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Effect Of Grinding Time on Particle Size and XRF Analysis Results of Nickel Ore Samples Prepared by Press Pellet Using Epsilon 4

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Abstract
Sample preparation is an important stage in nickel ore analysis using X-Ray Fluorescence (XRF) because particle size and sample homogeneity can affect the measured composition. This study evaluated the effect of grinding time and particle size on nickel ore samples prepared by the press pellet method and analyzed using an XRF Epsilon 4. Nickel ore samples were ground using a pulverizer for 1, 3, 5, 7, and 9 min, followed by sieving at 100 and 200 mesh. Each sample condition was analyzed twice. The measured parameters were Ni, Fe, MgO, and SiO_2. Increasing grinding time generally produced finer and more homogeneous particles and changed the measured elemental composition. At 200 mesh, Ni increased from 1.32% at 1 min to 1.48% at 7 min, MgO increased from 8.82% to 9.23%, and SiO_2 increased from 36.73% to 37.40%, while Fe decreased from 24.60% to 24.16%. At 9 min, the values changed slightly from the 7-min condition, indicating that longer grinding did not continuously improve the measured results. The optimum condition reported in this study was 7 min grinding with 200 mesh, producing Ni 1.48%, Fe 24.16%, MgO 9.23%, and SiO_2 37.40%. The results indicate that controlling grinding time and particle size is important for obtaining a more homogeneous and representative pressed-pellet sample for XRF analysis.
Keywords: *nickel ore; grinding time; particle size; press pellet; xrf epsilon 4*

Abstrak
Preparasi sampel merupakan tahapan penting dalam analisis bijih nikel menggunakan X-Ray Fluorescence (XRF) karena ukuran partikel dan homogenitas sampel dapat memengaruhi komposisi yang terukur. Penelitian ini mengevaluasi pengaruh waktu grinding dan ukuran partikel terhadap sampel bijih nikel yang dipreparasi dengan metode press pellet dan dianalisis menggunakan XRF Epsilon 4. Sampel bijih nikel digerus menggunakan pulverizer selama 1, 3, 5, 7, dan 9 menit, kemudian diayak pada ukuran 100 dan 200 mesh. Setiap kondisi sampel dianalisis sebanyak dua kali. Parameter yang dianalisis meliputi Ni, Fe, MgO, dan SiO_2. Peningkatan waktu grinding secara umum menghasilkan partikel yang lebih halus dan homogen serta mengubah komposisi unsur yang terukur. Pada ukuran 200 mesh, kadar Ni meningkat dari 1,32% pada 1 menit menjadi 1,48% pada 7 menit, MgO meningkat dari 8,82% menjadi 9,23%, dan SiO_2 meningkat dari 36,73% menjadi 37,40%, sedangkan Fe menurun dari 24,60% menjadi 24,16%. Pada 9 menit terjadi perubahan kecil dibandingkan kondisi 7 menit sehingga menunjukkan bahwa waktu grinding yang lebih lama tidak selalu meningkatkan hasil pengukuran. Kondisi optimum pada penelitian ini adalah grinding 7 menit dengan ukuran partikel 200 mesh, yang menghasilkan Ni 1,48%, Fe 24,16%, MgO 9,23%, dan SiO_2 37,40%. Hasil penelitian menunjukkan bahwa pengendalian waktu grinding dan ukuran partikel penting untuk memperoleh sampel press pellet yang lebih homogen dan representatif untuk analisis XRF.
Kata kunci: *bijih nikel; waktu grinding; ukuran partikel; press pellet; xrf epsilon 4*

1. Introduction

Nickel laterite is an important mineral resource for the production of stainless steel, nickel alloys, and battery-related materials. The increasing demand for nickel requires analytical procedures that can provide accurate and representative information on ore composition. *X-Ray Fluorescence* (XRF) is widely used for multi-element analysis because it can determine several elements rapidly and with relatively simple chemical preparation. However, the quality of XRF results is strongly influenced by sample preparation, particularly particle size, particle-size distribution, and sample homogeneity.

Previous studies have shown that sample preparation is a critical factor in XRF analysis because particle size, particle-size distribution, and sample homogeneity can influence the intensity of characteristic X-rays and the representativeness of the measured composition. In *pressed-pellet* preparation, finer particles generally provide a more uniform surface and reduce heterogeneity within the pellet. However,

excessive grinding may cause particle agglomeration and other physical changes that can affect measurement consistency. Therefore, grinding time should be optimized together with the target particle size. Evaluating these factors is important for establishing a reproducible preparation procedure for nickel ore samples analyzed by *ED-XRF*.

In addition, the grinding process can affect the distribution of mineral phases within the sample and the physical characteristics of the powder used for pellet formation. Differences in particle size may influence packing density, surface smoothness, and the interaction between the sample matrix and incident X-rays during measurement. These effects may subsequently contribute to variations in the measured concentrations of Ni, Fe, MgO, and SiO_2. A systematic evaluation of grinding time and particle size is therefore necessary to determine whether longer grinding consistently improves analytical results or whether an optimum condition exists. This study investigates grinding times of 1, 3, 5, 7, and 9 minutes combined with particle sizes of 100 and 200 mesh to evaluate their effects on the XRF analysis results of nickel ore prepared using the *press pellet* method.

2. Materials and Methods

2.1 Research period and material

The research was conducted for approximately one month during April–May 2026 in a laboratory. The material used was nickel ore, with binder used during press-pellet preparation. The sample mass was maintained at the same level for each test condition to ensure consistency during sample preparation and analysis.

2.2 Equipment

The main equipment consisted of a pulverizer, 100- and 200-mesh sieves, pellet press, XRF Epsilon 4, analytical balance, and sample containers. The reported pulverizer capacity was 100–1000 g per process, with a maximum feed size of 10–20 mm and a grinding product size down to 75 µm (200 mesh).

2.3 Experimental variables and procedure

The independent variables were grinding time of 1, 3, 5, 7, and 9 min and particle size of 100 and 200 mesh. Sample mass was kept constant. The sample was prepared, cleaned and dried when required, weighed, ground using the pulverizer, and sieved. The resulting powder was formed into a press pellet and analyzed using XRF Epsilon 4. Each condition was measured twice, and the mean was used for the graphical analysis and discussion.

2.4 Data analysis

The results were compared descriptively across grinding time and particle size. The mean value for each condition was calculated from the two replicate measurements. The response parameters were Ni, Fe, MgO, and SiO_2 contents.

3. Results and Discussion

3.1 Effect of grinding time and particle size on Ni

Ni content generally increased with longer grinding time and with the finer 200-mesh fraction. At 1 min, Ni was 1.28% for 100 mesh and 1.32% for 200 mesh. At 3 and 5 min, the values increased to 1.34–1.40% for 100 mesh and 1.38–1.44% for 200 mesh. The highest Ni content was 1.48% at 7 min and 200 mesh. At 9 min, Ni decreased slightly to 1.40% for 100 mesh and 1.45% for 200 mesh. The increase up to 7 min can be attributed to finer particles and improved mineral distribution within the pellet, whereas the slight decrease at 9 min may be related to agglomeration of very fine particles.

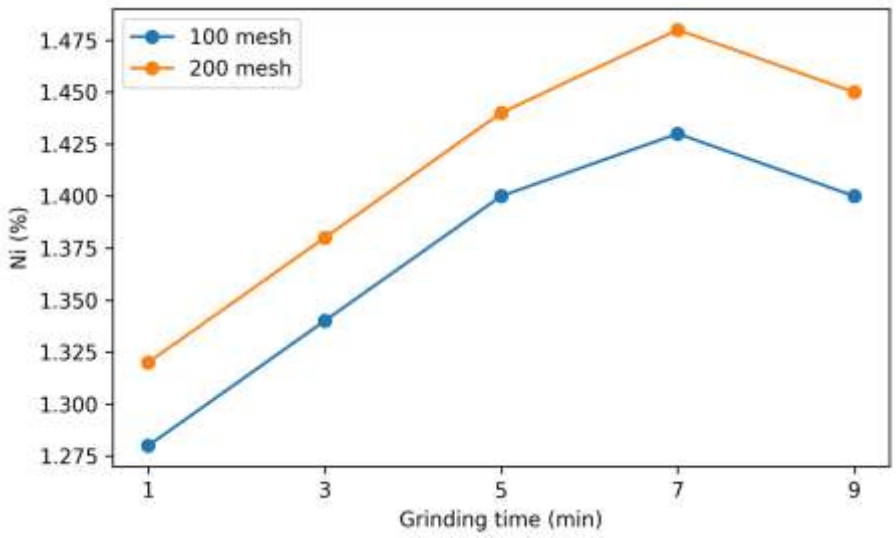


Figure 1. Ni content as a function of grinding time and particle size.

3.2 Effect of grinding time and particle size on Fe

Fe showed the opposite trend to Ni. At 1 min, Fe decreased from 24.82% at 100 mesh to 24.60% at 200 mesh. The same direction was observed at 3 and 5 min. The lowest Fe content was 24.16% at 7 min and 200 mesh, while at 9 min it increased slightly to 24.47% and 24.22% for 100 and 200 mesh, respectively. This trend may be related to the greater uniformity of finer particles, which can reduce sample heterogeneity and matrix-related variation during XRF measurement. The slight increase at 9 min may be associated with agglomeration caused by excessive grinding.

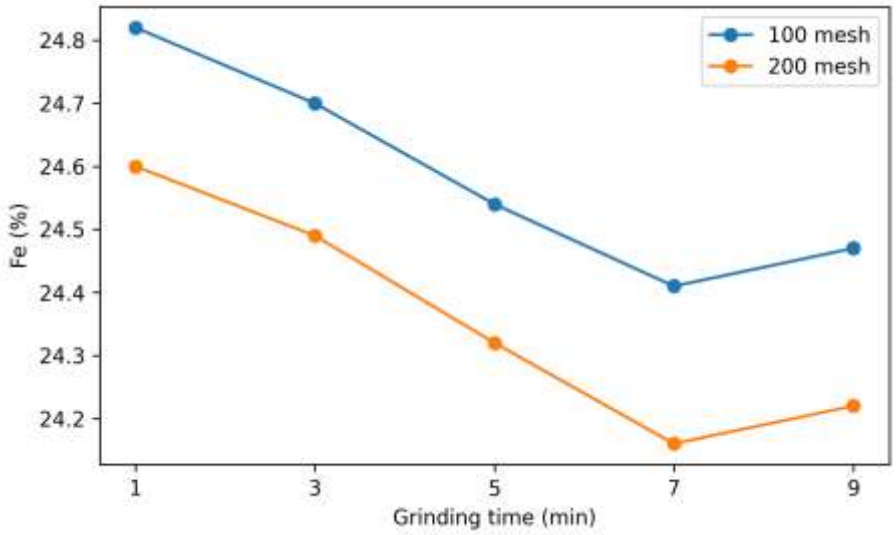


Figure 2. Fe content as a function of grinding time and particle size.

3.3 Effect of grinding time and particle size on MgO

MgO increased with grinding time up to 7 min and was consistently higher at 200 mesh than at 100 mesh. At 1 min, MgO was 8.71% and 8.82% for 100 and 200 mesh, respectively. The maximum value was 9.23% at 7 min and 200 mesh. At 9 min, MgO decreased slightly to 9.07% at 100 mesh and 9.16% at 200 mesh. This behavior indicates that finer particles improved sample homogeneity and elemental distribution, whereas excessive grinding may cause agglomeration and fluctuations in the measured value.

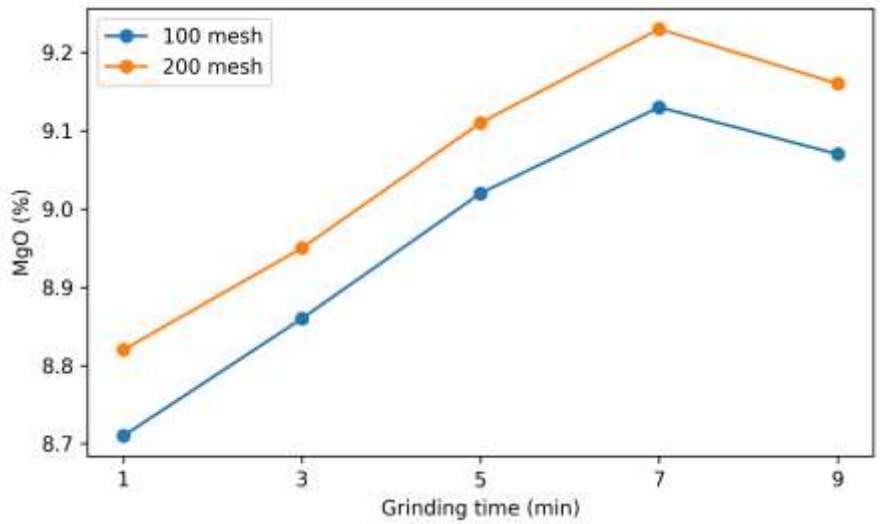


Figure 3. MgO content as a function of grinding time and particle size.

3.4 Effect of grinding time and particle size on SiO_2

SiO_2 followed a trend similar to MgO. At 1 min, the values were 36.41% at 100 mesh and 36.73% at 200 mesh. The values increased to 36.96% and 37.22% at 5 min, and the highest values were obtained at 7 min: 37.13% at 100 mesh and 37.40% at 200 mesh. At 9 min, SiO_2 decreased slightly to 37.03% and 37.28%. The increase up to 7 min may be related to finer and more homogeneous particles, while the decrease at 9 min may be associated with agglomeration of excessively fine particles.

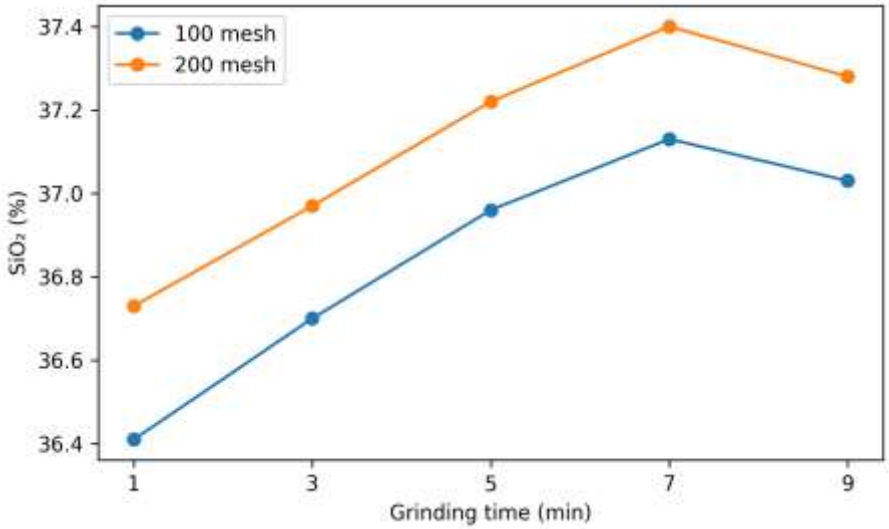


Figure 4. SiO_2 content as a function of grinding time and particle size.

Table 1. Mean XRF analysis results at each grinding time and particle size

Grinding time (min)	Ni 100	Ni 200	Fe 100	Fe 200	MgO 100	MgO 200	SiO_2 100	SiO_2 200
1	1.28	1.32	24.82	24.60	8.71	8.82	36.41	36.73
3	1.34	1.38	24.70	24.49	8.86	8.95	36.70	36.97
5	1.40	1.44	24.54	24.32	9.02	9.11	36.96	37.22
7	1.43	1.48	24.41	24.16	9.13	9.23	37.13	37.40
9	1.40	1.45	24.47	24.22	9.07	9.16	37.03	37.28

3.5 Optimum condition

Across the four measured parameters, the results indicate that 7 min of grinding with 200 mesh provided the optimum condition, as it resulted in the highest Ni, MgO, and SiO_2 contents and the lowest Fe content. The corresponding values were Ni 1.48%, Fe 24.16%, MgO 9.23%, and SiO_2 37.40%. At 9 min,

all four parameters showed slight changes compared with the 7-min condition, indicating that extending the grinding time beyond 7 min did not further improve the measured XRF results.

4. Conclusion

Grinding time and particle size affected the measured XRF composition of the nickel ore prepared by the press pellet method. Increasing grinding time generally produced finer and more homogeneous particles and increased the measured Ni, MgO, and SiO_2 contents while Fe tended to decrease. The 200-mesh fraction consistently produced higher Ni, MgO, and SiO_2 and lower Fe than the 100-mesh fraction. The optimum condition reported in this study was 7 min grinding with 200 mesh, yielding Ni 1.48%, Fe 24.16%, MgO 9.23%, and SiO_2 37.40%.

5. Recommendations

Future studies should investigate narrower grinding-time intervals around the 7-minute condition and include additional particle-size ranges to further determine the optimal sample preparation conditions. The accuracy and precision of the XRF analysis should also be evaluated quantitatively using recovery, relative standard deviation (RSD), bias, and certified reference materials (CRM).

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