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The Effect of Using Porcelain Mortar and Pestle in Sample Preparation for Scanning Electron Microscope-Energy Dispersive X-Ray (SEM-EDX) Analysis

Pengaruh Penggunaan Mortar dan Alu Berbahan Porselen pada Preparasi Sampel Pengujian Scanning Electron Microscope Energy Dispersive X-Ray (SEM-EDX)

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ABSTRACT

Porcelain mortars and pestles are among the most commonly used tools for simple sample preparation. However, repeated use can produce numerous scratches on their surfaces. These scratches may lead to the abrasion of the porcelain material, which primarily contains SiO_2, and are therefore suspected to be a potential source of silicon (Si) contamination during SEM–EDX analysis. In this study, a comparison was conducted among samples that were not ground, samples ground using a new porcelain mortar and pestle, and samples ground using an old porcelain mortar and pestle with extensive surface scratches. This comparison was performed to evaluate the possibility of contamination originating from the porcelain mortar and pestle. The results showed that all samples exhibited Si concentrations below the instrument detection limit (< 0.1 wt.%), as reported in the literature for routine SEM/SDD-EDS analysis. These findings indicate that the use of porcelain mortar and pestle for powder sample preparation under ambient temperature conditions does not significantly contribute to silicon contamination during SEM–EDS sample preparation. Therefore, porcelain mortar and pestle can be considered suitable sample preparation tools for SEM–EDS analysis under the investigated conditions.

Keywords*: Mortar, Porcelain, SEM-EDX, Silicon*

ABSTRAK

Mortar dan alu berbahan porselen merupakan salah satu alat preparasi sederhana. Penggunaan mortara dan alu porselen dapat menimbulkan banyak goresan pada permukaannya. Goresan pada mortar dan alu porselen menyebabkan terkikisnya bahan-bahan alu porselen yaitu SiO_2 yang diduga menjadi sumber kontaminan Si pada pengujian menggunkan SEM-EDX. Melalui penelitian ini, dilakukan perbandingan antara sampel yang tidak digerus, digerus dengan mortar baru, dan digerus dengan mortar lama (telah terdapat banyak goresan). Perbandingan ini dilakukan untuk mengetahui kemungkinan terjadinya kontaminasi yang bersumber dari mortar dan alu porselen. Seluruh sampel yang digunakan pada penelitian ini, memiliki hasil kandungan unsur Si di bawah nilai batas deteksi alat (< 0,1 wt %), seperti yang dilaporkan dalam literatur untuk analisis rutin SEM/SDD-EDS. Hal ini menunjukkan bahwa mortar dan alu porselen yang digunakan pada sampel serbuk dengan kondisi suhu sesuai suhu ruangan, tidak memberikan pengaruh yang signifikan terhadap jumlah kontaminasi silika (Si) dalam preparasi sampel uji SEM-EDX. Sehingga mortar dan alu porselen sesuai untuk digunakan sebagai alat preparasi uji SEM-EDX pada kondisi tersebut.

Kata Kunci : Mortar, Porselen, SEM-EDX, Silika

INTRODUCTION

A mortar and pestle are used to pulverize or grind solid substances (Mu'nisa et al., 2023). Additionally, they can be utilized to blend multiple solid materials, dissolve solids within a solvent, and perform extraction processes (Widianto E, 2022). Mortars and pestles can be fabricated from various materials, including porcelain, glass (borosilicate), metal, agate, and Teflon (Samodra, 2025). The most frequently used type for SEM sample preparation is the porcelain mortar and pestle. Generally, porcelain is composed of a mixture of raw materials consisting of feldspar [(K, Na) $AlSi_3O_8$], kaolinite clay ($Al_2O_3.2SiO_2$-$2H_2O$), and quartz (SiO_2) (Indiani et al., 2009).

Porcelain mortars and pestles are commonly used in laboratory sample preparation due to their mechanical strength and chemical stability. However, porcelain materials typically consist of silica-rich phases such as quartz, feldspar, and kaolinite, which may be susceptible to surface wear during repeated grinding operations (Ali, A. et al., 2023). Studies have demonstrated that even commonly used grinding tools, such as agate or ceramic-based mortars, may introduce trace elemental contamination during repeated use (Soomro, M.H. et al., 2024). This effect becomes more critical when the grinding equipment contains silica-bearing phases, as silicon can be unintentionally introduced into the sample during abrasion processes.

Scanning Electron Microscopy (SEM) is a type of electron microscopy that provides high-resolution images of sample surfaces (Septiano et al., 2021). Energy-Dispersive X-ray Spectroscopy (EDS, also known as EDX or EDAX) is an analytical technique used to determine the elemental composition and chemical characteristics of a specimen. The technique is based on the interaction between X-rays and the atomic structure of the sample, producing characteristic signals that are unique to each element (Sudrajat et al., 2014). The combination of Scanning Electron Microscopy (SEM) and Energy-Dispersive X-ray Spectroscopy (EDX) offers a powerful approach for microscopic observation and elemental composition analysis of materials (Mardika et al., 2024).

The accuracy of SEM–EDX elemental analysis can be influenced by contamination introduced during sample preparation. Since porcelain mortars and pestles contain silica-rich minerals such as quartz, feldspar, and kaolinite, there is a possibility that abrasion occurring during grinding may release silicon-containing particles that are subsequently transferred to the sample. This potential contamination may become more significant when the

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mortar surface has experienced wear or scratching due to prolonged use. Consequently, the use of porcelain grinding equipment raises concerns regarding its suitability for SEM–EDX sample preparation, particularly when silicon determination is of interest.

Although contamination originating from grinding equipment has been discussed in previous studies, investigations specifically addressing the potential transfer of silicon from porcelain mortars and pestles during SEM–EDX sample preparation remain limited. Furthermore, the influence of mortar condition, particularly the difference between new and used mortars with surface scratches, has not been thoroughly evaluated. As a result, uncertainty remains regarding whether the use of porcelain mortars contributes measurable silicon contamination that could affect SEM–EDX analytical results. Therefore, a systematic evaluation of silicon contamination associated with porcelain mortar use is necessary to ensure the reliability of elemental characterization.

In this study, the potential effect of porcelain mortar and pestle use on silicon contamination during SEM–EDX sample preparation was investigated. Samples prepared without grinding were compared with samples ground using a new porcelain mortar and pestle and samples ground using a used mortar and pestle exhibiting visible surface scratches. This approach was intended to evaluate whether mortar condition influences the occurrence of detectable silicon contamination and to assess the suitability of porcelain grinding equipment for SEM–EDX sample preparation.

The findings of this study are expected to contribute to a better understanding of contamination risks associated with porcelain grinding equipment during SEM–EDX sample preparation. Furthermore, the results may provide useful information for selecting appropriate sample preparation methods and for interpreting silicon-related analytical results obtained from SEM–EDX measurements.

MATERIALS AND METHODS

Materials

The samples used in this study consisted of standard powdered materials free of silicon. Hydrotalcite ($Mg_6Al_2CO_3(OH)_{16}\cdot 4H_2O$) and sodium chloride (NaCl) were selected as the model powdered materials. Distilled water (aquabidest) and technical-grade ethanol were used for cleaning the mortar and pestle. All chemicals were used as received without further purification. The samples were analyzed using a Phenom ProX SEM–EDX system. Conductive carbon tape was used for sample mounting,

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and the samples were sputter-coated using a Coxem sputter coater.

Cleaning of Sample Preparation Equipment

All porcelain mortars and pestles were washed three times with distilled water. The final rinse was performed using ethanol to remove any residual contaminants. Subsequently, the equipment was either air-dried or dried in a drying cabinet. Neither tissue paper nor cloth was used for drying to minimize the risk of contamination.

Sample Preparation

Three replicate samples were prepared for each treatment. The first sample remained unground. The second sample was ground using a new porcelain mortar and pestle. The third sample was ground using a previously used porcelain mortar and pestle exhibiting visible surface scratches resulting from repeated laboratory use. Grinding was performed under ambient conditions.

After completion of the three preparation procedures, each sample was oven-dried at 105 °C for 2 hours to ensure complete drying and moisture removal. The samples were subsequently subjected to SEM–EDX analysis.

SEM-EDX Analysis

Conductive carbon tape was attached to aluminum pin stubs corresponding to the number of samples to be analyzed. Each sample was gently dispersed onto the carbon tape surface. The samples were sputter-coated with gold (Au) using a sputter coater at a current of 4 mA for 100 seconds. Morphological observations were performed using SEM equipped with a backscattered electron (BSD) detector. Morphological analysis of the samples was performed using SEM equipped with a backscattered electron (BSD) detector at an accelerating voltage of 10 kV.

The elemental composition of the samples was determined by EDX analysis. The SEM was operated at an accelerating voltage of 15 kV for the analyses. EDX measurements were performed in triplicate., including two spot analyses at different locations and one area analysis covering the entire SEM micrograph. The results obtained from the three sample preparation methods were compared to evaluate potential silicon contamination.

RESULTS AND DISCUSSION

Sample preparation for SEM–EDX analysis involved mounting the samples onto conductive carbon tape. In this study, the samples were nonconductive or exhibited low electrical conductivity; therefore, they were sputter-coated with

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gold (Au) prior to SEM analysis (Widiyastuti D.A., 2016). In addition to gold, samples may also be coated with an approximately 10-nm-thick conductive layer such as silver (Ag), platinum (Pt), palladium (Pd), or chromium (Cr) (Erik Luyk, 2019).

This study generated surface morphology images and elemental composition data for samples prepared using three different preparation procedures, namely unground samples, samples ground using a new mortar and pestle (Figure 1), and samples ground using a worn mortar and pestle with extensive surface scratches (Figure 2). The elemental composition of the samples was determined by EDX analysis. This section focuses on the EDX results..

Figure 1 shows a new porcelain mortar with no visible surface scratches.. In contrast, Figure 2 shows a used porcelain mortar exhibiting surface scratches caused by repeated use that cannot be removed by washing. These scratches are expected to accumulate progressively with repeated use. The elemental composition of the porcelain mortar may represent a potential source of contamination, that may be detected by EDX analysis.

SEM–EDX analysis was performed using three repeated EDX measurements on a single morphology, consisting of two spot analyses s (indicated by red arrows) and

one area analysis analysis covering the entire image area.

.**Figure 1.** New porcelain mortar used in this study.

Figure 2. Previously used porcelain mortar exhibiting visible surface scratches

The spot and region EDX analyses for the hydrotalcite sample are shown in the morphology results in Figure 3, while those for the sodium chloride sample are presented in Figure 4.

Spot EDX analyses were performed at two different locations based on

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predefined criteria, including variations in dark and bright contrast within the morphology, which may reflect differences in atomic number contrast. In addition, the analysis points were also selected based on differences in particle morphology. Region EDX scanning provides an overall elemental analysis across the entire morphology.

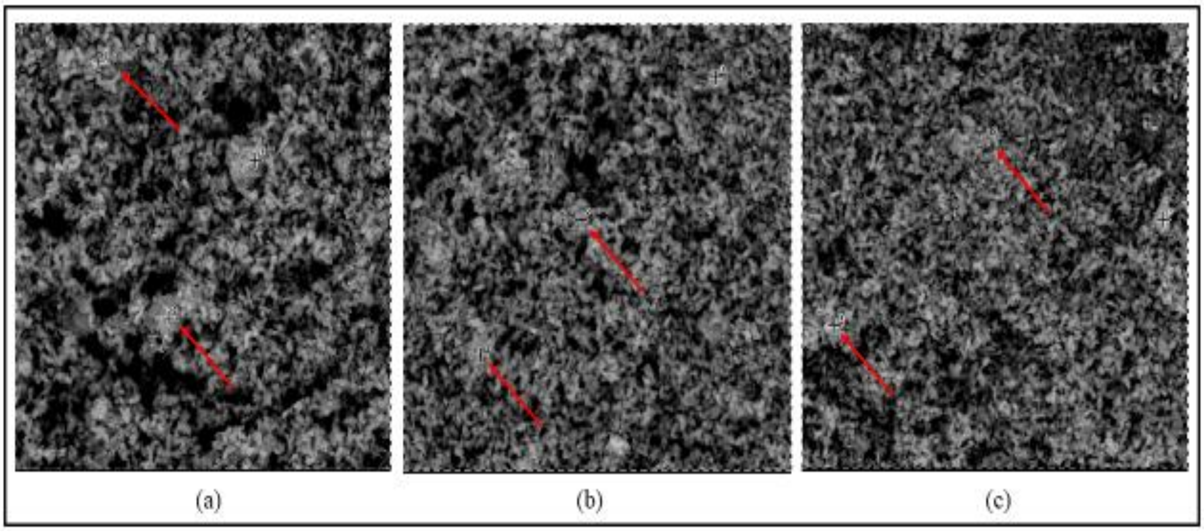

Figure 3. SEM micrographs and EDX results of hydrotalcite samples prepared using different grinding procedures

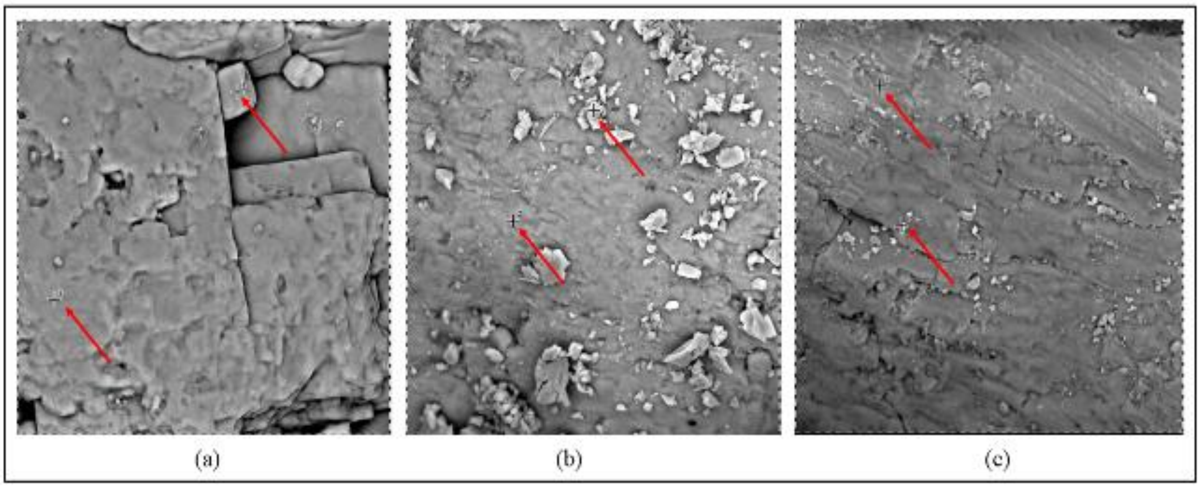

Figure 4. SEM micrographs and EDX results of sodium chloride samples prepared using different grinding procedures.

The hydrotalcite and sodium chloride samples without grinding treatment were used as reference samples to evaluate the initial presence of silicon. The EDX spectra of the hydrotalcite sample are shown in Figure 5, while the EDX spectra of the sodium chloride sample are presented in Figure 6.

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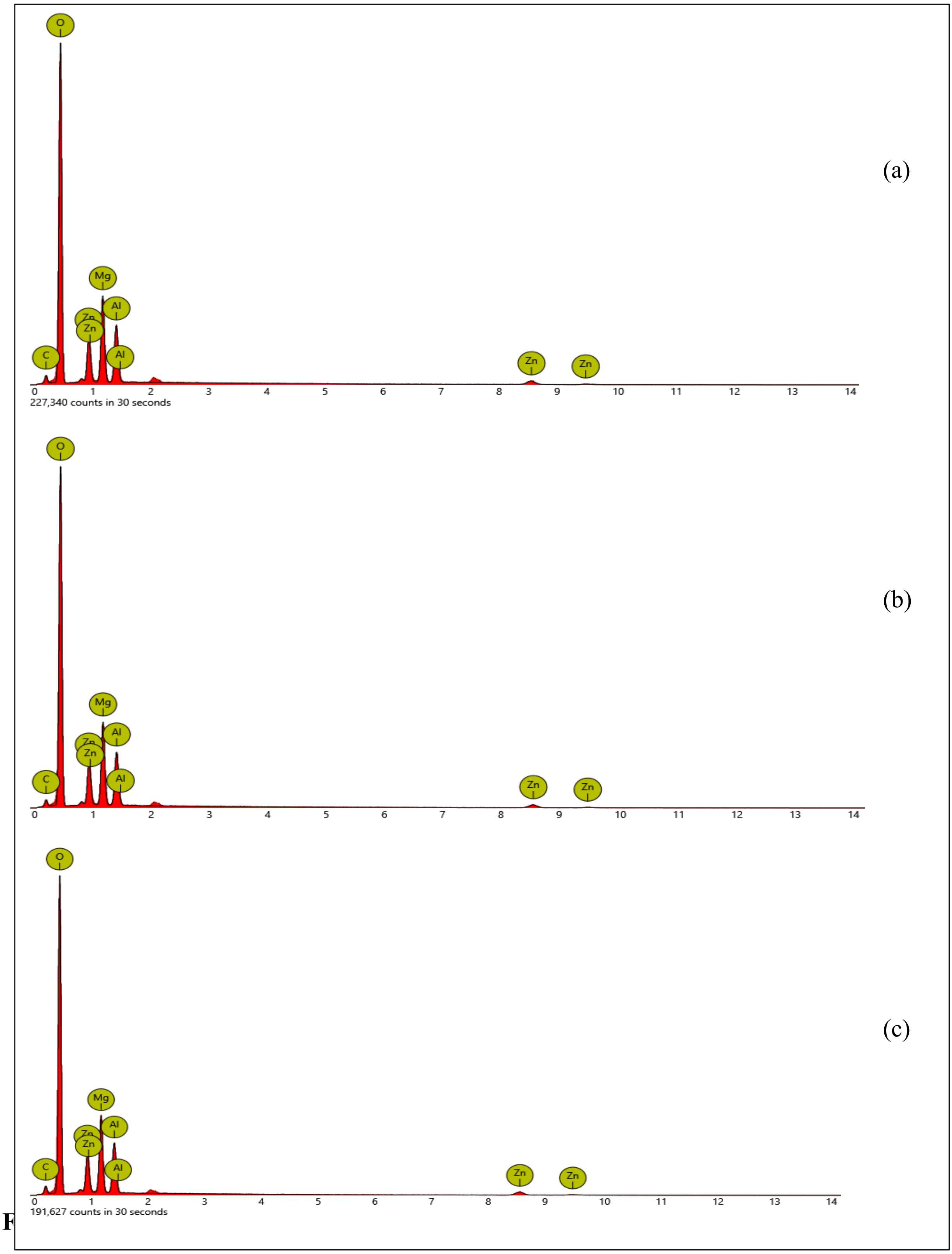

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with a New Mortar and Pestle; (c) Grinding with a Used Mortar and Pestle

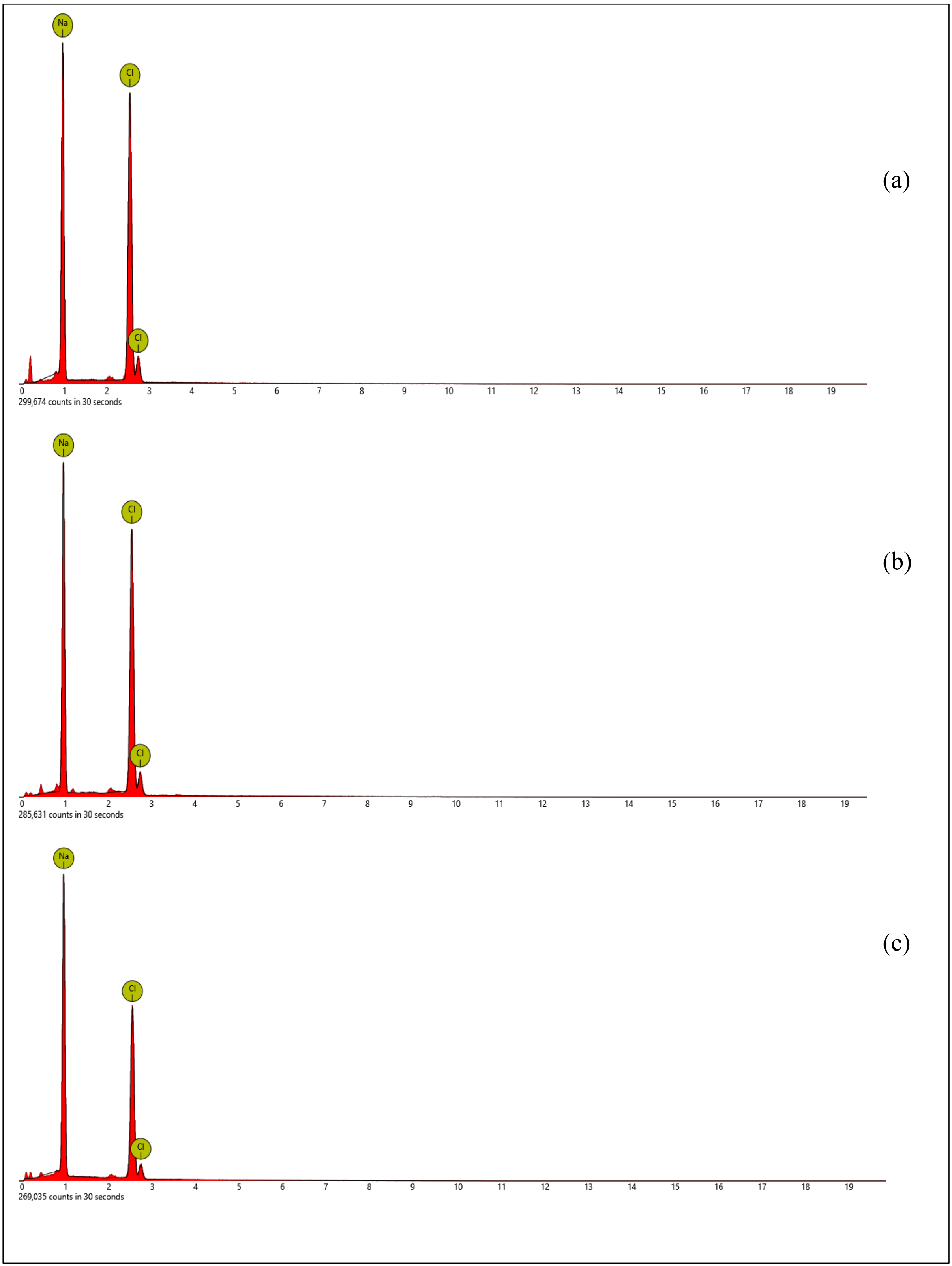

Figure 6. EDX Spectra of the Sodium Chloride Sample: (a) No Grinding Treatment; (b) Grinding with a New Mortar and Pestle; (c) Grinding with a Used Mortar and Pestle

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The EDX spectrum in Figure 5 shows that all sample preparation procedures consistently resulted in the detection of the same elements in hydrotalcite, namely C, O, Zn, Mg, and Al. The detected elements are consistent with the chemical formula of hydrotalcite, $Mg_6Al_2CO_3(OH)_{16}\cdot 4H_2O$. Hydrogen (H) cannot be detected by EDX because it has an atomic number of 1. As a result, hydrogen has no inner electron shells, and therefore no electron transitions occur that would produce characteristic X-ray emission.

Table 1. Average Elemental Composition of Hydrotalcite Determined by SEM–EDX

Hydrotalcite Sample Treatments	**Weight (wt %)**				
	C	**O**	**Zn**	**Mg**	**Al**
Without Grinding	5,51	59,62	10,94	14,10	9,85
Grinding with a New Mortar and Pestle	4,23	59,95	11,20	14,33	10,30
Grinding with a Used Mortar and Pestle (Heavily Scratched)	5,21	57,82	13,03	13,92	10,01
Average	4,98	59,13	11,72	14,12	10,05
Standard Deviation (SD)	0,67	1,14	1,14	0,21	0,23
Relative Standard Deviation (%RSD)	13,39	1,94	9,75	1,46	2,25

Table 2. Average Elemental Composition of Sodium Chloride Determined by SEM–EDX

Sodium Chloride Sample Treatments	**% Berat (wt %)**	
	Na	**Cl**
Without Grinding	42,99	57,01
Grinding with a New Mortar and Pestle	47,19	52,81
Grinding with a Used Mortar and Pestle (Heavily Scratched)	48,31	51,69
Average	46,16	53,84
Standard Deviation (SD)	2,81	2,81
Relative Standard Deviation (%RSD)	6,1	5,2

Standard deviation (SD) and relative standard deviation (RSD) were calculated using the following equations:

$$SD = \sqrt{\frac{\sum_{i=1}^{n}(x_i - \bar{x})^2}{n-1}}$$

$$RSD = \frac{SD}{\bar{x}} \times 100\%$$

where:

SD = standard deviation

RSD = relative standard deviation

x_i = the i-th value of x

$\bar{x}$ = mean value

n = number of data points

The relative standard deviation (RSD), or coefficient of variation (CV), was calculated to evaluate the dispersion of the obtained data. The criteria for CV assessment are as follows (Pandriadi et al., 2023):

- CV < 15% is classified as low variability, indicating that the data are relatively homogeneous with little variation around the mean.
- CV between 15% and 30% is classified as moderate variability, indicating a reasonably controlled level of variation in the data.
- CV > 30% is classified as high variability, indicating significant variation relative to the mean value.

The %RSD values for each element in the hydrotalcite samples across the three sample preparation procedures presented in Table 1 ranged from a minimum of 1.46% (Mg) to a maximum of 13.39% (C). All %RSD values were below 15%, indicating low variability according to the coefficient of variation (CV) classification. These results indicate that the three sample preparation procedures did not significantly influence the elemental composition of the hydrotalcite samples.

To further support these findings, SEM–EDX analysis was additionally performed using the same sample preparation procedures on sodium chloride (NaCl) samples. The SEM–EDX results for the NaCl samples are presented in Figure 4 and Table 2. Figure 4 shows the presence of sodium (Na) and chlorine (Cl) were detected, which is consistent with the chemical composition of sodium chloride.

The %RSD values obtained for each element in sodium chloride across the three sample preparation procedures presented in Table 2 were 6.1% for Na and 5.2% for Cl. All RSD values were below 15%, corresponding to a low CV category. This indicates that the three sample preparation procedures did not significantly influence the measured elemental composition of the sodium chloride samples.

Silicon (Si), the suspected contaminant, was not detected in Figures 5 and 6 or in Tables 1 and 2. This finding indicates that the Si concentration was below the detection limit of the SEM–EDX instrument. According to the technical specifications and application notes provided by Phenom/Thermo Fisher, the EDX system is capable of performing elemental analysis from boron (B) to californium (Cf) and provides quantitative analysis based on a silicon drift detector (SDD). However, a universal limit of detection (LOD) is generally unavailable, as the actual detection limit depends on the analytical conditions used.

Recent studies indicate that SEM systems equipped with Silicon Drift

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Detectors (SDD) can achieve detection limits of approximately 0.1 wt.% (≈1000 ppm) under routine analytical conditions for common elements in mineral and rock samples. At this concentration level, the quantitative performance of SEM/SDD-EDS has been reported to be comparable to that of conventional Wavelength Dispersive Spectroscopy (WDS) for major and minor element analyses (Chen et al., 2023).

The Si concentration in all samples remained below the detection limit of the instrument (< 0.1 wt.%), indicating that any Si potentially introduced during sample preparation remained below the detectable threshold. These results suggest that the use of a porcelain mortar and pestle does not contribute significantly to silicon contamination during the grinding process for SEM–EDX sample preparation.

Potential contamination may also originate from other stages of the sample preparation process, as well as from variations in sample characteristics and processing temperature. Consequently, particular attention should therefore be given to sample preparation procedures to minimize contamination and ensure data reliability, given that SEM–EDS is capable of providing accurate semi-quantitative elemental analysis.

CONCLUSION

Samples in this study were prepared using three different procedures: no grinding, grinding with a new porcelain mortar and pestle, and grinding with an aged porcelain mortar and pestle exhibiting numerous surface scratches. SEM–EDX analysis demonstrated that the Si content in all samples remained below the instrumental detection limit, which has been reported to be below 0.1 wt.% under routine analytical conditions.

These findings indicate that using a porcelain mortar and pestle for powder sample preparation under ambient conditions does not significantly contribute to silicon (Si) contamination during SEM–EDX sample preparation. Therefore, porcelain mortars and pestles can be considered suitable tools for powder sample preparation for SEM–EDS analysis under the experimental conditions investigated in this study. The findings of this study provide valuable information for laboratories that routinely perform SEM–EDX analysis

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REFERENCES

Ali, A et al. 2023. Mineral Characterization Using Scanning Electron Microscopy (SEM): A Review of the Fundamental, Advancements, and Research Directions. MDPI, 13, 12600. https://doi.org/10.3390/app132312600

Chen, Y. et al. (2023). Quantifying common major and minor elements in minerals/rocks by economical desktop scanning electron microscopy/silicon drift detector energy-dispersive spectrometer (SEM/SDD-EDS). Solid Earth Sciences. 8, 49-67. https://doi.org/10.1016/j.sesci.2022.12.002

Erik Luyk. 2019. Sputter Coating for SEM : How This Sample Preparation Technique Assists Your Imaging. https://www.thermofisher.com/blog/materials/sputter-coating-for-sem-how-this-sample-preparation-technique-assists-your-imaging/.

Indiani, E & Ngurah Ayu K.U. 2009. Keramik Porselen Berbasi Feldspar sebagai Bahan Isolator Listrik. Jurnal Telkomnika, 7(2), 83-92.

Mardika, H.P.K et al. 2024. Penggunaan SEM-EDX untuk Gambaran Ultrastruktural Kerusakan Jaringan pada Insang Ikan Nila Alibat Logam Berat Cadmium. Jurnal Simpul Inovasi, 1(2), 48-52

Mu'nisa, A et al. 2023. Teknik Manajemen dan Pengelolaan Laboratorium Kesehatan Veteriner. Makassar : FMIPA Universitas Negeri Makassar

Patriadi et al. 2023. Statistika Dasar. Jawa Barat : Penerbit Widina Media Utama

Samodra, F.P. 2025. Fungsi Mortar dan Alu: Peralatan Esensial Laboratorium. https://www.liputan6.com/feeds/read/5827762/fungsi-mortar-dan-alu-peralatan-esensial-laboratorium?page=5

Septiano, Alvin F et al. 2021. Analisa Citra Hasil *Scanning Electron Microscopy Energy Dispersive X-ray* (SEM EDX) Komposit Resin Timbal dengan Metode *Contrast to Noise Ratio* (CNR). Indonesian Journal of Mathematics and Natural Sciences, 44(2), 81-85

Soomro, H.M. et al. 2024. Elution of Divalent Cations from Iron Ore Mining Waste in an Indirect Aqueous Mineral Carbonation for Carbon Capture and Storage. MDPI, *16*(2), 836. https://doi.org/10.3390/su16020836

Sudrajat, A & A.P Bayuseno. 2014. Analisis Korosi dan Kerak Pipa Nickel Alloy N06025 pada Waste Heat Boiler. Jurnal Teknik Mesin S-1, 2(1), 40-45.

Toisuta, Juan G et al. (2024). Analisa Kapaitas Saluran Drainase Ruas Jl. Rijali Kota Ambon dan Alternatif Penanganannya. Jurnal Simetrik, 14(1), 829-836

Widianto, E. (2022). Mortar dan Alu : Pengertian, Fungsi, dan Cara Menggunakan. https://bloglab.id/mortar-dan-alu-pengertian-fungsi-dan-cara-menggunakan/

Widiyastuti, D.A. 2016. Pengamatan *Scanning Electron Microscopy* (SEM) pada Struktur dan Mineral Batuan dari Sungai Aranio Kabupaten Banjar. Jurnal Sains dan Terapan Politeknik Hasnur, 4(2), 16-21