Result of GBW 07105 Analysis

| Compound | GBW 07105 (m/m%) | | |
|---|---|---|---|
| | CRM | Ratio 1:3 | Ratio 1:4 |
| $SiO_2$ | 44.64 | 43.06 | 43.49 |
| $Al_2O_3$ | 13.83 | 18.24 | 18.45 |
| MgO | 7.77 | 6.65 | 6.49 |
| CaO | 8.81 | 7.76 | 7.71 |
| $Na_2O$ | 3.83 | 3.96 | 3.87 |
| $K_2O$ | 2.32 | 2.29 | 2.26 |

The results of the XRF analysis with ratio of CMC and rock sample varied 1:4 and 1:3 are shown in Table 2. A SiO2 and Al2O3 are the highest components of the sample. This is because these components are the main part of igneous rock formation [3]. The results of the analysis of each ratio were compared with the value of the GBW 07105 CRM certificate. The analysis result of the GBW 07105 sample that has sample ratio 1: 4 that approached the value of the certificate were SiO2 and Na2O components. Whereas the result analysis of components Al2O3, MgO, CaO and K2O which are close to the value of the certificate is the sample ratio of 1: 3. Based on the data from the analysis, the ratio of binder to GBW 07105 that approached the value of the GBW 07105 certificate is ratio 1:3.

The results of the XRF analysis with varied in homogenization technique during the sample preparation are shown in Table 3. A mortar or shaker technique is used to homogenize the mixture of CMC and rock samples. A CaO, $SiO_2$ and $Al_2O_3$ are the highest components of the sample. This is because these components are the main part of igneous rock formation [3]. The results of the analysis of each homogenization technique were compared with the value of the indent sample CRM certificate. The analysis result of the Indent Sample that use mortar homogenization technique which approached the value of the certificate were CaO, Al2O3, MgO, SO3, CuO, Fe2O3, TiO2, SrO,V2O5, Cr2O3 and Rb2O components. The analysis result of the Indent Sample which use shaker as homogenization technique that approached the value of the certificate were SiO2, K2O, MnO, BaO and ZnO components. Whereas the result analysis of components Na2O with homogenization technique using mortar and shaker has same result. Based on the data from the analysis, the optimum homogenization technique of sample and CMC mixture is use mortar.

Tabel 3. Result of Indent sample Analysis using Mortar dan Shaker homogenization technique

| Compound | m/m% | | |
|---|---|---|---|
| | CRM | Mortar | *Shaker* |
| CaO | 38.14 | 45.47 | 45.94 |
| $SiO_2$ | 29.9 | 45.47 | 33.97 |
| $Al_2O_3$ | 10.05 | 9 | 8.83 |
| MgO | 9.95 | 2.09 | 2.06 |
| $SO_3$ | 2.65 | 0.419 | 0.417 |
| CuO | 2.46 | 0.0233 | 0.0222 |
| $K_2O$ | 2.36 | 1.75 | 1.77 |
| $Fe_2O_3$ | 2.32 | 4.07 | 4.15 |
| MnO | 0. 616 | 0.0941 | 0.0975 |
| $Na_2O$ | 0.477 | 1.09 | 1.09 |
| $TiO_2$ | 0.396 | 0.402 | 0.43 |
| BaO | 0.141 | 0.0688 | 0.0766 |
| SrO | 0.0699 | 0.173 | 0.176 |
| ZnO | 0.0423 | 0.0341 | 0.0358 |
| $V_2O_5$ | 0.0256 | 0.0055 | 0.0018 |
| $Cr_2O_3$ | 0.0127 | 0.0145 | 0.0168 |
| $Rb_2O$ | 0.0119 | 0.0083 | 0.0077 |

The results of the XRF analysis with pressed powder pellet and fused glass bead technique of CRM JR-1 sample are shown in Table 4. A SiO2 and Al2O3 are the highest components of the sample. This is because these components are the main part of igneous rock formation [3]. The results of the analysis of each technique are compared with the value of the JR-1 CRM certificate. The analysis result of JR-1 that use pressed powder pellet technique which approached the value of the certificate were K2O, Na2O, Fe2O3, CaO, MgO, MnO and TiO2 components. Whereas the result analysis of components SiO2 and Al2O3 which are close to the value of the certificate is use fused glass bead technique. Based on the data from the analysis, the optimum preparation technique for JR-1 sample

which approached the value of the certificate is pressed powder pellet technique.

Tabel 4. . Result of JR-1 analysis using Pressed Powder Pellet dan Fused Glass Bead Preparation Techniques

| Compound | JR-1 (m/m%) | | |
|---|---|---|---|
| | **CRM** | **Pelet** | ***Fused*** |
| $SiO_2$ | 75.45 | 73.12 | 73.93 |
| $Al_2O_3$ | 12.83 | 14.65 | 14.44 |
| $K_2O$ | 4.41 | 4.19 | 3.91 |
| $Na_2O$ | 4.02 | 4.09 | 3.8 |
| $Fe_2O_3$ | 0.35 | 0.82 | 0.867 |
| CaO | 0.67 | 0.705 | 0.633 |
| MgO | 0.12 | 0.148 | 0.201 |
| MnO | 0.099 | 0.0974 | 0.0949 |
| $TiO_2$ | 0.11 | 0.0947 | 0.0764 |

## CONCLUSION

The pressed powder pellet is a simple but quite accurate technique for analysis process using XRF instrument. This technique provides optimal results for chemical compound determining in a standard rock sample. The XRF analysis result approach the value of CRM certificate. The ratio of CMC:GBW 07105 which close to the value of a CRM certificate is ratio 1: 3. Whereas the optimum homogenization technique of sample and CMC mixture is use mortar. However, further study are needed to optimize the accuracy and precision of analysis results in accordance with statistical approach.

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