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Utilization of Stone Wool Kiln Ash in Cement-Based Materials †

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Abstract: The main goal of this study was to validate a circular production route utilizing the waste ash generated during stone wool production in cementitious binders. To achieve this goal, two types of waste ash with different compositions and particle sizes were used. The performance evaluation results showed that silica-based ashes exhibit pozzolanic behavior, reduce the amount of portlandite, and can improve the strength of the mortar even at early ages. The ashes increased the initial setting time regardless of their composition. The outcomes of this study create an economic value for large volumes of material of previously zero value.

Keywords: stone wool ash; circular economy; cement-based mortar; performance

1. Introduction

For the last decade, there has been increasing awareness of the urgent need to find solutions to initiate climate mitigation action leading to a more sustainable future. While the cement and concrete industry plays a vital role in the European economy, their production process significantly contributes to global warming and is responsible for 8% of CO_2 emissions [1,2]. While the chemical capture of CO_2 seems to be the only feasible approach, high-volume cement production creates technical and economic challenges. At this time, the pressure to reduce CO_2 emissions has only succeeded in reducing the demand for cement.

Our previous efforts in revalorizing the End-of-Life (EOL) of cementitious systems enabled us to develop a comprehensive understanding of the activation processes in EOL and utilize them as Supplementary Cementitious Materials (SCMs), substantially lowering the environmental impact of the construction industry [1,2]. However, the feasibility of regenerated EOL in cementitious systems is still open to discussion due to its effect on strength and durability. Another possible use of EOL is geopolymer binder systems, where reactive EOL, such as fly ash and slag, is activated through chemical synthesis [3]. While there is extensive knowledge on utilizing EOL in geopolymers, it is still a major challenge to regenerate the waste materials in cementitious systems.

Fulfilling the ambition of maintaining global carbon neutrality, utilizing alternative zero-carbon resources is becoming more essential in the design of building materials. Efforts to develop alternative supplementary cementitious materials are becoming popular due to their low environmental impact and ability to reduce CO_2 emissions [4–6]. The mineral wool waste collected from construction debris could be valorized as a potential pozzolanic material for Portland cement concrete. However, completely circular production can only be obtained through a "zero-waste" production route. The ashes collected from the cokefired hot blast kiln are a significant portion of the waste stream generated from mineral wool production. To achieve a sustainable circular production for mineral wools,

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valorizing the waste stream obtained during production is becoming more essential for the producer.

The main goal of this study is to validate a circular production route utilizing the waste ash generated during mineral (stone) wool production in cementitious binders. The performance evaluation was performed on cement-based samples where 20% of the cement was replaced with waste ash. The results showed that W20 exhibits a pozzolanic behavior and can improve the strength of the mortar even at early ages, while W10 does not contribute to strength and acts as a filler material. Both ashes increased the initial setting time and reduced the workability of the mortar. The outcome of this study creates an economic value for large volumes of material of previously zero value while proposing a new cementitious composite product for mineral wool producers.

2. Materials and Methods

2.1. Material Selection and Characterization

The samples were prepared with Ordinary Portland Cement (OPC) CEM I 42.5 R, and two types of waste ash with different compositions and particle sizes were used to achieve this goal. The ashes were classified as W10, calcite-based ash with an average particle size of 75 microns, and W20, silicate-based ash with an average particle size of 22 microns. Table 1 summarizes the chemical composition of the binders used in the study.

Table 1. Composition of the binders, % weight in the composition.

Binder	SiO_2	Al_2O_3	CaO	$CaCO_3$	Fe_2O_3	MgO	SO_3	K_2O
Cement	10.70	2.65	69.90	5.00	2.35	0.71	3.37	0.70
W10	23.60	6.86	-	48.00	4.13	4.00	5.15	2.65
W20	46.40	1.96	-	8.54	2.82	7.53	4.26	13.60

Standard sand, according to the EN 196-1 norm, was used in the mortar mixes. A polycarboxylate ether (PCE) superplasticizer (SP) was used when it became necessary to maintain the same workability of all samples. Table 2 summarizes the weight of the ingredients in the 3 different mixes.

Table 2. Material proportions of blended cement-based mortars. W10 and W 20: Stone wool kiln ash.

	Cement (g)	W10 (g)	W20 (g)	Water (g)	Standard Sand (g)
Mix#1	600	-	-	300	1800
Mix#2	480	-	120	300	1800
Mix#3	180	120	-	300	1800

2.2. Compressive Strength

The mortar samples were prepared according to ASTM C305-14 norms [7]. The water-to-cement ratio (w/c) and the sand-to-cement ratio were kept at 0.5 and 3, respectively. To calculate the strength activity index based on ASTM C311 [8], 20% of the binder was replaced by kiln ashes (Table 2). The mortar samples were cast in 50 × 50 × 50 mm cm cubes and kept in a humid environment at 21 °C for 24 h. Upon removal of the molds, the samples were cured in a moist environment at ambient conditions until testing. Compressive strength testing was conducted according to the ASTM C109-13e1 standard at 3, 7, and 28 days on triplicate samples [9].

2.3. Vicat Needle Test

A modified Vicat Needle test was conducted to determine the setting time of the cement pastes. The cement paste samples were prepared using a modified ASTM C191-13

Standard [10]. Instead of determining the w/c ratio that would yield a "normal consistency" paste, as suggested in the standard, the w/c ratio was kept constant at 0.50 to be consistent with the compressive strength test. The initial setting of the cement paste samples was determined according to the penetration depth of the Vicat needle. Analyses were conducted based on triplicate samples.

2.4. Thermogravimetric Analysis (TGA) of Cement Pastes

TGA was performed with cement paste samples prepared with a w/c of 0.50. Following the mixing, samples were cast into prismatic molds. The specimens were initially cured at 100% relative humidity at 21 °C for 24 h. Upon removal of the molds, the samples were cured in a moist environment at ambient conditions until testing. TGA testing was conducted on the samples at 7 and 28 days. The samples were tested with TGA-DTA Analyzer (Perkin Elmer, Shelton, CT, USA). The analysis was conducted by heating the samples from 40 °C to 1100 °C, and mass loss was recorded as a function of time. The decomposition of calcium hydroxide (portlandite) was determined between 450 and 550 °C, and calcium carbonate ($CaCO_3$) decarbonization was measured between 700 °C and 900 °C [11].

3. Results and Discussion

3.1. Pozzolanicity of Stone Wool Kiln Ash and Its Impact on the Compressive Strength

Figure 1 displays the results of the compressive strength and strength activity index (SAI) tests. Based on the findings, W20 possesses pozzolanic activity, while W10 does not contribute to compressive strength development. The pozzolanic activity of the stone wool kiln ash was assessed based on the ASTM C 311 [8] and C 618 [12] standards. ASTM C618 specifies that the activity of mortars containing pozzolans at 7 and 28 days must not fall below 75%, represented by the line labeled as minimum. The evaluation of SAI also revealed that W20 exhibits pozzolanic activity while W10 does not. This was attributed to the different chemical and physical properties of these two different ashes.

W20 is silicate-based ash with an average particle size of 22 microns. Even though W10 has 24% SiO_2 in its composition, the coarser particle size decreases its reactivity. W10 acts as a filler instead of a binder. At last, the influence of W20 was more pronounced after 7 days of mixing.

3.2. Impact on Fresh State Properties: Initial Setting Time

Figure 2 represents the modified Vicat needle test results. The different initial setting times were evaluated by comparing them with the reference Mix #1 samples with an initial setting time of approximately 6 h. Incorporating both stone wool kiln ashes significantly increased the initial setting time. Replacing 20% of the binder with W10 and W20 resulted in approximately 10.5 h of setting time. This indicates that neither of these ashes possesses hydraulic activity. Based on the data obtained from the strength and setting time tests, it is evident that W20 is a silicate-based pozzolan resembling the behavior of F-type fly ash and that W10 is a calcite-based filler like limestone. Further, mix design optimization should be performed considering the characteristics of these waste materials.

3.3. Impact on Composition: Calcium Carbonate and Calcium Hydroxide Content

Table 3 summarizes the mass percentages of the portlandite and $CaCO_3$ calculated via TGA. Based on the data obtained, it is evident that the incorporation of W20 in the mix decreased the amount of portlandite even at seven days. The results align with the compressive strength results, showing that W20 acts as a pozzolanic material, reacts with portlandite even as early as seven days after mixing, and starts improving strength.

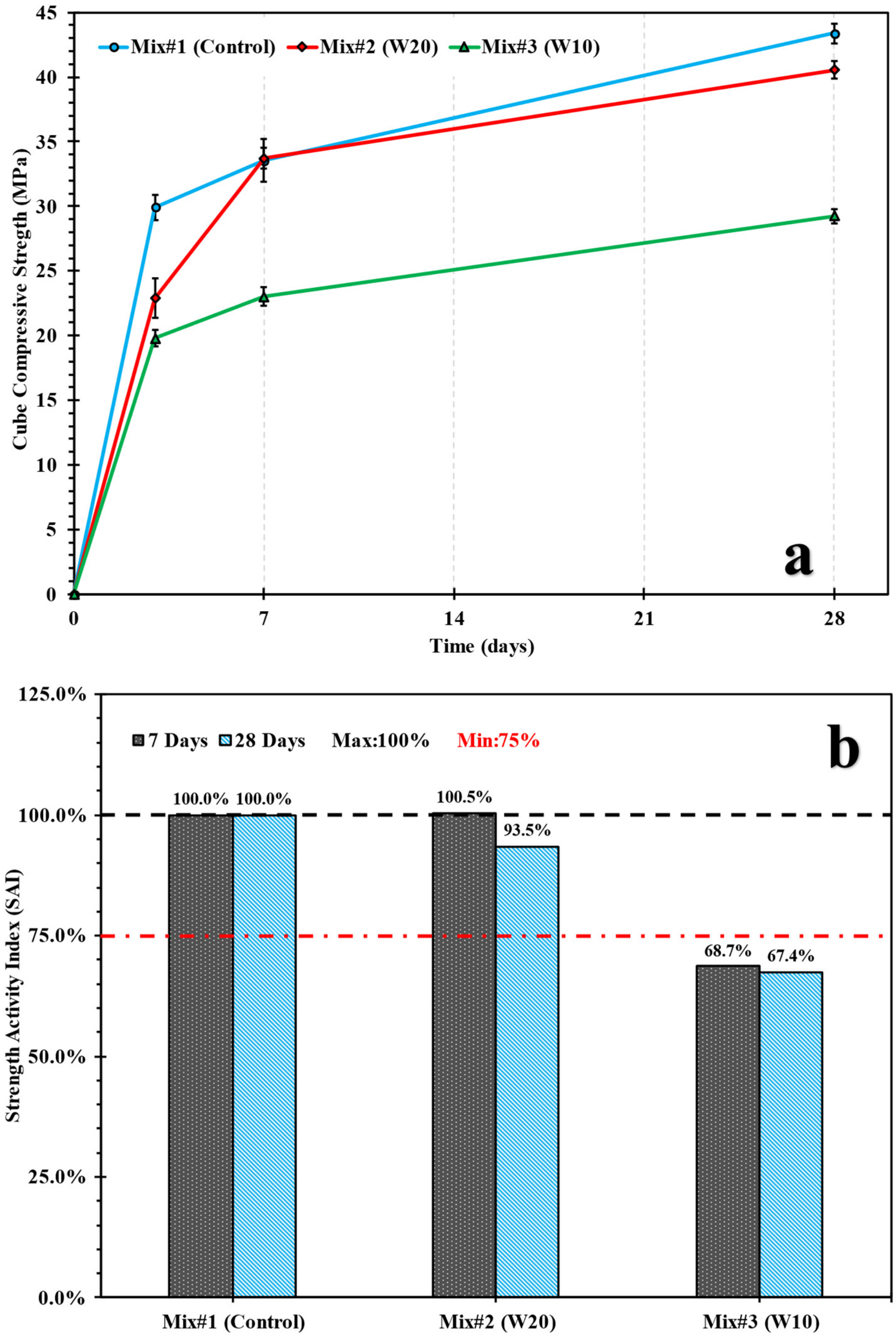

Figure 1. Influence of stone wool kiln ash on compressive strength. (**a**) Average compressive strength at 3, 7, and 28 days. (**b**) Strength Activity Index.

Table 3. Portlandite and $CaCO_3$ content in cement paste samples.

	7-Days		28-Days	
	Portlandite (%)	**$CaCO_3$(%)**	**Portlandite (%)**	**$CaCO_3$(%)**
Mix#1	6.5	2.3	7.0	2.7
Mix#2	5.3	3.2	5.0	2.7
Mix#3	6.1	4.1	5.3	4.1

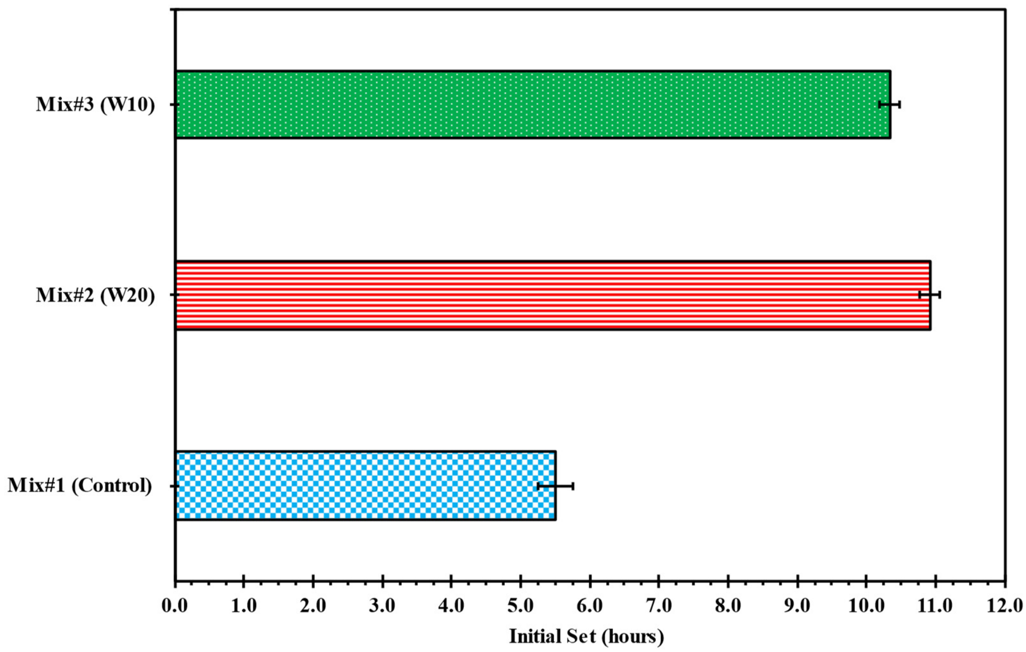

Figure 2. Modified ASTM C191-13 initial setting times of different cement paste mixes. The bars represent the average initial setting time values based on triplicate cement paste samples, and the error bars represent the one standard deviation.

Further evaluation has to be performed to understand the material's performance at later ages (i.e., one year). Similarly, Mix #3, with a 20% W10 replacement in the binder, resulted in a $CaCO_3$ content due to the composition of W10. While W10 is unsuitable for use as a binder replacement, it is still possible to utilize the waste material as a filler in cement-based materials.

4. Conclusions

This study was undertaken to revalorize stone wool kiln ash as a pozzolan in cement-based materials. To achieve this goal, two types of waste ash with different compositions and particle sizes were used. The ashes were classified as W10, calcite-based ash with an average particle size of 75 microns, and W20, silicate-based ash with an average particle size of 22 microns. The results showed that W20, with its higher silicate content and finer particle size, could exhibit pozzolanic behavior and react with portlandite. The pozzolanic tendency of W20 resulted in an increase in compressive strength. In contrast, W10, with its coarser particle size and low alumina silicate content, possessed no reactivity and acted as a filler in the system. Both ashes resulted in a longer setting time compared to the control sample. The outcomes will contribute to the circular economy by proposing a circular production method for the stone wool industry to utilize their waste stream and RM to design building elements for practical applications. The outcomes of this project will provide alternative solutions to climate change and the efficient use of local raw materials for alternative building materials.

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