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Strength Investigation of Slag-Based Geopolymer Composites Incorporating Different Amounts of Colemanite Waste and Silica Fume Under Different Exposure Conditions

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Abstract

In this study, it is aimed to investigate the strength performance of slag-based geopolymer mortar with different percentages of silica fume and colemanite waste by mixing Na_2SiO_3 and NaOH as the alkaline activator for the geopolymerization reaction, and cured at room temperature were prepared, in terms of compressive strength, flexural strength, ultrasonic pulse velocity and freeze-thaw resistance parameters. Five different mixtures were prepared by using different amounts of silica fume and colemanite waste by using the same amount of ground granulated blast furnace slag, sand and 8M sodium hydroxide for these five mixtures. The mixture, including a paste proportion of 20% slag, 40% colemanite waste, and 40% silica fume, was used as a control mix. The maximum compressive strength (21.24 MPa, 38.32 MPa) flexural strength (5.86 MPa, 6.98), weight loss caused by freeze-thaw effect (0.56%) and ultrasonic pulse wave test (3082 m/s) results were noted as for 7th and 28th day, respectively. After -60 cycles [1 cycle consists of (-18 ºC) for 90 minutes and (+4 ºC) for 30 minutes], the maximum compressive and flexural strength was observed as (40.18 MPa and 4.92 MPa, respectively). The results indicated that the strength results were consistently increased as silica fume increased. The addition of a certain amount of silica fume gave promising results both in terms of the strength and durability aspects. Overall, according to this experimental study, the utilization of 30% colemanite waste and 50% silica fume can be recommended so as to balance both sustainability and engineering aspects.

Keywords: Compressive strength, Geopolymer, Freeze-thaw, Colemanite waste, Silica fume

Farklı Miktarlarda Kolemanit Atığı ve Silika Dumanı İçeren Cüruf Esaslı Geopolimer Kompozitlerin Farklı Maruz Kalma Koşullarında Dayanımlarının İncelenmesi

Öz

Bu çalışmada, Geopolimerizasyon reaksiyonu için alkali aktivatör olarak Na_2SiO_3 ve NaOH karıştırılarak oda sıcaklığında kürlenen, farklı yüzdelerde silis dumanı ve kolemanit atığı içeren cüruf bazlı geopolimer

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harcının mukavemet performansının araştırılması amaçlanmaktadır. Basınç dayanımı, eğilme dayanımı, ultrasonik hızı ve donma-çözülme direnci parametreleri açısından. Bu beş karışım için aynı miktarda öğütülmüş granül yüksek fırın cürufu, kum ve 8M sodyum hidroksit kullanılarak farklı miktarlarda silis dumanı ve kolemanit atığı kullanılarak beş farklı karışım hazırlanmıştır. Macun oranında %20 cüruf, %40 kolemanit atığı ve %40 silis dumanı içeren karışım, kontrol karışımı olarak kullanıldı. Maksimum basınç dayanımı (21,24 MPa, 38,32 MPa), eğilme dayanımı (5,86 MPa, 6,98), donma-çözülme etkisinden kaynaklanan ağırlık kaybı (%0,56) ve ultrasonik hız testi (3082 m/s) sonuçları ise 7. ve 7. sıralarda kaydedildi. Sırasıyla 28. gün. -60 döngüden sonra [1 döngü 90 dakika süreyle (-18 °C) ve 30 dakika süreyle (+4 °C) oluşur] maksimum basınç ve eğilme dayanımı (sırasıyla 40,18 MPa ve 4,92 MPa) olarak gözlemlendi. Sonuçlar, silika dumanı arttıkça mukavemet sonuçlarının tutarlı bir şekilde arttığını gösterdi. Belli bir miktar silis dumanının ilavesi hem mukavemet hem de dayanıklılık açısından ümit verici sonuçlar vermiştir. Genel olarak, bu deneysel çalışmaya göre, hem sürdürülebilirlik hem de mühendislik yönlerini dengelemek amacıyla %30 kolemanit atığı ve %50 silis dumanı kullanımı önerilebilir.

Anahtar Kelimeler: Basınç dayanımı, Geopolimer, Donma-çözülme, Kolemanit atığı, Silis dumanı

1. INTRODUCTION

The modern construction industry majorly relies on ordinary Portland cement to develop every infrastructure. Portland cement is highly criticized for its massive amount of poisonous substances, hazarding natural ecosystems, and its contribution to global warming. Carbon dioxide (CO2) is one of the significant substances in this worry; CO2 emissions from the cement industry only constitute nearly 6%–7% of total CO2 outflows around the globe [1]. Another concern is that the cement sector is considered unsustainable and energy-intensive [2].

Ordinary Portland Cement consists of calcium carbonate (limestone) calcination at very high temperatures of about 1450-1500 °C, and silico-aluminous material. This means that the production of every 1 metric tonne of cement generates 1 metric tonne of CO2 [3]. As a result of this, the more consumption of cement, the more contribution to the devastation of the ecosystem and atmosphere. The cement industry is one of the biggest causes of atmospheric pollution amongst the industries.

As a result of this situation, the current trend is that cement production and utilization to be transformed to a more sustainable and eco-friendly in practice. In this regard, to mitigate the negative impacts of clinker production in conventional construction practices, several scholars made a significant effort to minimize the environmental footprint of conventional cement by introducing eco-friendly alternative materials, such as geopolymer, in the current construction sector [4].

The main benefit of geopolymer cement is that it reduces carbon dioxide emissions since the chemical process emits zero carbon dioxide, consuming fuel much less. As a result of this, carbon dioxide emissions are decreased by 80%–90%. It also contributes to the reuse of industrial wastes (fly ash, ground granulated blast furnace slag, silica fume, etc.) and diminishes global warming.

Nowadays, fly ash, metakaolin, ground granulated blast furnace slag (GGBS), silica fume (SF), and others are regarded as the most widely utilized pozzolanic materials in the cement industry [5]. Generally, these materials are large enough to combine with Portland cement or (with) each other forming a sustainable binder to produce a mortar and concrete. GGBS, which is classified among supplementary cementitious materials utilized in geopolymer development, primarily constitutes lime, silica, alumina, and magnesia [6]. Studies showed that the combination of slag with low calcium pozzolanas could significantly improve the setting and hardening time of the geopolymer matrix at standard conditions. Apart from this, it has a valuable impact on the resulting matrix's short and long-term strength parameters. Susan et. al. [7] stated that the availability of calcium compounds, which are dominantly found in slag, speeds up the geopolymerization process. Davidovits [8], in his study, demonstrated that the combination of slag with metakaolin produces composites possessing excellent engineering and durability properties.

Rostami and Behfarnia [9] examined the influence of replacing slag with SF on compressive performance and permeability of alkali-activated slag under water-curing conditions. The outcomes indicated that the inclusion of SF has enhanced the compressive strength and reduced the permeability of the resulting concrete matrix. As a result, the existence of SF positively contributes to the mechanical and durability characteristics of alkali-activated slag.

Francis et. al. [10] analyzed the effect of SF on the durability aspect of fly ash-based geopolymer concrete. The results showed that the specimens containing SF were found to be highly resistant to the harsh (sulfate and chloride attack) environment compared to the samples without SF. Also, Okoye et. al. [11] revealed the direct relationship between compressive strength and SF content in the geopolymer concrete. The possible reasons for improving mechanical strength and durability characteristics of geopolymer concrete incorporating silica fume could be attributed to enhanced geo-polymerization, reduction in pore diameter, and permeability of acid solution in the concrete matrix.

Boron is among Turkey's most widely available resources, estimated at around 60% of the global boron reserves. Colemanite, tincal, and ulexite are widely marketed forms of boron ores in the country [12]. The term "colemanite" is used when the basic form of boron compounds (B2O3 and H2BO3) is found with calcium, whereas with calcium-sodium, it is named "ulexite" [13]. Several studies have examined the effect of boron waste inclusion on the different properties of mortar and concrete matrices. Abali et. al. [14] investigated the effect of tincal waste in combination with other materials such as fly ash, volcanic tuff, and bentonite. The results showed that the compressive strength of the specimens reduced with the increase of tincal waste content. Also, Elbeyli [15] has reported the same result whilst hiring borax wastes.

On the other hand, Samir Ushah El-Kameesy et. al. [16] developed material as nuclear radiation protection using boron and rubber. The boron addition has improved the shielding effect of the resulting matrix. It is essential to consider that the concrete hired in the nuclear application must also possess desirable structural and mechanical properties. Therefore, the utilization of CW in the cement industry has dual advantages in terms of environmental protection and engineering application. However, only a limited number of studies have addressed the effect of this material on geopolymeric products.

SF is a material consisting of very fine spherical particles, containing a high percentage of amorphous silica; it is obtained from the decomposition of high purity quartz containing coal in electric arc furnaces in the production of silicon and ferrosilicon alloys.

SF displays an excellent pozzolanic material feature because of its high amount of SiO2 content and its very fine amorphous structure. It is mostly a grey coloured nano-sized powder. SF grains fill the spaces between large cement granules, enhancing granulometry and generating a denser structure.

SF is known that strengthen the bonding at the aggregate interface of the cement matrix, fill the spaces in the concrete microstructure, create a more composite and denser structure with high composite, increase the compressive and tensile strengths, prevent the formation of bleeding and plastic shrinkage, reduces permeability, strengthens the concrete structure against harmful chemicals.

In this experimental study, the influence of SF and CW on the various properties of slag-based geopolymer composites was investigated. The properties of the resulting matrix were assessed using compressive strength, flexural strength, freeze-thaw, and the magnitude of ultrasonic pulse velocity (UPV) results of the composites.

Similarly, the previous studies in the literature increasing silica fume affects geopolymer positively. So, this study supports the other studies in this regard.

2. MATERIALS AND METHODS

2.1. Materials

GGBS was obtained from Bolu Cement Company situated in Turkey, and colemanite was provided from Eti Mine Bigadic Boron Works located in Turkey. The specific gravities of GGBS and CW were evaluated as 2.91 g/cm3 and 2.42 g/cm3, respectively. The chemical composition of GGBS was SiO2: 41%, CaO: 36%, Al2O3: 13%, and MgO: 5.8%. These oxides only constitute approximately 95% of the total makeup of the GGBS used. The total of remaining compounds such as Fe2O3, TiO2, K2O, Na2O, and loss on ignition contributes only 5%. In the case of CW, B2O3, LOI, CaO, and SiO2 hold around 97% of the total composition, i.e. 40, 25, 27, and 5%, respectively. The oxides such as MgO, Fe2O3, Al2O3, and Na2O share only the aggregate proportion of 3%. The SF used in this study contains more than 91% of SiO2. The proportion of MgO, K2O, LOI, Al2O3, Fe2O3, CaO, and Na2O were 4.1%, 2.6%, 1.7%, 0.38%, 0.15%, 0.32%, and 0.55%, respectively. SF was collected from Eti Antalya Electrometallurgy Inc. (Kepez-Antalya/Turkey), having a specific gravity of 2.20 g/cm3.

In the study, Rilem sand complying with the BS EN 196-1 [17] and Rilem Cembureau standards were utilized. The NaOH with the concentration of 8M and Na2O3 with a molar ratio (SiO2/Na2O) of 3.29 was used as alkaline activators.

2.2. Experimental Procedure

The details of mixtures experimented in this study is given in Table 1 and Figure 2, Respectively. Due to the lack of standard codes to prepare geopolymer mortar, previous works of literature are used as a code of reference in the mixing process. The incorporation of SF and CW was considered a partial substitution with GGBS to prepare five mix designs as listed in Table 1. For all mixes, the GGBS ratio was adjusted to 20% of the total binder content. However, the different proportions of CW (Figure 1a) and SF (Figure 1b) were added to each independent mix design to determine the most appropriate proportion of the variable ingredients.
In the beginning, SF and CW were mixed with alkaline solutions, and then 160 g GGBS was added to the mixing pan. Afterward, the prepared paste was mixed with sand to produce geopolymer mortar. The mortar was cast into oiled moulds with lubricating oil, and cured at room temperature until an intended date of the tests.

A compressive strength test was conducted after the 7th and 28th days using the (50×50×50) mm cubes; the procedure was done according to ASTM C 109 [18]. A flexural strength test was performed on 40×40×160 mm prismatic specimens after 7 and 28 days of curing period according to ASTM C 348 [19] standard procedure. Each series of mixes were checked for freeze-thaw cycles; they have been exposed to freeze-thaw cycles for a total of 60. The freezing and thawing period was done per cycle, with the temperature fluctuating between –18 °C for 90 minutes to 4 °C for 30 minutes. Also, ultrasonic pulse velocity tests were conducted in order to inspect the homogeneity of the manufactures matrix, the existence of possible cracks and defects. In this investigation, ultrasonic pulse velocity test was carried out for the manufactured samples to evaluate the performance of the matrix with the studied parameters and the investigated categories. After freezing-thaw exposure, the specimens were removed from the testing machine. The freezing-thaw effect on mortar specimens was investigated, and compressive strength, flexural strength, UPV, and weight loss were noted.

Table 1. Details of mixtures used

Mix ID	SF (gr)	CW (gr)	GGBS (gr)	SAND (gr)	SS (gr)	SH (8 M)
20S40C40SF (Control)	320	320	160	2400	270	90
20S45C35SF	280	360	160	2400	270	90
20S35C45SF	360	280	160	2400	270	90
20S50C30SF	280	400	160	2400	270	90
20S30C50SF	400	280	160	2400	270	90

*SF: silica fume, CW: colemanite waste, GGBS: ground granulated blast furnace slag, SAND: Rilem sand, SS: sodium silicate, SH: sodium hydroxide.

Figure 1. a) Colemanite waste sample, **b)** Silica fume sample

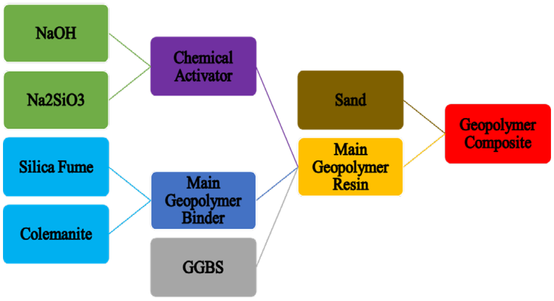

Figure 2. Mixing process in the conducted study

3. RESULTS AND DISCUSSIONS

3.1. Compressive Strength

As illustrated in Figure 3, the 7^{th} and 28^{th} days comparison of the compressive strength results of the geopolymer mortars produced with the partial replacement of SF and CW is seen.

Overall, all series of the mixtures gained slightly more than 50% of their 28 days compressive strength on the 7^{th} day. The compressive strength of the mortar samples increased with the increase of the SF ratio.

The 28-day compressive strength of the mortar mixture containing an equal proportion of SF and CW (20S40C40SF) stood at 33.31 MPa, around 47% higher than its 7-day compressive strength. This result dropped by around 4.77% with the application of 45% CW and 35% SF. However, the compressive strength of 20S35C45SF (35% CW and 45% SF) rose to 36.27 MPa, increased by approximately 9% compared to specimens produced using an equal share of CW and SF.
For the following mixture forms 20S50C30SF and 20S30C50SF, the 28-day compressive strength values were found as 28.65 MPa and 38.32 MPa, respectively. The maximum strength value was monitored in the mixture 20S30C50SF among the whole five mixtures. In contrast to this, the addition of 50% CW and 30% SF recorded the lowest strength result in comparison to the rest. However, the value obtained was still acceptable for use in the practical construction sector.

The further substitution of CW may negatively affect the compressive strength of the resulting specimen. As a result, the partial replacement of CW should be limited to a certain amount. According to this study, CW addition of up to 35% of the total binder ratio resulted in a better compressive strength. On the other hand, adding SF content up to 50% (maximum SF used in this study) positively contributed to the strength development throughout the mixes produced. These results are consistent with the studies conducted by Mucteba et. al. [20] and Ertugrul Erdogmus [21].

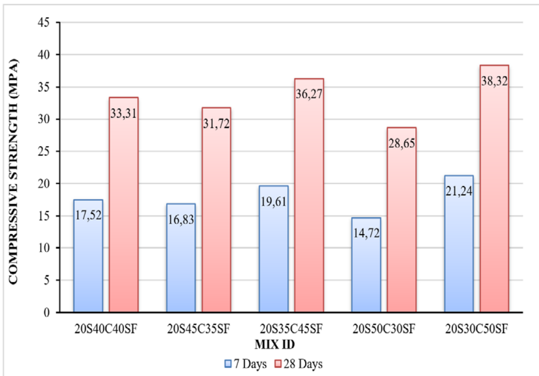

Figure 3. Compressive strength results of the mixtures at the 7^{th} and 28^{th} days

3.2. Flexural Strength

As illustrated in Figure 4, the 7-day and 28-day flexural strength results followed the same pattern as the compressive strength results.

In the beginning, the 7-day and 28-day flexural strength results of the mortar specimens prepared by adding an equal amount of SF and CW into slag (20S40C40SF) were recorded at 4.74 and 6.26 MPa, respectively. These values reduced by around 22.6% and 23% as the proportion of CW increased to 45% and SF decreased to 35% of the total paste content, where the slag ratio remained constant. In the third series of the mixture (20S35C45SF), where SF was 45%, and CW 35%, the 7-day and 28-day flexural strength rose to 5.63 and 6.58 MPa, respectively, which is approximately 18.8 and 5.1% higher than the flexural strength of specimens manufactured from a one-to-one ratio of SF and CW (20S40C40SF). According to this experimental study, the mortar specimens containing the highest percentage (50% of the total paste content) of CW reached the maximum reduction in flexural strength. However, the samples with the higher SF content (50%) gave the highest strength results.

Generally, the flexural strength rose consistently as the proportion of SF increased. In contrast, the strength decreased continuously as the percentage substitution of SF with CW increased. It was also observed that the specimens with a higher amount of SF could achieve more than 83% of their 28 days flexural strength in 7 days.

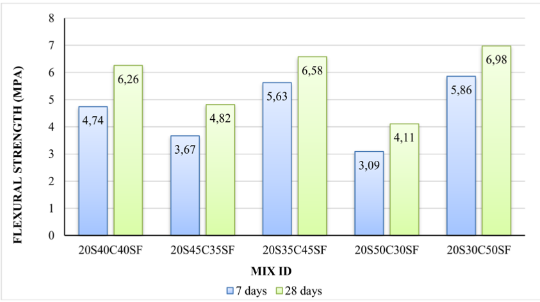

Figure 4. Flexural strength results at the 7th and the 28th days

3.3. Ultrasonic Pulse Velocity (UPV)

UPV test is a non-destructive test used to assess the homogeneity of the matrix and the dynamics in the structure of the mortar or concrete products. An ultrasonic pulse velocity test was carried out in this study, and the results are displayed in Figure 5. According to Mouhcine et al. [22], the UPV test is illustrated as an appropriate method to determine qualitative properties of the concrete, such as the existence of defects, uniformity, and homogeneity of the matrix.

As observed from Figure 5, the increase in silica fume content caused a continuous rise in ultrasonic pulse velocity. For instance, the velocity of a mortar sample prepared from 50% SF content gave 2964 m/s, which is around 5.48% and 8.3% higher than the samples produced by adding 40% and 35% SF content, respectively. On the other hand, the results of the specimens with a higher amount of CW experienced a similar trend to that of compressive and flexural strength results. The samples with 50% CW resulted in the lowest ultrasonic pulse velocity, whereas those with 35% CW gave the highest velocity.

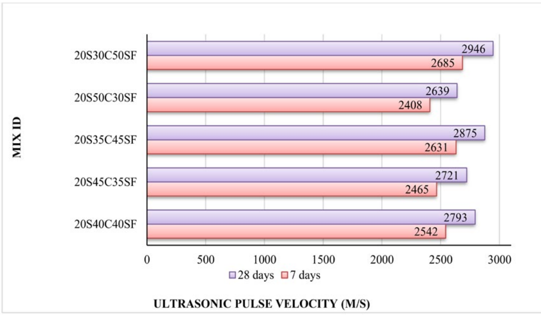

Figure 5. Ultrasonic pulse velocity test results at the 7th and 28th days

3.4. Freeze-Thaw Resistance

The impact of freezing-thawing on the geopolymer mortar mixtures was investigated in terms of Compressive strength, flexural strength, weight loss, and UPV. In Table 2, the quantitative details of all the parameters (compressive strength, flexural strength, UPV and weight loss) were given.

As shown in Table 2, both compressive strength and UPV results arose after being subjected to freeze-thaw cycles. On top of these, the specimen's weight was also increased after exposure to the freezing-thaw cycle. This can be attributed to the development of dense and compact structures due to exposure to iterative freeze and thaw cycles. Therefore, water could not penetrate the mortar matrix easily. Also, it can be justified as the continuation of the geopolymerization process due to the surrounding environment, which enhanced the geopolymer matrix's microstructural characteristics. This result is in line with the study by Yunsheng et. al. [23]. On the other hand, the flexural strength of mortar samples saw a reduction after being subjected to freeze-thaw cycles. Based on the experimental results and analysis, the addition of SF and CW into slag-based geopolymer mortar exhibits excellent resistance to freeze-thaw cycles.

Table 2. The results before and after the freeze-thaw test

Mix ID	Compressive strength (MPa)		Flexural strength (MPa)		UPV (m/s)		Weight loss (%)
	Before	After	Before	After	Before	After	
20S40C40SF	33.31	36.65	6.26	4.31	2793	2875	0.34
20S45C35SF	31.72	34.93	4.82	3.45	2721	2814	0.49
20S35C45SF	36.27	38.79	6.58	4.59	2875	2956	0.27
20S50C30SF	28.65	31.45	4.11	2.85	2639	2743	0.56
20S30C50SF	38.32	40.18	6.98	4.92	2946	3082	0.19

3.5. Correlation Between Some Properties of the Resulting Matrix

To assess the consistency between the properties of the samples, the correlation relationship between 'compressive strength vs. UPV' and 'compressive strength vs. flexural strength' was carried out by using Linear Regression Analysis with Microsoft Office Excel program. The coefficient of correlation (R2), which represents the degree of correlation, between the 'compressive strength vs. UPV' and 'compressive strength vs. flexural strength' were calculated as 0.9956 (99.5%) and 0.9095 (90.9%), respectively (strong positive relationship) which is significantly higher than the expected minimum correlation factor of 75% as can be seen from Figures 6 and 7. According to these values, it can be stated that the Linear Regression Analysis showed a satisfactory relation between the investigated parameters. cycles.

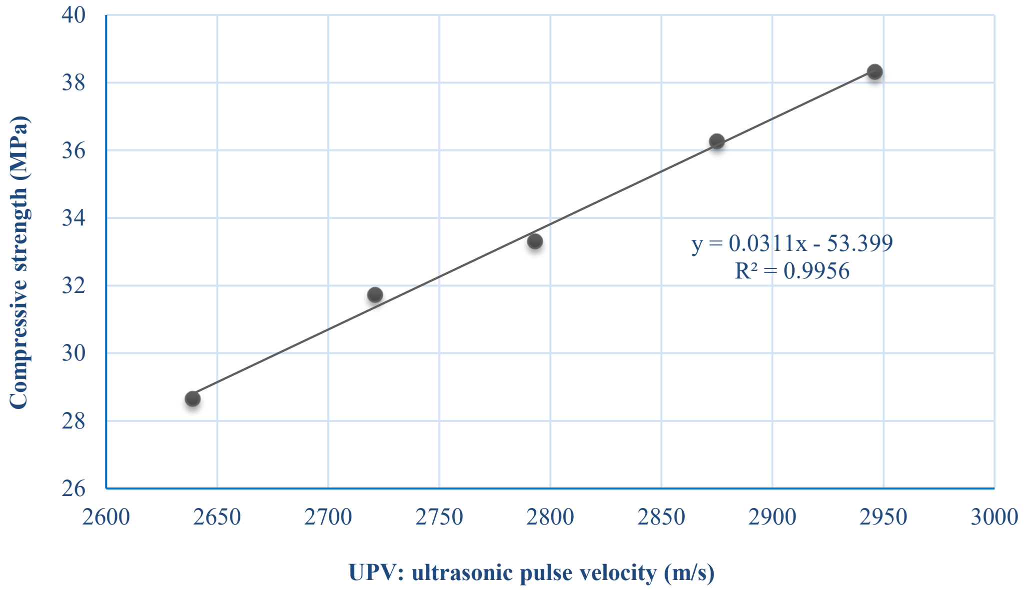

Figure 6. Linear regression analysis between UPV and compressive strength (28-day results)

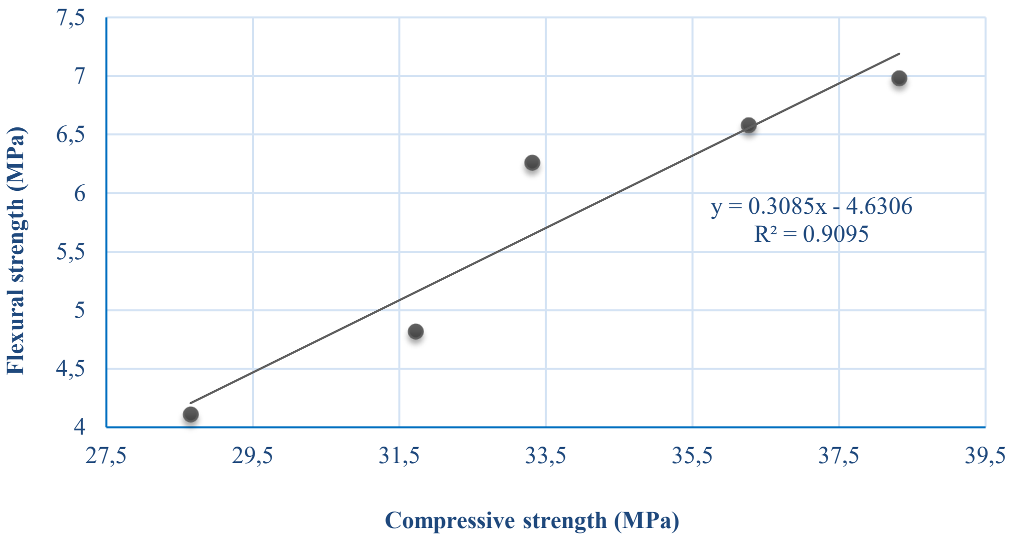

Figure 7. Linear regression analysis between compressive strength and flexural strength (28-day results)

4. CONCLUSION

The impact of the various proportions of SF and CW in slag-based geopolymer mortar on strength properties and freeze-thaw resistance were investigated, and the results were discussed. The geopolymer mortar containing higher SF gives better compressive strength, flexural strength and UPV results comparing to the specimens produced using a higher proportion of CW. In general, these values increased with the increase of SF content. According to this research, 50% inclusion of SF recorded the highest strength results, and the correlation relationship is satisfactory among different properties of the resulting mortar samples. However, to maintain sustainability and reduce the possible upcoming pressure on a single source of materials, the diversified use of materials should be practised without compromising core environmental and engineering properties. Therefore, the inclusion of a certain amount of CW must be practised since it does not cause a significant reduction in the overall strength of the resulting samples.

In conclusion, geopolymer concrete might be considered an eco-friendly, innovative, groundbreaking alternative as a material to traditional Portland cement concrete. In the future, by using geopolymer, the detrimental effects of the cement industry on the environment and atmosphere can be minimised, and the reusing of the industrial wastes can be increased. However, further investigations are required so as to decide if geopolymer can be replaced with Portland cement.

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