ANALYSIS OF THE APPLICATION OF LIMESTONE WASTE IN CEMENTITIOUS SYSTEMS BASED ON A SYSTEMATIC LITERATURE REVIEW

ANÁLISE DA APLICAÇÃO DO RESÍDUO DE CALCÁRIO EM SISTEMAS CIMENTÍCIOS A PARTIR DE UMA REVISÃO SISTEMÁTICA DE LITERATURA

ANÁLISIS DE LA APLICACIÓN DE RESIDUOS DE CALIZA EN SISTEMAS CEMENTICIOS A PARTIR DE UNA REVISIÓN SISTEMÁTICA DE LA LITERATURA

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ABSTRACT
Limestone is a sedimentary rock composed mainly of calcite and aragonite, both composed of $CaCO_3$, and may contain magnesium (dolomite). Its mining causes environmental problems due to the inadequate disposal of the waste generated. This study aims to identify the applicability of limestone residue from rocks in cementitious systems and to evaluate the techniques used to analyze these residues and their products. The method used was bibliographic research with Systematic Literature Review (SLR) as a tool. Limestone waste is generally calcitic and originates from quarries that extract carbonate rocks. Chemical and/or mineralogical analysis of the residue is not always performed, and there is no predominance of characterization regarding particle size, shape, and texture. Furthermore, most studies do not describe the state of the residue at the collection site and do not detail the preparation of the residue for use. Limestone waste has generally been applied in the form of a filler, partially replacing fine aggregate or cement in mortars. Its use in mortar production is viable and beneficial and can maximize new research on the use of waste, as well as help reduce the extraction of non-renewable natural resources.

KEYWORDS
Limestone waste; cementitious systems; characterization techniques; limestone filler; carbonate rocks

RESUMO
O calcário é uma rocha sedimentar composta principalmente por calcita e aragonita, ambas compostas por $CaCO_3$, podendo conter magnésio (dolomita). A sua mineração traz problemas ambientais devido a deposição inadequada dos resíduos gerados. Esse trabalho objetiva identificar a aplicabilidade do resíduo de calcário proveniente de rochas em sistemas cimentícios e avaliar a partir de quais técnicas esses resíduos e seus produtos vêm sendo analisados. Utilizou-se como método a pesquisa bibliográfica com Revisão Sistemática de Literatura (RSL) como ferramenta. O resíduo de calcário geralmente é calcítico e originado de pedreiras que fazem a extração de rochas carbonáticas. A análise química e/ou mineralógica dos resíduos nem sempre é realizada, bem como não há predominância de caracterização quanto à granulometria, forma e textura das partículas. Além disso, a maioria dos estudos não descrevem o estado do resíduo no local de coleta e não detalham a preparação do resíduo para uso. O resíduo de calcário vem sendo aplicado geralmente na forma de fíler, substituindo parcialmente o agregado miúdo ou cimento em argamassas. O seu uso na produção de argamassa é viável e benéfico, podendo maximizar novas pesquisas sobre o uso do rejeito, bem como auxiliar na redução da extração de recursos naturais não renováveis.

http://dx.doi.org/10.29183/2447-3073.MIX2025.v11.n1.189-203
ISSN: 2447-0899 (IMPRESSA) | 2447-3073 (ONLINE)
Mix Sustentável | Florianópolis | v.11 | n.1 | p.189-203 | JUN. | 2025

PALAVRAS-CHAVE

Resíduo de calcário; sistemas cimentícios; técnicas de caracterização; fíler calcário; rochas carbonáticas

RESUMEN

La caliza es una roca sedimentaria compuesta principalmente de calcita y aragonito, ambas compuestas de $CaCO_3$*, y puede contener magnesio (dolomita). Su extracción ocasiona problemas ambientales debido a la inadecuada disposición de los residuos generados. Este trabajo tiene como objetivo identificar la aplicabilidad de residuos calizos de rocas en sistemas cementosos y evaluar las técnicas utilizadas para analizar estos residuos y sus productos. El método utilizado fue la investigación bibliográfica teniendo como herramienta la Revisión Sistemática de Literatura (RSL). Los residuos de piedra caliza son generalmente calcíticos y proceden de canteras que extraen rocas carbonatadas. No siempre se realizan análisis químico y/o mineralógico de los residuos, no existiendo predominio de caracterización en cuanto a tamaño de partícula, forma y textura. Además, la mayoría de los estudios no describen la condición de los residuos en el lugar de recolección ni detallan la preparación de los residuos para su uso. Los residuos de piedra caliza se aplican generalmente en forma de relleno, sustituyendo parcialmente al agregado fino o al cemento en los morteros. Su uso en la producción de mortero es viable y beneficioso, y puede maximizar nuevas investigaciones sobre el uso de residuos, además de ayudar a reducir la extracción de recursos naturales no renovables.*

PALABRAS CLAVE

Residuos de caliza; Sistemas cementicios; Técnicas de caracterización; Relleno de caliza; Rocas carbonatadas

1. INTRODUCTION

The use of mineral waste has been gaining ground in the replacement and manufacture of construction materials and components to contribute to their proper final disposal, as well as to reduce the environmental impacts generated by their extraction.

Thus, countries have been adopting policies aimed at optimizing the use of local materials for several reasons, and this may be due to the housing deficit or the scarcity of component materials for agglomerated mixtures in certain regions (BÉDÉRINA et al., 2005). In addition, the increase in construction activity has led to a huge demand for good quality construction and building materials, leading to a considerable increase in the mining rate of natural fine aggregates and the consequent generation of large amounts of solid and powder waste (CHOUHAN et al., 2019, 2020). In addition, other industries in various segments benefit from their use, such as paper, plastics, soil conditioner for agriculture, animal feed supplements, toothpaste, medicines and cosmetics (HALDAR, 2020).

Limestone is one of the most prominent and commonly available rocks found worldwide; It has bright colors and a smooth surface and is used for decorative purposes in the form of ornaments, tiles, and flooring in buildings and monuments around the world (CHOUHAN et al., 2020). In addition to availability, Haldar (2020) states that sedimentary rocks are relatively easy to cut into blocks and are long-lasting, making them commonly used in architecture, sculptures, historical monuments and buildings around the world.

Limestone is a sedimentary rock composed mainly of calcite (trigonal crystal system) and aragonite (orthorhombic crystal system), both composed of calcium carbonate - CaCO3 (BARROS et al., 2020), and may contain magnesium in its composition, which can be characterized as dolomitic (HALDAR, 2020).

Mining activity brings serious environmental problems due to the inadequate disposal of the waste generated, generally in open areas (TAMMAM; UYSAL; CANPOLAT, 2022), landfills or other inadequate disposal sites (BARROS et al., 2020), such as soil waterproofing, closing of river courses, partial degradation of the environment, contamination of rivers and soil, among others. Barros et al. (2020) point out that such waste can be in the form of solids, resulting from tailings in mines or processing units, or sludge, generated as a semi-liquid substance composed of particles from sawing and polishing processes.

Despite the discussions that have taken place in recent decades, there is still a search to mitigate the increase in the amount of waste (TAHER et al., 2021). Jamshidi (2024) reiterates that a solution to this problem still lies in recycling limestone waste to produce materials for various applications in construction projects, such as concrete, artificial stones and cement, and points out that such a measure leads to sustainable development through the added value of extraction and job creation.

Limestone waste has been studied timidly, but research indicates that it can be beneficially used in cementitious/agglomerated systems, partially replacing components of mixtures. Thus, initiatives to evaluate the appropriate disposal of this waste as construction material are presented as a viable alternative.

To study and analyze aspects related to the composition and structure of components in cementitious systems, it is necessary to use available characterization techniques that help understand their behavior when subjected to different experimental projects, which can range from the analysis of replacement of components in the mixture to its response when subjected to environmental adversities. Generally, the techniques are accompanied by instruments that provide data related to the analyzed material and, often, accompanied by analysis software. The results obtained can help in the discussion about the usability of a given material, as well as point out efficiency and durability criteria.

The main objective of this work is to identify the applicability of limestone residue from rocks in cementitious systems and to evaluate the techniques used to analyze these residues and their products, as well as to verify the importance of these techniques in their characterization.

2. METHODOLOGY

This study used bibliographic research as a strategy with Systematic Literature Review (SLR) as a tool. SLR is a type of scientific research that has gained recognition in different international scientific scenarios due to its high level of evidence (GALVÃO; RICARTE, 2019). The authors also define that to ensure the quality of the review, it is necessary to comply with its development stages, which are:

a) delimitation of the issue to be addressed in the review;

b) selection of bibliographic databases for consultation and collection of material;

c) development of strategies for advanced search;

d) selection of texts and systematization of information found.

In SLR, it is essential to record all research stages, both for the sake of reproducibility in other research, and to show that the process follows all previously defined stages (RAMOS; FARIA; FARIA, 2014). Figure 01 outlines the stages for the present study.

The databases used were Periódicos Capes and Science Direct due to the researchers' familiarity with the platforms and availability of institutional use, as well as these being widely used inmaterials, concentrating renowned journals at an international level.

To begin the search process, keywords in English were discussed that would direct to studies that met the objective of the study, that is, studies that addressed limestone waste and the characterization tests generally used, such as X-ray Fluorescence and Diffraction (XRF and XRD), Scanning Electron Microscopy (SEM), Thermogravimetric Analysis (TG/DTG) and BET surface area (BET), for example. It was assumed that studies that used XRD, XRF, SEM, TG and BET techniques could perform and present other characterization techniques and, if the use and relevance of other techniques were found, these would be included in the discussions.

The definition of Boolean operators was also an important step, since these, if used appropriately, optimize the search, helping to identify and/or eliminate papers according to their relevance to the research, but their use is quantitatively limited in a single search in Science Direct (eight, maximum). Thus, the following keywords and Boolean operators were defined in Periódicos Capes: "limestone waste" AND (cement OR paste OR mortar OR concrete) AND (XRD OR XRF OR EDX OR SEM OR TG OR BET). In Science Direct, two sets of keywords were used due to the limitation of Boolean operators and were: "limestone waste" AND (cement OR paste OR mortar OR concrete) AND (XRD OR XRF OR EDX) and "limestone waste" AND (cement OR paste OR mortar OR concrete) AND (SEM OR TG OR BET).

To direct the authors to relevant works on the topic, it was determined that these should be present in the title, abstract or keywords. To this end, the search was performed in the advanced search field of the Science Direct platform; the Periódicos Capes platform automatically performs this search in the main field. Eight and two articles were found in Periódicos Capes and Science Direct, respectively, but there was an overlap between them, totaling eight distinct articles published in the last 17 years. Considering the reasonable number of articles, as well as the relevance of the journals responsible for the publications, the eight articles were considered suitable for analysis.

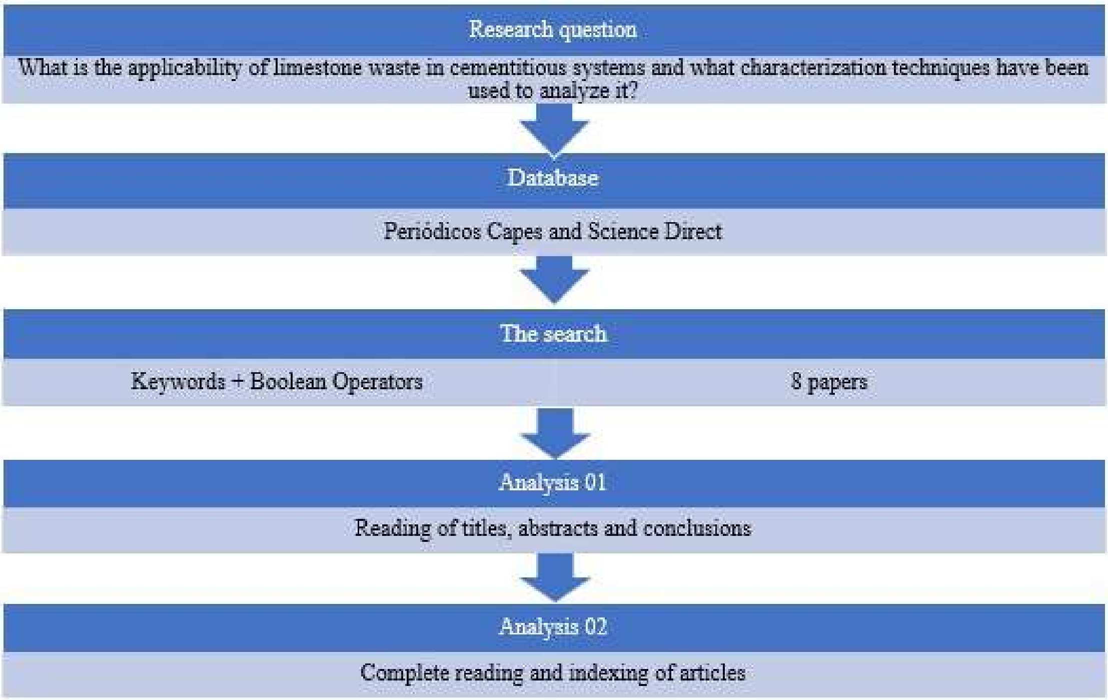

Figure 1: Research design.

Source: The authors.

Table 01 lists the identified works in chronological order of publication, indicating the title, source, database and application of limestone residue.

Although the study by Barros et al. (2020) is not necessarily about limestone residue in an agglomerated system, its characterization results proved to be relevant to the discussions.

No.	Title	Source/ authorship	Periódicos Capes	Science Direct	Application of residue
1	Use of limestone obtained from waste of the mussel cannery industry for the production of mortars	Ballester et al. (2007)	X		Mortar
2	Encapsulation of limestone waste in concrete after arsenic removal from drinking water	Chintalapat et al. (2009)	X		Concrete
3	Gainful utilization of dimensional limestone waste as fine aggregate in cement mortar mixes	Chouhan et al. (2019)	X	X	Mortar
4	Ecological bricks from dimension sto-ne waste and polyester resin	Barros et al. (2020)	X	X	Ecological brick
5	Investigating use of dimensional limestone slurry waste as fine aggregate in mortar	Chouhan et al. (2020)	X		Mortar
6	The Influence of Crystalline Admixtures on the Properties and Microstructure of Mortar Containing By-Products	Hodul; Žižková; Borg (2020)	X		Mortar
7	Effects of alternative ecological fillers on the mechanical, durability, and microstructure of fly ash-based geopolymer mortar	Tammam; Uysal; Canpolat (2022)	X		Mortar
8	Effect of Waste Filler Materials and Recycled Waste Aggregates on the Production of Geopolymer Composites	Tammam et al. (2023)	X		Mortar

Table 1: Data from the articles analyzed.
Source: The authors.

3. RESULTS AND DISCUSSIONS

To aid in understanding the discussions that follow, Appendix 1 is presented at the end of this paper, summarizing: the objectives and conclusions of the articles analyzed; the origin, form of use and preparation of the residue, as well as the details of the characterization techniques used. Next, the results of the characterizations of the limestone residue and the products generated from its application are compared and discussed. Such discussion is relevant to understanding the objective of each technique, as well as pointing out familiarity, or not, in its use.

3.1 Chemical composition and physical properties

The chemical composition, when determined and presented, was performed using XRF. Table 02 presents a summary of the chemical compositions of the limestone residues studied, as well as specific physical properties. The residues are calcitic (magnesium content below 5%). The results also show that the limestone residues used have calcium oxide (CaO) as the main component, followed by quartz (SiO2). To analyze the mineralogy of the residue, XRD tests were performed.

The studies by Barros et al. (2020) and Hodul et al. (2020) used XRD (Figure 02) to analyze the mineralogy of limestone residue. Figure 02, in the upper part, shows predominant peaks of calcite (1 - CaCO3), dolomite (2 - CaMg(CO3)2), quartz (3 - SiO2) and kaolinite (4 - Al2O3). In the lower part, the diffractogram also shows a limestone with a predominantly calcite composition (C - CaCO3) with smaller peaks of quartz (Q - SiO2). The peaks of greatest intensity are located at similar 2θ angles. The mineralogies can be correlated with the chemical compositions presented in Table 02.

Source/ authorship	Chemical composition (%)							Physical properties				
	CaO	SiO_2	Al_2O_3	Fe_2O_3	MgO	P_2O_5	K_2O	Specific Gravity	Density (g/cm^3)	Fineness modulus	Water absorption (%)	Blaine (cm^2/g)
Ballester et al. (2007)	-	-	-	-	-	-	-	-	-	-	-	-
Chintalapat et al. (2009)	-	-	-	-	-	-	-	-	-	-	-	-
Chouhan et al. (2019)	37,85	23,5	3,1	1,94	-	-	-	2,70	0,96-1,29	1,29-1,99	3,50-8,80	
Barros et al. (2020)	89,49	1,08	-	0,48	2,6	0,1	0,07	-	2,76	-	-	-
Chouhan et al. (2020)	-	-	-	-	-	-	-	2,59	0,96	1,29	8,77	
Hodul; Žižková; Borg (2020)	-	-	-	-	-	-	-	-	-	-	-	-
Tammam; Uysal; Canpolat (2022)	51,97	4,93	0,82	0,58	0,58	-	-	2,79	-	-	-	2500
Tammam et al. (2023)	51,97	4,93	0,82	0,58	0,58	-	-	2,70			4,60	2500

Table 2: Chemical compositions and physical properties of waste.

Source: The authors.

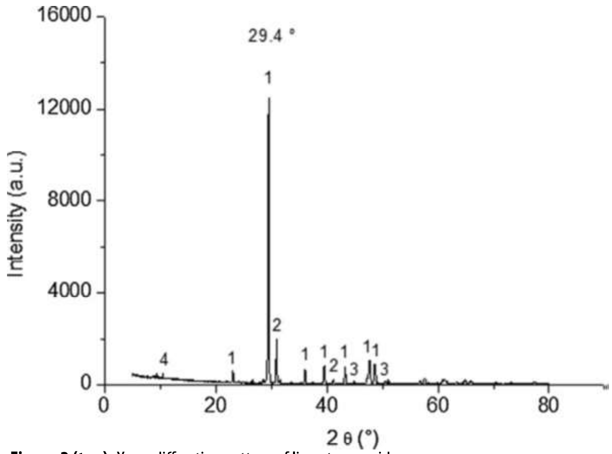

Figure 2 (top): X-ray diffraction pattern of limestone residue.

Source: Barros et al. (2020).

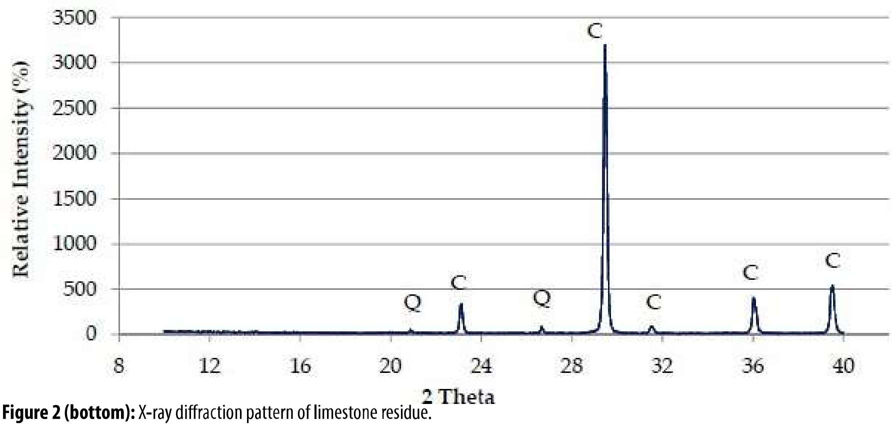

Figure 2 (bottom): X-ray diffraction pattern of limestone residue.

Source: Hodul; Žižková; Borg (2020).

The limestone residue was used in different ways, both in terms of its granulometry and its replacement function. Although Chintalapati et al. (2009) do not indicate the method for defining granulometry, the authors state that the limestone had particle dimensions of 0.5 to 1 mm, consistent with the granulometric range for sand according to the American standard.

Figure 03 shows the granulometric distribution of the aggregates used by Ballester et al. (2007) (top) and Chouhan et al. (2019) (bottom), characterizing the use of the residue in the form of filler. Ballester et al. (2007) detail that the granulometry was determined using a laser analyzer.

Chouhan et al. (2020) presented more details on the granulometry of residues from limestone sludge (DLSW), where 36.4% of the particles were smaller than 0.15 µm, appearing finer in relation to river sand, of which only 6.3% of the particles were smaller than 6.3 µm.

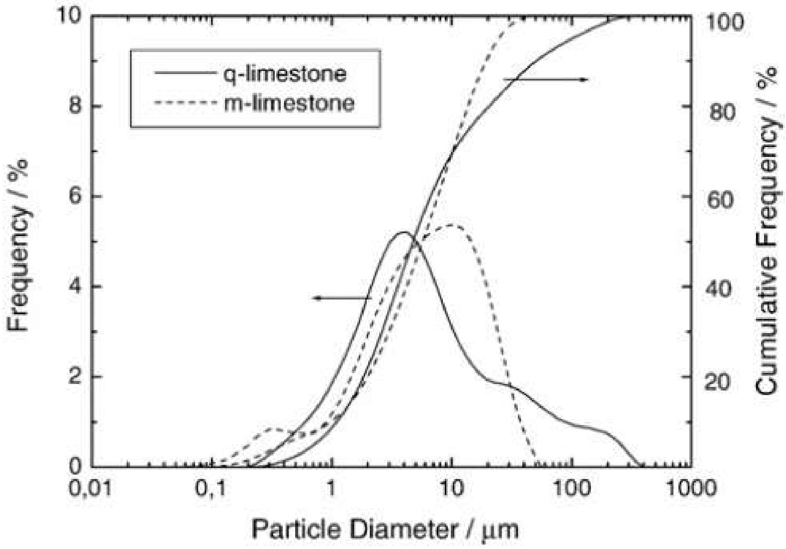

Figure 3 (top): Granulometric distribution of limestone residues.
Source: Ballester et al. (2007).

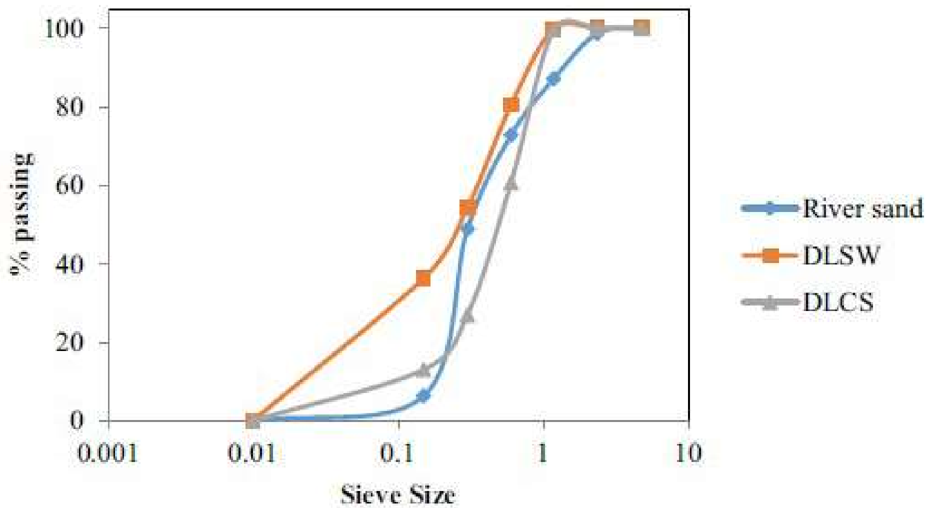

Figure 3 (bottom): Granulometric distribution of limestone residues.
Source: Chouhan et al. (2019)..

The beams were analyzed at 502 days of age, exhibiting an average compressive strength of 32.4 MPa and an elasticity modulus of 34.8 GPa. This analysis was selected to eliminate the influence of compressive strength variation on the experimental results. This criterion was necessary because different degrees of corrosion required varying acceleration periods (ranging from 10 to 45 days). Stabilizing compressive strength was crucial to ensure that the analyses exclusively reflected the effects of corrosion on structural performance.

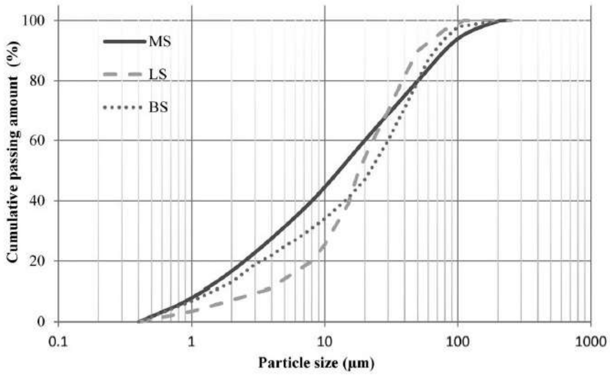

Figure 4: Granulometric distribution of limestone residues (LS).
Source: Tammam et al. (2022).

Thus, regarding the granulometry defined for the waste from the analyzed works, this is presented in different ranges depending on the applicability of the waste, but generally in the format of:

a) limestone sand partially replacing fine aggregate (BALLESTER et al., 2007);

b) limestone filler partially replacing fine aggregate (CHINTALAPATI et al., 2009; CHOUHAN et al., 2019, 2020; TAMMAM et al., 2023; TAMMAM; UYSAL; CANPOLAT, 2022);

c) limestone filler partially replacing cement (HODUL; ŽIŽKOVÁ; BORG, 2020);

d) limestone filler with only the addition of resin (BARROS et al., 2020).

3.2. Microstructure

SEM was the technique used to analyze the morphology of the limestone residues. Figure 5 shows the microstructure of the two limestone residues studied by Ballester et al. (2007), where it is possible to observe different particle sizes and shapes. The quarry limestone (top) has rounded particles ranging in size from 2 to 6 µm, and, in turn, the mussel limestone (bottom), not the focus of this study, has prismatic particles up to 14 µm in length and a small fraction of particles smaller than 1 µm, which is confirmed by the distribution of particle sizes. The authors justify the gain in strength precisely by the intercrossing of particles with prismatic morphology, a characteristic of mussel limestone, which can result in the formation of a network where the cement particles strongly bind to the aggregates after the solidification of the cement paste, thus increasing the strength of the mortar.

This result was also confirmed by Tammam et al. (2023), in which as the replacement rate of fine aggregate by limestone filler residues increased, there was also an improvement in compressive, tensile and flexural strengths due to the higher limestone content (up to 75%) increasing the reactive phase and reducing water absorption. Another relevant observation was that such different granular characteristics between limestones influenced the consistency of the mortars produced, where limestone with prismatic particles led to more workable mortars.

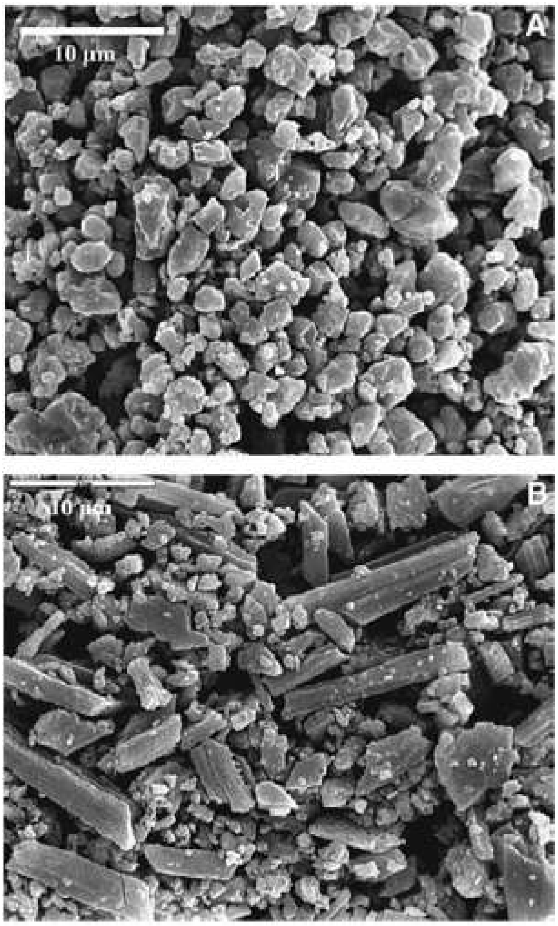

Figure 5: SEM images of limestone residues: (A) quarry limestone and (B) mussel limestone.
Source: Ballester et al. (2007).

Chouhan et al. (2019), when evaluating two residual forms of limestone (Figure 06), limestone slurry (DLSW) and crushed limestone sand (DLCS), identified that both have an irregular shape and rough texture, especially when compared to conventional river sand (RS), which has a smooth surface and rounded shape (CHOUHAN et al., 2019, 2020).

Corroborating the morphologies presented previously, the residues from Barros et al. (2020) and Hodul et al. (2020) have an irregular shape (Figure 07), probably related to calcium carbonate grains. Both studies used residues from quarries, from the cutting of limestone rocks. Tammam et al. (2023) also presented the SEM result for the limestone filler studied, with no discussions about its shape; however, according to Figure 07, the residue also has an irregular shape.

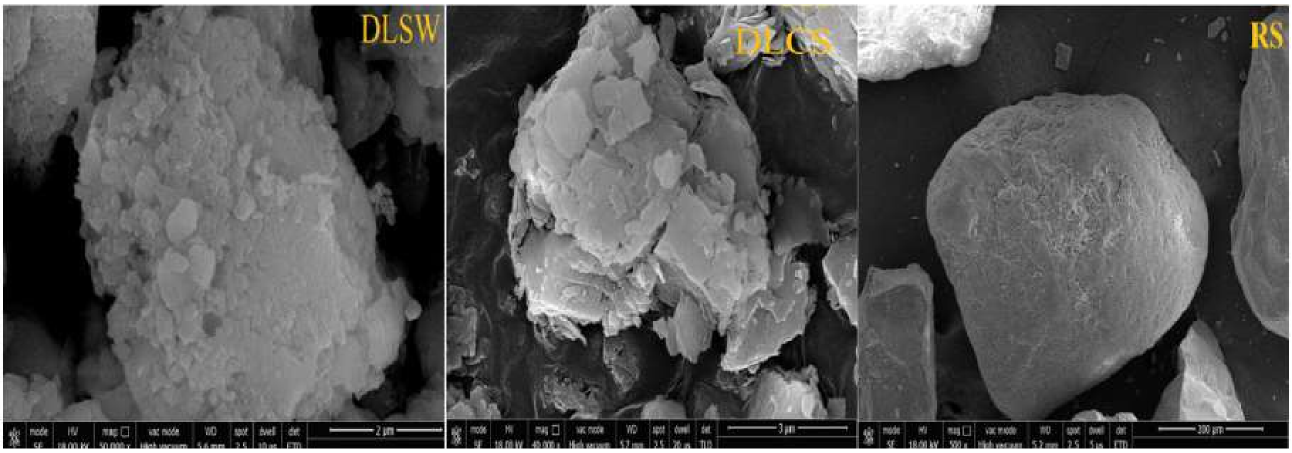

Figure 6: SEM images of limestone slurry residue (left), crushed limestone sand (center) and conventional river sand (right).

Source: Chouhan et al. (2019).

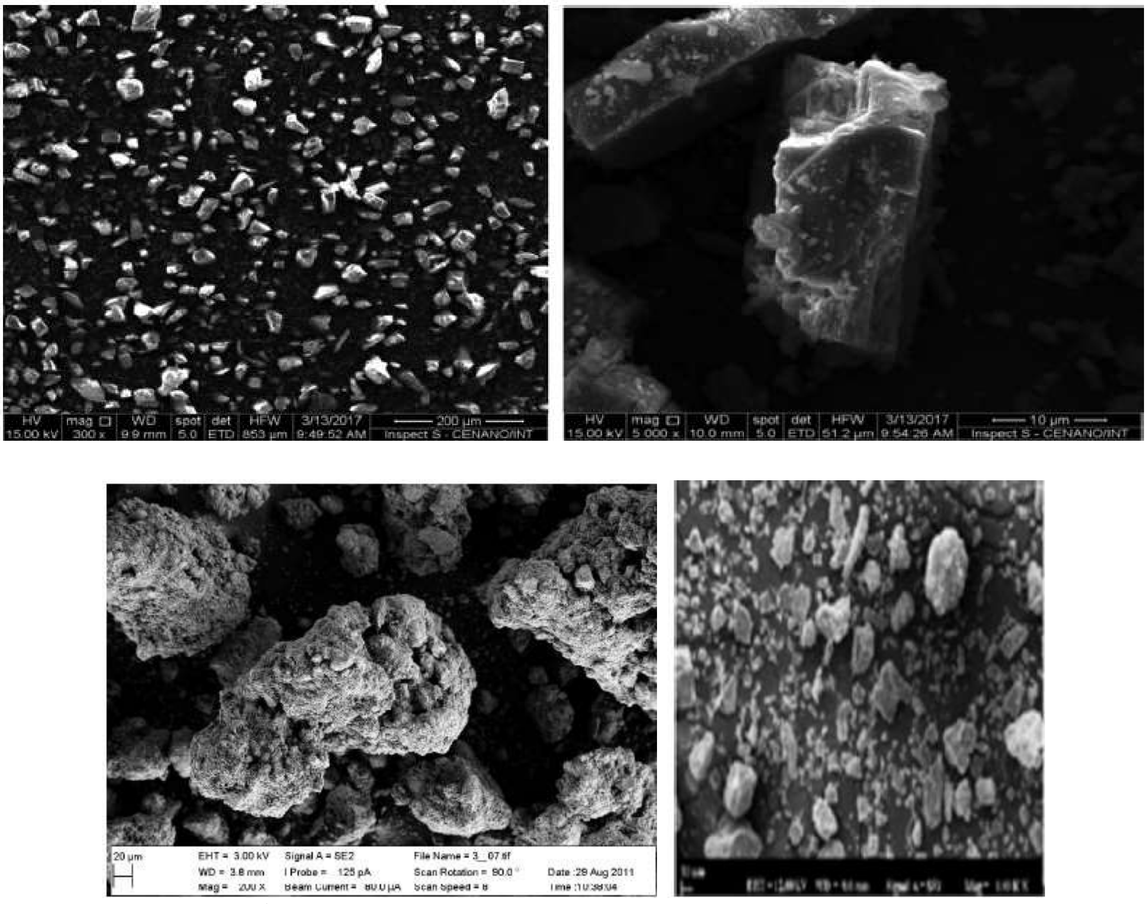

Figure 7: SEM images of limestone residues.

Source: top: Barros et al. (2020); bottom left: Hodul; Žižková; Borg (2020); bottom right: Tammam et al. (2023).

When combined, XRD and SEM can be useful for analyzing changes in the mineralogy of cementitious products after different levels of component replacement. Thus, SEM was also used to evaluate the microstructure of the products generated in the studies. Ballester et al. (2007) identified that a rounded morphology (Figure 08) in the aggregate should prevent the formation of the interconnected system that helps to bind the different components as cement hydration progresses.

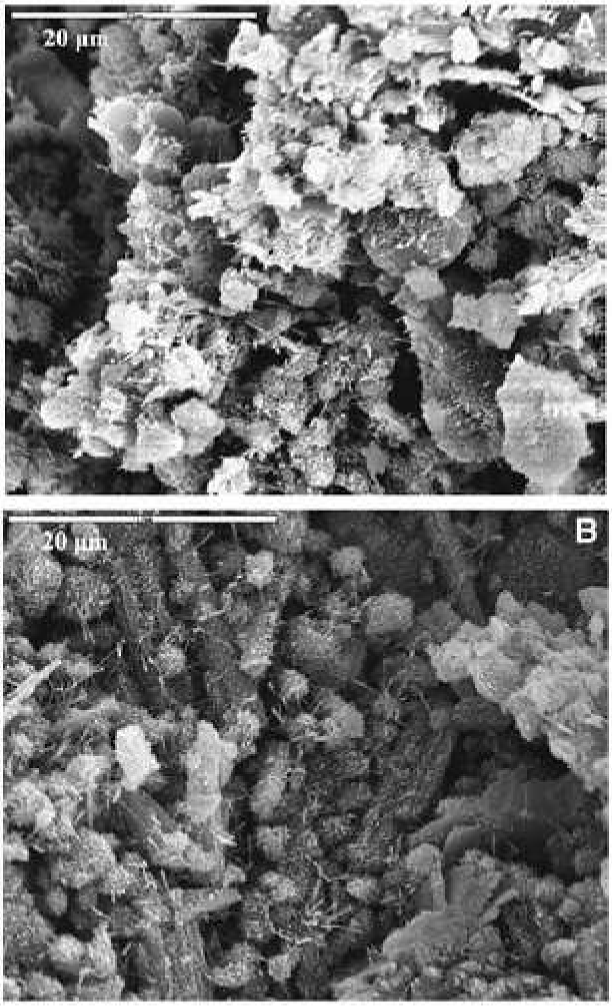

Figure 8: SEM images of limestone residues: (A) quarry limestone and (B) mussel limestone.
Source: Ballester et al. (2007).

Chouhan et al. (2020), in turn, analyzed the interfacial transition zone (ITZ), ettringite, calcium silicate hydrate (C-S-H) and the pores present in the microstructure of the mortar with dry limestone mud residues (Figure 09), concluding that as the replacement level increases, a more packed structure with dense formation of C-S-H emerges and results in increased strength. Chouhan et al. (2019) add that, for this reason, the mortar with the residue formed a denser and more compact structure and that, above 60% replacement, internal cracks can be observed.

In the case of Tammam et al. (2022) and Tammam et al. (2023), it was observed that the mortars had a compact, homogeneous structure, without cracks and a good degree of bonding with the matrix components (Figure 10). Thus, they concluded that the limestone residue particle fills the pores between the hydration products, reduces the porosity in the matrix, improves the degree of hydration of the binder and generates more hydration products.

3.3. THERMAL ANALYSIS

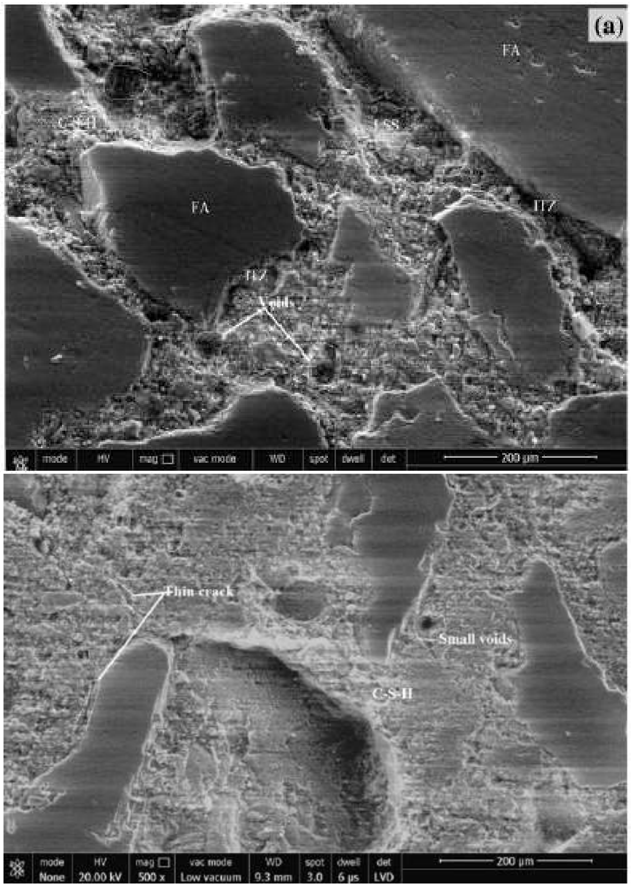

Figure 9: SEM images of the sample without residue (top) and with 40% replacement (bottom).
Source: Ballester et al. (2007).

The thermal analysis test, when used in the studies, was the Thermogravimetric Analysis (TG/TGA). Barros et al. (2020) were the only researchers who performed the analysis on the limestone residue, presenting TG and DTG curves (Figure 11), with an initial mass loss temperature around 700°C and a final one around 810°C, with a peak

https://doi.org/10.29183/2447-3073.MIX2025.v11.n1.189-203

in the DTG curve at around 783.4°C and a residual mass of 6.52%, typical of the decomposition of $CaCO_3$ into CaO releasing CO_2.

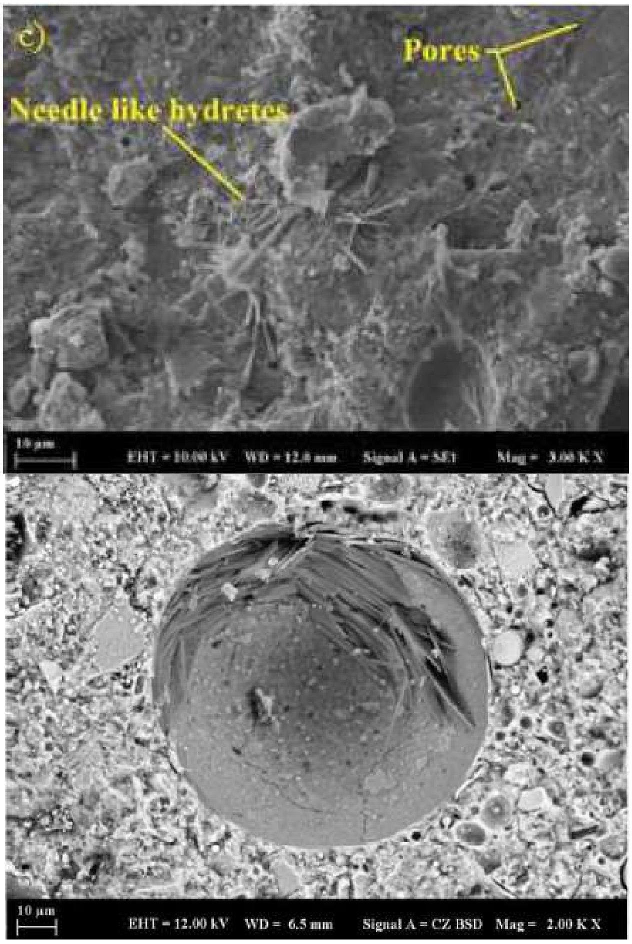

Figure 10: SEM images of mortars with limestone residue.
Source: top: Tammam; Uysal; Canpolat (2022); bottom: Tammam et al. (2023).

Other studies that used thermal analysis did so on the cementitious products produced. Ballester et al. (2007) used the technique to interpret the correlation observed between limestone aggregates and the strength of mortars, comparing the mass loss at each stage of the different curing periods and, thus, identifying the influence of the aggregate on the development of the setting process. The authors were able to identify four phases associated with different physicochemical processes: (i) weight loss due to adsorbed water (25-120°C); (ii) loss of chemically bound water (120-450°C); (iii) weight loss associated with the dehydroxylation of $Ca(OH)_2$ (450-490°C); and (iv) loss of CO_2 due to the decomposition of carbonates.

Chouhan et al. (2020) also identified four temperature ranges (Figure 12): (i) between 30 and 300°C, the curve trend showed a gradual decrease indicating moisture evaporation; (ii) between 300 and 450°C a gradual loss of 4% was observed for the control mixture and 5-6% for the mixtures with substitution attributed to the dehydroxylation of $Ca(OH)_2$; (iii) between 450 and 600°C there was a constant drop in the curve, indicating a weight loss of around 5% for the control mixture and 6-7.5% for the mixtures with substitution, which indicates the beginning of the phase transition or decomposition of calcite into CaO; (iv) between 600 and 800°C a sharp drop in the curve was observed, with a weight loss in the control curve of 6.5%, with the curves of the mixtures with substitution indicating a loss of 14.5% to 31% depending on the increase in the substitution content, justified by the decomposition of $CaCO_3$ into calcium oxide and release of CO_2.

3.4. POROSITY

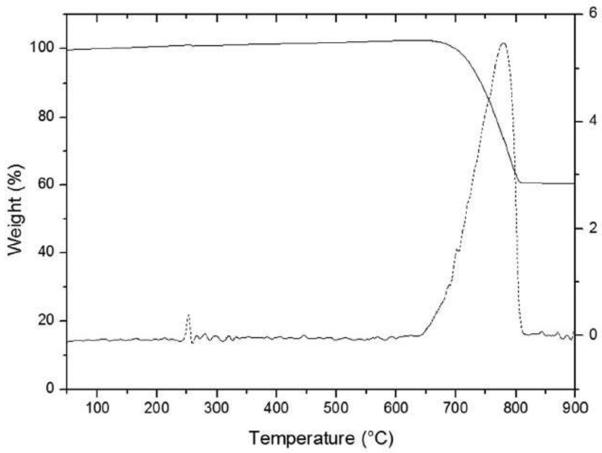

Figure 11: TG (-) and DTG (---) curves of limestone residue.
Source: Barros et al. (2020).

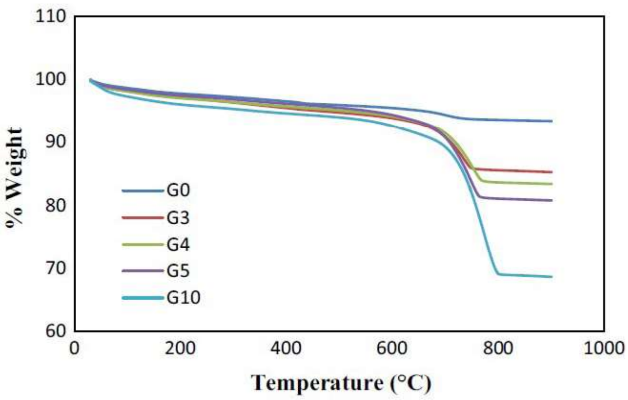

Figure 12: TGA curves for mortars with 0, 30, 40, 50 and 100% replacement of limestone residue.
Source: Chouhan et al. (2020).

199

Mercury injection porosimetry (MIP) was the technique used to analyze the porosity of the products. For Ballester et al. (2007), MIP revealed the presence of smaller pores in mortars based on prismatic particle limestone (mussel limestone), consistent with a more efficient filling of the pores and responsible for the increase in compressive strength.

On the other hand, Chouhan et al. (2020) concluded that with substitution of up to 40% (G4, in Figure 13), the results were comparable to the control sample, and, above this limit, the mortars presented high porosity, indicating a reduction in compaction efficiency due to finer particles. Figure 13 illustrates the mercury intrusion rate in relation to pore size.

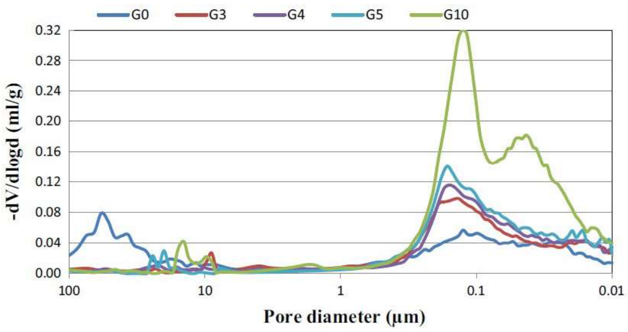

Figure 13: Mercury intrusion rate versus pore size.
Source: Chouhan et al. (2020).

4. CONCLUSION

The main objective of this study was to evaluate the usability of limestone residue in cementitious systems and, in addition, to list the main techniques that have been used to evaluate the raw materials and products generated. For this, the bibliographic review was used as a method with the Systematic Literature Review (SLR) as a tool.

It was found that limestone residue commonly originates from quarries that extract carbonate rocks. However, other sources have been explored, such as the mussel canning industry.

Quantitatively, the compositions of the limestone can be different depending on the type, source/location of generation and degree of contamination by other components. Despite the general knowledge that limestone is composed mainly of Calcium Oxide (CaO), it is possible to conclude that there is no consensus, in the studies analyzed, that the chemical and/or mineralogical analysis of the residues is presented as an indispensable preliminary analysis, since some of the studies do not perform it or do not present it. However, the limestones identified here are calcitic, which corroborates the literature that identifies that calcitic limestones as a component present better performance in rheological characteristics and mechanical properties (ISAIA; RIZZATTI, 2020).

The residue generally has an irregular shape and rough texture. Despite this, it is worth noting that there is no predominance of a complete characterization regarding the granulometry, shape and texture of the particles. The lack of this information contributes to the non-reproducibility of characterization tests, as well as not providing an enrichment of the literature on the characterization of solid waste from rocks.

Limestone waste has generally been applied in the form of a filler, partially replacing fine aggregate or cement in mortars. Even when combined with other components, studies show that the partial replacement of fine aggregate with limestone filler can improve the mechanical properties of mortars. It was noted that, as the replacement rate of fine aggregate with limestone filler residue increased, there was also an improvement in compressive, tensile and flexural strengths due to the higher limestone content increasing the reactive phase, increasing the density of the samples and reducing water absorption. That is, the beneficial characteristics are present when the residues are finely ground, with a smaller particle size than the replacement components, since they act as filler material, increasing the degree of packing in the mixtures.

Studies on limestone waste have shown that its use in mortar production is viable and beneficial and can maximize new studies on the use of waste, as well as help reduce the extraction of non-renewable natural resources. On the other hand, barriers can still be found in the use of waste, ranging from concerns about the performance of the products generated to contamination of the material due to different exposures and the lack of regulations that direct its use.

Regarding characterization techniques, XRF and XRD are used to identify the chemical and mineralogical composition of limestone residue, respectively. XRF is used only on the raw material, i.e., before application to the cement product, to identify the chemical composition. XRD, however, can be used in both phases, i.e., both for mineralogical definition and for analysis of the interaction of the residue in the product.

Regarding the physical characterization of the materials, data on density, fineness modulus and water absorption, Blaine surface area and granulometry are described, but without further details on the method/standard used. To define the granulometry, only one study indicates the laser analyzer as the equipment used, while the others do not provide details, but describe the data that indicate the use of traditional sieving as equipment.

In the case of morphological analysis of residues and cementitious products, all the research analyzed here used SEM as a technique, which can be used on raw materials, generated products, or both. SEM was mainly used to identify the shape and texture of limestone. However, confirmation of particle size distribution, as well as analysis of the interaction of materials as products, were performed thanks to the use of SEM. This reflects and justifies the power and popularity of the technique within the research of cement materials in the analysis of microstructure.

Thermal analysis, using TGA, and porosimetry, using MIP, were also techniques identified in some studies. When used on limestone, TG helped confirm the mineral due to the observed decomposition phases and, when used on mortar, allowed a better understanding of its mechanical behavior. MIP, in turn, was applied to analyze the porosity of mortars produced with limestone partially replacing fine aggregate, also helping in discussions about mechanical behavior.

Finally, regarding characterization techniques, the timid use of the FTIR technique (Fourier Transform Infrared Spectroscopy) was identified. FTIR allows the identification and analysis of chemical changes and composition variations (AZEEZ; SHENBAGARAMAN, 2025). When used only on limestone, FTIR confirmed its mineralogical origin and, when used on mortar, it served as a parameter in the analysis of mechanical behavior, thus proving to be a complementary technique.

Despite the predominance observed in detailing data on equipment and procedures, such as manufacturer, parameters, software, sample preparation, among others, some studies did not do so, which can make comparisons between results from different studies difficult, as well as reproducibility in new research. In addition, most studies do not describe the state of the waste at the collection site (sludge, pieces of rock or sawdust powder, for example), nor do they address the form of waste preparation, such as drying, transportation and comminution.

In future, it is suggested that investigations be carried out into the other components (aggregates, additives, additions, etc.) of the mixtures that may also influence the results obtained, since the studies analyzed here use different compositions in the mortar mixes produced. In addition, a new analysis of the characterization of the limestone residue used as filler can be carried out, finding it in literature as limestone powder or dust, which generally come from industries that process and commercialize it.

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HOW TO CITE THIS ARTICLE:

BORGES, A. V. G; CIRINO, M. A. G.; DAMINELI, B. L.; CABRAL, A. E. B. Analysis of the application of limestone WASTE in cementitious systems based on a systematic literature review. **MIX Sustentável**, v.11, n.1, p.189-203. ISSN 2447-3073. Disponível em: <http://www.nexos.ufsc.br/index.php/mixsustentavel>. Acesso em: _/_/_.

SUBMITTED ON: 22/10/2024
ACCEPTED ON: 04/04/2025
PUBLISHED ON: 16/06/2025
RESPONSIBLE EDITORS: Lisiane Ilha Librelotto e Paulo Cesar Machado Ferroli

Record of authorship contribution:
CRediT Taxonomy (http://credit.niso.org/)

AVGB: Conceptualization, data curation, formal analysis, investigation, methodology, writing – original draft, and writing – review & editing.

MACG: Conceptualization, formal analysis, investigation, visualization and writing – original draft.

BLD: Project administration, supervision and writing – review & editing.

AEBC: Supervision and writing – review & editing.

Conflict declaration: nothing to declare.