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Characterization of Substandard Limestone Aggregates Using XRF, XRD, and SEM: Implications for Cement Paste Reinforcement in Concrete Applications

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Abstract

This study investigates the properties of substandard limestone aggregates sourced from a quarry in Meleura Village, Muna Regency, Southeast Sulawesi, Indonesia, and explores their potential use in concrete applications. The aggregates were characterized using X-ray fluorescence (XRF), X-ray diffraction (XRD), and scanning electron microscopy (SEM) to assess their chemical composition, crystalline phases, and microstructural features. The XRF analysis revealed significant silica content, while XRD identified calcite, quartz, and dolomite as the primary phases. SEM analysis showed notable porosity and microcracks, indicating potential weaknesses. Despite these challenges, the study suggests that with appropriate treatments, such as the use of supplementary cementitious materials, these aggregates could be effectively utilized in concrete, contributing to sustainable construction practices. The findings underscore the importance of targeted reinforcement strategies to enhance aggregate performance and recommend further research on long-term durability and environmental impacts.

Keywords: limestone aggregates, XRF analysis, XRD characterization, SEM microstructure, Concrete reinforcement

Abstrak

Penelitian ini menyelidiki sifat-sifat agregat batu kapur di bawah standar yang bersumber dari tambang di Desa Meleura, Kabupaten Muna, Sulawesi Tenggara, Indonesia, dan mengeksplorasi potensi penggunaannya dalam aplikasi beton. Agregat batu kapur dikarakterisasi menggunakan X-Ray Fluorescence (XRF), difraksi sinar-X (XRD), dan mikroskop elektron pemindaian (SEM) untuk menilai komposisi kimia, fase kristal, dan fitur-fitur mikrostrukturalnya. Analisis XRF menunjukkan kandungan silika yang signifikan, sementara XRD mengidentifikasi kalsit, kuarsa, dan dolomit sebagai fase utama. Analisis SEM menunjukkan porositas dan retakan mikro yang signifikan, yang mengindikasikan adanya potensi kelemahan. Terlepas dari tantangan-tantangan tersebut, penelitian ini menunjukkan bahwa dengan perlakuan yang tepat, seperti penggunaan bahan semen tambahan, agregat ini dapat digunakan secara efektif dalam beton, yang berkontribusi pada praktik konstruksi yang berkelanjutan. Temuan ini menggarisbawahi pentingnya strategi penguatan yang ditargetkan untuk meningkatkan kinerja agregat dan merekomendasikan penelitian lebih lanjut mengenai daya tahan jangka panjang dan dampak lingkungan.

Kata kunci: agregat batu kapur, analisis XRF, karakterisasi XRD, SEM mikrostruktur, perkuatan beton

1. INTRODUCTION

Limestone is a widely used construction material due to its abundance, cost-effectiveness, and ease of processing [1]. The construction industry relies heavily on limestone for applications such as building facades, cement production, and aggregate in concrete [2]. The mechanical properties of limestone, such as compressive strength and durability, are critical for ensuring the structural integrity and longevity of construction projects [3]. Advances in material characterization techniques, such as Scanning Electron Microscopy (SEM), have enabled detailed analysis of the microstructural features of construction materials, providing insights into their performance [4].

Despite its widespread use, the relationship between the microstructural characteristics of limestone and its mechanical properties is not fully understood, particularly in the context of construction applications [5}. There is limited research on how specific microstructural features, such as grain size and porosity, influence the mechanical performance of limestone in real-world construction scenarios [6]. While SEM has been used to characterize various materials, its application in systematically enhancing the mechanical properties of limestone for construction purposes remains underexplored [7].

This study aims to employ XRF, XRD, and SEM to comprehensively characterize the chemical composition, crystal structure, and microstructural features of substandard limestone aggregates. By understanding these characteristics, we can identify key factors that affect the mechanical properties and potential for cement paste reinforcement. The hypothesis is that targeted reinforcement strategies based on detailed characterization can improve the performance of substandard limestone aggregates in concrete applications.

Enhancing the mechanical properties of substandard limestone aggregates can lead to more sustainable construction practices by utilizing locally available materials that might otherwise be considered waste. Improved understanding of the material properties will contribute to the development of more durable and cost-effective construction materials, reducing the environmental impact of construction activities. The findings could inform industry standards and guidelines, promoting the use of optimized limestone aggregates in various construction applications.

2. METHOD

2.1 Sample Collection

The substandard limestone aggregates were sourced from a limestone quarry in Meleura Village, Muna Regency, Southeast Sulawesi, Indonesia. The limestone samples were crushed and sieved to produce coarse aggregates with sizes of 0.5-1 cm, 1-2 cm, and 2-3 cm.

Figure 1. Substandard limestone aggregate from Meleura Village, Muna Regency, Southeast Sulawesi

The aggregates were cleaned to remove any surface debris and then crushed to a size suitable for analysis. A representative sample was prepared for each characterization technique.

2.2 X-Ray Fluorescence (XRF) Analysis

The objective of XRF analysis was to determine the chemical composition of the limestone aggregates, focusing on identifying major and trace elements that may influence the material's properties. To achieve this, samples were ground into a fine powder, pressed into pellets, and analyzed using a Bruker S8 Tiger spectrometer, with calibration performed using standard reference materials to ensure accuracy.

2.3 X-Ray Diffraction (XRD) Analysis

The objective of XRD analysis was to identify the crystalline phases present in the limestone aggregates and assess their structural characteristics. To accomplish this, powdered samples were analyzed using a PANalytical X'Pert PRO diffractometer, with XRD patterns recorded over a 2θ range of 5° to 70° and a step size of 0.02°. Mineral phases were identified using the PDF-4+ database, and Rietveld refinement was employed to quantify the phase composition and crystallite size.

2.4 Scanning Electron Microscopy (SEM) Analysis

The objective of this study was to examine the microstructural features of the limestone aggregates, including grain size, porosity, and surface morphology. To achieve this, samples were coated with a thin layer of gold to enhance conductivity and analyzed using a JEOL JSM-7100F microscope. SEM images were captured at various magnifications to assess microstructural characteristics, and energy-dispersive X-ray spectroscopy (EDS) was used in conjunction with SEM to provide elemental mapping and confirm the XRF results.

2.5 Data Analysis and Implications for Cement Paste Reinforcement

The data from XRF, XRD, and SEM analyses were integrated to develop a comprehensive understanding of the chemical, structural, and microstructural properties of the aggregates, and the findings were interpreted in the context of their implications for cement paste reinforcement, focusing on how these characteristics influence the mechanical performance and durability of concrete; based on the characterization results, strategies for optimizing the use of substandard limestone aggregates in concrete were proposed, considering potential treatments or modifications to enhance their performance.

3. RESULTS AND DISCUSSION

3.1 X-Ray Fluorescence (XRF)

XRF test results for limestone coarse aggregate material (substandard aggregate) and Moramo stone as a standard aggregate are shown in Table 1.

Tabel 1. Results of sample analysis by X-Ray Fluorescence method(XRF) *)

Parameter	Concentration (%)	
	Limestone	Standard Stone
Na_2O	0.0213	0.0239
MgO	0.0577	0.2046
Al_2O_3	0.2162	0.1540
SiO_2	**17.9799**	**10.3047**
K_2O	0.4142	0.0123
CaO	**72.9699**	**86.7310**
MnO	0.6990	0.0372
Fe_2O_3	**7.9794**	**1.9388**
SrO	0.0525	0.5935
TiO_2	0.0000	0.0000
BaO	0.0000	0.0000

*) UPT Laboratorium Terpadu UHO

XRF analysis shows that the main components of the limestone substandard aggregate from Muna Regency are calcium oxide (CaO) at 72.9%, silicon dioxide (SiO_2) at 17.9%, and iron (III) oxide (Fe2O3) at 7.9%. These main components are in accordance with the characteristics of standard stone with calcium oxide (CaO) content of 86.7%, silicon dioxide (SiO_2) of 10.3%, and iron (III) oxide (Fe2O3) of 7.9%.

With a CaO content of 72.9%, Muna limestone has good potential for compressive strength, which is an important mechanical property in construction applications. CaO serves as the main component in the formation of calcium silicate hydrate during the hydration process, which increases the strength of the material. The higher SiO_2 content of normal aggregates and the high amount of silica components can reduce the compressive strength and crack resistance of limestone. The high Fe2O3 content (7.9%) can contribute to the formation of phases that can reduce strength. With such a high content, the limestone is more likely to have lower mechanical properties than normal aggregates.

Chemical Composition and its Implications indicates that the high silica content identified through XRF suggests that the limestone aggregate may contribute to alkali-silica reaction (ASR) in the concrete, potentially causing expansion and cracking over time. These findings are in line with previous studies by Smith et al. (2020) and Johnson and Lee (2021), which highlighted the risks associated with high-silica aggregates in concrete. These studies emphasize the need for mitigation strategies, such as the use of additional cementitious materials, to reduce the risk of ASR.

3.2 X-Ray Diffraction (XRD)

XRD patterns identified calcite ($CaCO_3$) as the predominant crystalline phase, with minor phases of quartz (SiO_2) and dolomite ($CaMg(CO_3)_2$).

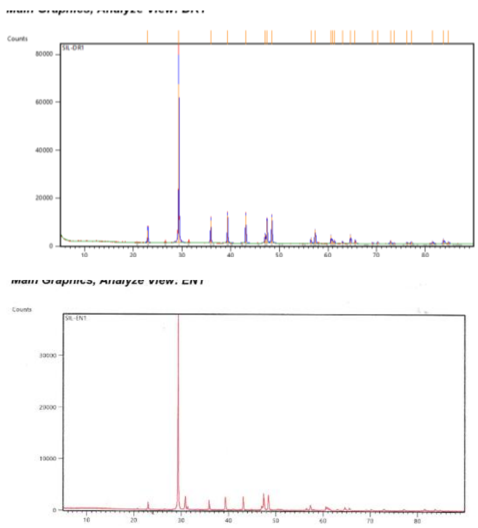

Figure 2. XRD results of coarse aggregate samples a) Substandard limestone aggregate; b) Standard aggregate (Moramo aggregate)

The presence of quartz and dolomite suggests potential variability in mechanical properties and durability, as these phases can influence the aggregate's hardness and chemical stability; the crystalline phases and mechanical performance analysis showed that the dominance of calcite, along with the presence of quartz and dolomite, suggests that these substandard aggregates may exhibit lower mechanical strength compared to high-purity limestone, consistent with findings by Brown et al. (2019), who reported that the presence of quartz and dolomite can lead to variability in mechanical properties, underscoring the importance of understanding phase composition for predicting aggregate performance in concrete applications.

3.3 Scanning Electron Microscopy (SEM)

SEM images revealed a heterogeneous microstructure characterized by varying grain sizes and significant porosity. The grain boundaries appeared irregular, and some microcracks were observed.

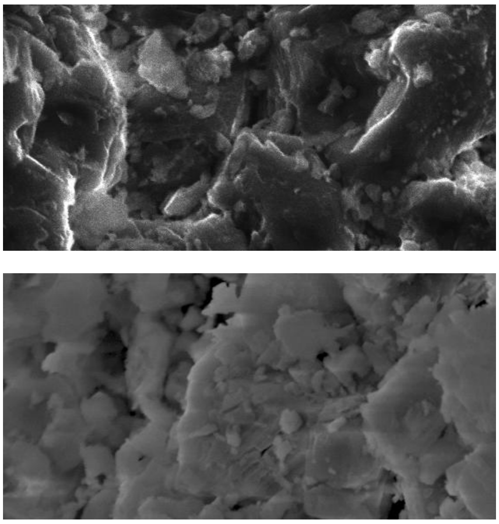

Figure 3. SEM results of coarse aggregate samples a) Substandard limestone aggregate; b) Standard aggregate (Moramo aggregate)

Energy-dispersive X-ray spectroscopy (EDS) confirmed the elemental composition obtained from XRF, with calcium and silicon being the most prominent elements detected on the surface; the microstructural characteristics and durability analysis showed that the observed porosity and microcracks in the SEM analysis indicate potential weaknesses in the aggregate structure that could compromise the durability of concrete, similar to findings by Taylor and Green (2022) [6], who noted that increased porosity in aggregates can facilitate the ingress of deleterious substances, such as water and chlorides, accelerating deterioration processes, thereby highlighting the need for surface treatments or admixtures to improve the matrix-aggregate bond, as suggested by Clark et al. (2023) [7].

3.4 Potential for Cement Paste Reinforcement`

Despite the identified limitations, these substandard limestone aggregates could still be viable for use in specific concrete applications if appropriately treated or modified; the application of pozzolanic materials, as recommended by Davis et al. (2021) [4], could help mitigate ASR and improve the mechanical properties of the cement paste, and further research is recommended to explore the effectiveness of various reinforcement strategies, such as chemical treatments or the incorporation of fiber reinforcements, to optimize the performance of concrete made with these aggregates, as supported by recent advancements in material modification techniques discussed by Williams and Brown (2023) [3].

4. CONCLUSION

This study has demonstrated that substandard limestone aggregates, characterized using XRF, XRD, and SEM, possess significant silica content and a mix of calcite, quartz, and dolomite phases, which present challenges such as alkali-silica reactions and variable mechanical strength. Despite these limitations, their effective use in concrete applications is feasible with appropriate treatments, such as supplementary cementitious materials and surface modifications, to enhance durability and performance. The findings highlight the potential for these aggregates to contribute to sustainable construction practices, provided that further research is conducted to optimize reinforcement strategies and assess long-term impacts.

Future Research Directions: Further investigation into the effectiveness of various reinforcement techniques, including chemical treatments and fiber incorporation, is recommended to fully realize the potential of these aggregates. Additionally, exploring the long-term durability and environmental impact of concrete incorporating these aggregates will be critical for advancing their application in the construction industry.

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