

Determination of geological sample ages using nuclear analysis methods

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Abstract: *The determination of geological sample ages is a fundamental aspect of geoscience, offering insights into the history and development of Earth's crust. This study employs the Uranium-Thorium-Lead (U-Th-Pb) method to accurately date sediment samples from the Viet Phuong 2 quarry in Loc Thanh, Binh Phuoc province, Vietnam. Using Total Reflection X-Ray Fluorescence (T-XRF) and gamma spectrometry, the study measured lead, uranium, and thorium concentrations in the samples. The analysis revealed that the ages of the samples ranged from approximately 228 million to 435 million years, consistent with the geological history of the area. Specifically, the U-Pb dating method provided a more accurate and consistent age range, while the Th-Pb method showed slightly higher variability. These results confirm the reliability of the U-Th-Pb dating method and suggest its broader application in geological studies.*

Keywords: *Geological age determination, Gamma spectrometry, T-XRF*

1. Introduction

The uranium-lead dating method is one of the oldest and most refined radiometric dating methods, with a history that dates to the discovery of radioactivity in 1896. In 1902, Ernest Rutherford and Frederick Soddy discovered that radioactive decay produces alpha particles, which can be used to determine the age of rocks and minerals. However, it was not until the 1940s that the uranium-lead dating method was fully developed, thanks to the pioneering work of Arthur Holmes and Clair Cameron Patterson (Faure, 1987)

Arthur Holmes, a British geologist, was the first to use uranium-lead dating to determine the age of rocks (Arthur Holmes, 1911). In 1911, he used the method to date rocks from the British Isles, and later, he applied it to rocks from Africa and Australia. Holmes also developed the concept of geologic time scale, which provided a framework for understanding the history of the Earth.

In the 1940s, Clair Cameron Patterson, an American geochemist, began to refine the uranium-lead dating method by developing techniques to measure the isotopic composition of lead (Patterson, 1956). Patterson's work was critical in establishing the age of the Earth at 4.55 billion years, a value that is still accepted today. He also discovered that lead contamination from industrial sources was affecting the accuracy of uranium-lead dating, which led to efforts to reduce lead pollution.

Since the work of Holmes and Patterson, the uranium-lead dating method has been continuously refined and improved. Advances in analytical techniques have enabled scientists to measure extremely small amounts of isotopes with high precision, and to date rocks that are billions of years old. This study utilizes the U-Th-Pb method to accurately date geological samples from the Viet Phuong 2 quarry in Binh Phuoc province, Vietnam. By combining modern analytical techniques, such as T-XRF and gamma spectrometry, we aim to improve the accuracy and consistency of geological age determination, providing valuable insights into the region's geological history.

2. Materials and Method

Sampling location

The survey area is Viet Phuong 2 quarry (Fig.1) in Can Duc hamlet, Loc Thanh Commune, Loc Ninh District, Binh Phuoc Province, Vietnam. The quarry is quarried and reveals a series of sedimentary rocks belonging to the third member of the Chau Thoi Formation, which is known for its sedimentary deposits dating back to the Triassic period. The quarry is approximately 7.5 km from the standard section near Can Le Bridge, making it a key location for geological studies due to its well-preserved sedimentary layers

(Cronan, 1972; Herron, 1988; Krumbein & Sloss, 1951; Mỹ et al., 1994; Quý et al., 2014; Thanh & Khúc, 2005).



Fig. 1. Sampling area location

The geological structure of the Viet Phuong 2 quarry features more than 20 lithological layers, primarily consisting of siltstone interbedded with sandstone, and in some areas, conglomerates and pebbles embedded within the sandstone. The structural dip ranges from 40 to 60 degrees, with a dip azimuth direction of 310 degrees (Quoc Tuan, 2021). This complex sedimentary structure makes the quarry an ideal site for studying the geological history of the region.



Fig. 2. Some images of 12 sediment samples

A total of 12 sediment samples were systematically collected from different exposed sections of the Viet Phuong 2 quarry. To enhance the understanding of the study's methodologies and findings, Fig. 2 provides visual documentation of the geological samples analyzed. This figure includes images of the sediment samples taken from various sections of the Viet Phuong 2 quarry, along with close-up views of the sedimentary structures and textures.

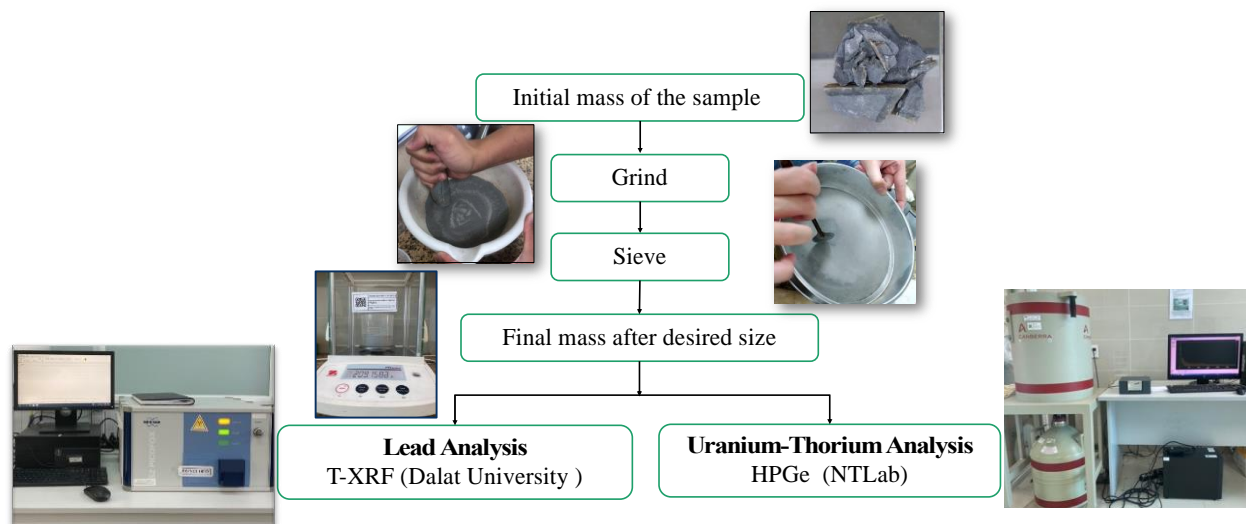


Fig. 3. Summary diagram of the sample preparation

Figure 3 provides a detailed overview of the sample preparation process utilized to guarantee consistency and minimize contamination. Initially, the samples were meticulously dried, followed by crushing and sieving to achieve particles smaller than 0.2 mm. Subsequently, each sample was carefully divided into segments designated for lead content analysis and uranium-thorium analysis. This diagram effectively outlines each critical step, from the initial collection to the precise handling procedures, ensuring the methodological soundness and reliability of the subsequent analytical measurements.

3. Method

U-Pb dating method involves measuring the isotopic ratios of Uranium and Lead in a sample. Uranium has two naturally occurring isotopes, ^{238}U and ^{235}U , which decay into Lead isotopes ^{206}Pb and ^{207}Pb respectively (Blackburn et al., 2013; Condon et al., 2017; Dalrymple, 2004). The ratio of these isotopes can be used to calculate the age of the sample using the decay constant. U-Pb dating method is generally used for dating rocks and minerals, but it can also be used for dating sedimentary rocks and sediment samples that contain small amounts of U and Pb.

Th-Pb dating method is like U-Pb dating but uses the decay of Thorium isotopes to Lead isotopes. Thorium also has two isotopes, ^{232}Th and ^{230}Th , which decay into Lead isotopes ^{208}Pb and ^{206}Pb respectively. The ratio of these isotopes can be used to calculate the age of the sample. Th-Pb dating is often used to date materials that are older than those that can be dated by U-Pb dating method.

The decay systems, decay constants (λ), and half-lives ($T_{1/2}$) are show in Eq (1), Eq (2):

$$^{238}\text{U} \rightarrow ^{206}\text{Pb} + 8\ ^4\text{He}; \lambda_{238} = 1.55125 \times 10^{-10} \text{ a}^{-1}; T_{1/2} = 4.468 \cdot 10^9 \text{ years} \quad (1)$$

$$^{232}\text{Th} \rightarrow ^{208}\text{Pb} + 7\ ^4\text{He}; \lambda_{232} = 4.948 \times 10^{-11} \text{ a}^{-1}; T_{1/2} = 1.4 \cdot 10^{10} \text{ years} \quad (2)$$

The decay constant of ^{232}Th being shorter than that of ^{238}U indicates that ^{232}Th has a longer half-life, meaning it decays more slowly. This suggests that Th-Pb dating should be suitable for older samples. However, in practice, U-Pb dating is often used for samples up to several billion years old, while Th-Pb dating is generally applied to samples older than 100 million years. The basic age equation is:

$$T = \frac{1}{\lambda} \ln \left(1 + \frac{n_D}{n_P} \right) \quad (3)$$

where T is the age, n_D refers to a molar quantity isotope of Pb (^{206}Pb , and ^{208}Pb), n_P refers to the respective molar quantity isotope of Uranium (or Thorium), and λ is the decay constant for each respective radioactive isotope ^{238}U , ^{232}Th .

Uranium-Thorium Analysis

The analysis of Uranium (^{238}U) and Thorium (^{232}Th) isotopes was conducted using Ortec high-purity Germanium (HPGe) gamma spectrometry. The gamma spectra of the samples were recorded and analyzed using the GammaVision-32 software, with efficiency calibration performed using the ANGLE-3 software, distributed by ORTEC. The gamma spectrometry system, cooled by the X-Cooler III, allowed precise measurement of gamma rays, detecting energy levels from 3 keV upwards (Ngan Thy et al., 2024) From the processing of the recorded gamma spectrum, the specific radioactivity of the parent isotope in the collected sample, A (Bq kg^{-1}), was calculated using the following Eq (4):

$$A = \frac{S}{\varepsilon' \times m \times f \times t \times K_c \times K_w} \quad (4)$$

where S is the net peak area (count); ε' is the attenuation corrected efficiency; f is the branching ratio of the peak gamma energy emitted by the isotope; m is the sample mass (kg); t is the collection live time (s) of the collect, K_c is the correction factor for the nuclide decay during the counting $K_c = \frac{T_{1/2}}{\ln(2)t_s} \left[1 - e^{-\frac{\ln(2)t_s}{T_{1/2}}} \right]$,

where $T_{1/2}$ is the half-life of the nuclide in question, and t_s is the elapsed real clock time during the measurement, and K_w is the correction factor for the radioactive decay considered from the time of the sample collection to the point that the sample is measured by the spectroscopy $K_w = e^{\left(\frac{\ln(2)t_w}{T_{1/2}} \right)}$, where t_w is the elapsed clock time from the time the sample was taken to the beginning of the measurement (Canberra Industries, 2006).

The half-life of ^{238}U (4×10^9 years) is much larger than the half-life of the daughter ^{234}Th (24 days). Thus, the ^{238}U activity is determined via the gamma energy peaks at 63.3 keV and 92.6 keV of ^{234}Th . The half-life of ^{232}Th (14×10^9 years) is much larger than the half-life of the daughter ^{228}Ra (5.7 years) with much larger than the half-life of the daughter ^{228}Ac (6.15 hours). Thus, the ^{232}Th activity is determined via the energy peaks of 338.3 keV and 911.1 keV of ^{228}Ac . Moreover, all the K_s and K_w correction factors equal 1 in this case.

The isotopic ratios of uranium and thorium in the samples were used to determine their content, which were then used in age calculations. The formula calculates the content of the radioactive isotope in the sample:

$$m = \frac{M \times A}{\lambda \times N_A} \tag{5}$$

where m is the content of the radioactive isotope of interest (g/g); M is atomic mass of the isotope of interest (u); A is specific activity of the isotope of interest (Bq/g); N_A is Avogadro constant; and λ is the half-life constant of the isotope of interest (year⁻¹)

Lead Analysis

In this research, the lead (Pb) content in geological samples was measured using the S2 PICOFOX™ TXRF spectrometer, a specialized device used for trace elemental analysis, provided by Dalat University. The S2 PICOFOX spectrometer operates based on the principle of Total Reflection X-ray Fluorescence (TXRF). It is advantageous for its ability to perform multi-element analysis with minimal sample preparation and no matrix effects (Towett et al., 2013). The spectrometer utilizes a high-energy X-ray tube operating at a voltage of 50 kV with a maximum power of 50 W, employing a molybdenum (Mo) target to generate X-rays (Towett et al., 2013). This high-energy X-ray source is crucial for exciting the atoms within the sample, causing them to emit characteristic X-rays at specific wavelengths. By measuring the intensity of these emitted X-rays, the concentration of lead (Pb) within the sample can be accurately determined (Sang et al., 2020). The S2 PICOFOX™ TXRF spectrometer is equipped with a silicon drift detector (SDD), known for its high sensitivity and low noise, which significantly enhances the precision of trace element quantification, including elements like lead (Pb) with L-line energy at 10.55 keV (Towett et al., 2013). During the analysis, the sample is homogenized and deposited as a thin layer on a quartz glass carrier. This setup ensures that the X-ray fluorescence emitted by the sample is accurately detected and analyzed to determine lead content.

Age Determination

From the lead content results, the lead content ²⁰⁶Pb and ²⁰⁸Pb were calculated based on the natural abundances of 24.1% and 52.4%, respectively. Finally, the age results of each sample were calculated using the two U-Pb and Th-Pb methods from the Eq. (6), Eq. (7), respectively:

$$T_{U-Pb} = \frac{1}{\lambda_{U-238}} \ln \left(1 + \frac{238 \times m_{Pb-206}}{206 \times m_{U-238}} \right) \tag{6}$$

$$T_{Th-Pb} = \frac{1}{\lambda_{Th-232}} \ln \left(1 + \frac{232 \times m_{Pb-208}}{208 \times m_{Th-232}} \right) \tag{7}$$

where T_{U-Pb} , T_{Th-Pb} is sample's age (year) using the two U-Pb and Th-Pb methods, respectively; m_i is the content of the radioactive isotope of interest i (g/g); and λ_i is the half-life constant of the isotope of interest i (year⁻¹).

4. Results and discussion

The analysis of 12 sediment samples from the Viet Phuong 2 quarry in Loc Thanh, Binh Phuoc province, provides compelling evidence regarding their geological ages. The U-Th-Pb dating methods applied to these samples reveal an age range predominantly clustering around the Middle Triassic period, aligning with the established understanding of the Chau Thoi Formation's geological timeline (Quoc Tuan, 2021; Quý et al., 2014).

Tab. 1. Summary of U-Th-Pb age determination results

Sample ID	U-Pb Age (Ma)	Th-Pb Age (Ma)
S1	247.09 ± 2.90	245.84 ± 4.29
S2	261.71 ± 4.30	237.12 ± 4.95
S3	230.07 ± 4.74	243.99 ± 5.12
S4	435.39 ± 6.45	422.88 ± 6.16

S5	228.74 ± 3.72	258.63 ± 6.40
S6	227.40 ± 3.71	266.92 ± 4.93
S7	244.32 ± 3.13	236.81 ± 4.68
S8	237.46 ± 2.80	249.29 ± 4.50
S9	218.48 ± 2.48	229.86 ± 5.22
S10	242.09 ± 2.56	237.87 ± 6.36
S11	252.19 ± 3.13	248.47 ± 6.73
S12	231.67 ± 3.38	234.44 ± 4.65

U-Pb and Th-Pb Age Determination

The U-Pb dating method yielded ages ranging from 218.48 to 435.39 million years, while the Th-Pb method showed ages from 229.86 to 422.88 million years. A detailed summary of these results is presented in Table 1. The mean age for the 12 samples was calculated as 254.72 ± 16.79 million years for U-Pb and 259.34 ± 15.17 million years for Th-Pb. These results predominantly fall within the Triassic period, with sample S4 showing a notably older age, which may suggest the presence of Permian materials.

Revised Mean Ages

After excluding the outlier from Sample S4, the revised mean ages were 238.29 ± 3.32 million years (U-Pb) and 244.48 ± 3.79 million years (Th-Pb). This adjustment provides a more accurate reflection of the geological ages, closely aligning with the expected Middle Triassic age for the formation (Quoc Tuan, 2021; Quý et al., 2014).

Statistical Analysis

The statistical analysis, including variance and median, further reinforces the reliability of the U-Pb method due to its lower variability compared to Th-Pb. The standard deviation and variance suggest that U-Pb dating offers a more precise estimation of sedimentary ages, supporting its preference in geological studies (Blackburn et al., 2013; Condon et al., 2017; Dalrymple, 2004; Faure, 1986). Table 2 provides a comprehensive summary of these statistical measures.

Tab. 2. Results of the statistical analysis of geological sample Age

Parameter	12 Samples (Original)	11 Samples (Excluding S4)
Mean (U-Pb, Ma)	254.72 ± 16.79	238.29 ± 3.32
Mean (Th-Pb, Ma)	259.34 ± 15.17	244.48 ± 3.79
Standard Deviation	58.15 (U-Pb) / 52.56 (Th-Pb)	12.59 (U-Pb) / 11.03 (Th-Pb)
Variance	3381.30 (U-Pb) / 2762.88 (Th-Pb)	158.43 (U-Pb) / 121.62 (Th-Pb)
Median (U-Pb, Ma)	239.78	237.46
Median (Th-Pb, Ma)	244.92	243.99
Range	216.91 (U-Pb) / 193.02 (Th-Pb)	43.23 (U-Pb) / 37.06 (Th-Pb)

Interpretation and Implications

The results underscore the utility of U-Th-Pb dating methods in sedimentary environments, particularly highlighting the robustness of the U-Pb method in providing accurate and consistent age determinations. The presence of a significant outlier in Sample S4 prompts further investigation into potential geological complexities, such as intrusion of older materials or methodological anomalies during sample collection (Buatois & Mángano, 2011; Khuc, 2000).

These findings enhance our understanding of the region's geological history, confirming significant sedimentary deposition during the Middle Triassic. The minor discrepancies observed between the U-Pb and Th-Pb methods reflect the inherent differences in uranium and thorium geochemistry, emphasizing the

importance of selecting the appropriate method based on the sample characteristics (Faure, 1986; Khuc, 2000).

Discussion

The analysis of sediment samples from the Viet Phuong 2 quarry offers important insights into the geological history of the Chau Thoi Formation. The ages of these samples primarily fall within the Middle Triassic period, aligning with previous studies and geological maps that highlight the significance of Triassic sedimentary deposits in shaping the region's stratigraphy (Quoc Tuan, 2021; Quý et al., 2014).

The U-Pb dating method consistently provided ages ranges from 218.48 to 435.39 million years, while the Th-Pb method yielded a slightly narrower range from 229.86 to 422.88 million years. This demonstrates the precision and reliability of the U-Pb method, which exhibited lower variability and tighter error margins compared to the Th-Pb method. The preference for U-Pb is supported by established decay constants and minimal lead loss, factors that enhance its suitability for sedimentary age determination, as referenced in studies by Blackburn et al. (2013), Condon et al. (2017), Dalrymple (2004), Faure (1986).

Sample S4, which suggests an older age, poses an interesting anomaly. It may indicate the presence of older geological materials or potential methodological errors during sample collection. This highlights the importance of comprehensive sampling and methodological rigor in ensuring accurate geological interpretations (Buatois & Mángano, 2011; Khuc, 2000).

Incorporating a statistical approach, the revised mean ages, after excluding the outlier, are 238.29 ± 3.32 million years for U-Pb and 244.48 ± 3.79 million years for Th-Pb. This adjusted range offers a more accurate reflection of the geological age, corroborating the Middle Triassic age of the Chau Thoi Formation (Quoc Tuan, 2021; Quý et al., 2014). The statistical measures, including variance and median, further bolster the reliability of the U-Pb method, reinforcing its utility in geological studies (Blackburn et al., 2013; Condon et al., 2017; Dalrymple, 2004; Faure, 1986).

The minor discrepancies observed between the U-Pb and Th-Pb dating methods are expected due to the differing geochemical behaviors of uranium and thorium. These variations emphasize the importance of selecting appropriate methodologies based on the characteristics of the samples to enhance the accuracy of age determinations and geological interpretations (Faure, 1986; Khuc, 2000).

This study confirms significant sedimentary deposition during the Middle Triassic period following Permian limestone formations in Binh Phuoc province. The findings also indicate possible geological processes, such as folding, faulting, or later intrusions, that could influence the stratigraphic sequence (Buatois & Mángano, 2011; Khuc, 2000).

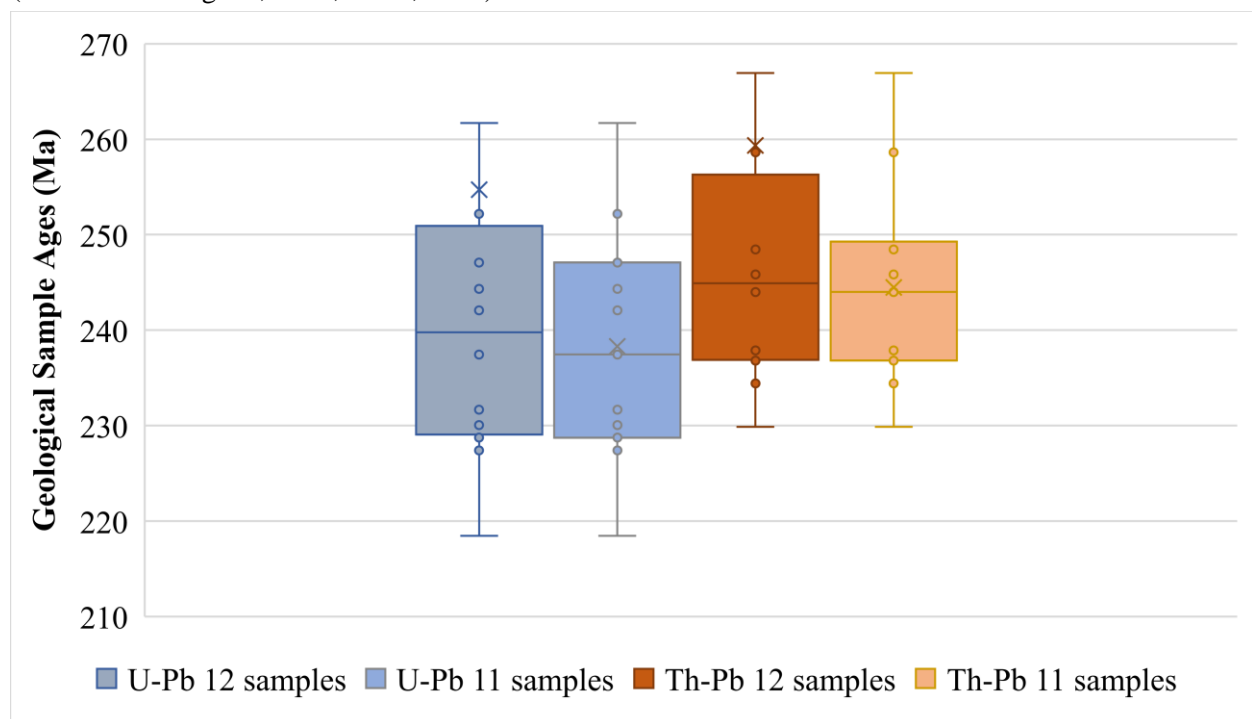


Fig. 4. Statistical distribution of geological sample age

A bar graph (illustrated in Figure 4) provides a visual comparison of the U-Pb and Th-Pb methods,

demonstrating their close agreement in most cases and highlighting any outliers. This visual representation supports the conclusion that the U-Pb method offers more precise and reliable age determinations, making it a robust tool for dating sedimentary deposits in complex geological settings like the Viet Phuong 2 quarry.

Overall, the study confirms the efficacy of U-Th-Pb dating methods in sedimentary environments, contributing valuable insights into the geological history of Southeast Vietnam. The combination of nuclear analysis for age determination and sedimentary structure analysis provides a comprehensive understanding of the region's sedimentary processes, supporting the hypothesis that these deposits belong to the Middle Triassic period.

This research not only enriches the regional geological timeline but also contributes to global geoscientific efforts by demonstrating the importance of precise dating techniques in understanding Earth's history and managing its resources sustainably. The methodologies and findings of this study offer practical significance for other geological investigations, providing a reliable framework for future geochronological studies and applications in resource exploration, environmental assessments, and beyond.

5. Conclusion

This study advances the understanding of sedimentary ages within the Chau Thoi Formation in Southeast Vietnam through the application of precise U-Th-Pb dating methods. By establishing a more accurate geological timeline, the research contributes significantly to refining the regional stratigraphy and understanding the complex geological history of the area.

U-Th-Pb Dating Methods: These methods enhance the precision in determining the age of sedimentary deposits, offering a more refined chronological framework for the Chau Thoi Formation. Accurate dating is crucial for reconstructing the geological history and stratigraphic relationships within the region.

The comparison of U-Pb and Th-Pb dating techniques highlights the importance of selecting appropriate methodologies based on sample characteristics, thereby enhancing the precision and reliability of geological interpretations.

Comparison of Dating Techniques: U-Pb and Th-Pb methods are compared to determine their suitability based on sample characteristics. This comparison underscores the necessity of selecting the right technique to improve the accuracy of age determinations and geological interpretations.

These findings have practical implications not only for resource exploration and environmental assessments in Southeast Vietnam but also for similar geological contexts worldwide.

Practical Implications: The study's outcomes are relevant for practical applications such as resource exploration, environmental assessment, and land-use planning. The enhanced understanding of geological timelines aids in locating and managing natural resources.

The study's methodologies provide a reliable framework for future geochronological studies, aiding in the exploration of natural resources and the reconstruction of past environmental conditions.

Methodological Framework: By presenting a robust methodological framework, this study sets a precedent for future research, ensuring consistency and reliability in geochronological investigations.

As a result, this research offers valuable insights into both regional geology and broader geoscientific endeavors, underscoring the importance of accurate dating techniques in understanding Earth's history and managing its resources sustainably.

Geoscientific Contributions: The research not only enhances regional geological knowledge but also contributes to global geoscientific efforts by demonstrating the importance of precise dating techniques in sustainable resource management and historical reconstruction.

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