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Original article

Improving the Chemical Properties of Libyan Cement by Waste Glass Incorporation

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Abstract
This study is concerned with the recycling of colorless glass waste (produced from crushing the glass waste). Prepared in the form of a powder and used as an alternative to cement. The addition of glass powder improved the chemistry of Portland Libyan cement. It is suitable for various applications, such as the cementing process for oil wells. Chemical analysis of a sample of Libyan Portland cement originating from the Libda Cement Factory in Alkhoms city and the Union Cement Factory in Zliten city. The glass powder was mixed and analyzed with Libda and Union cement samples at a ratio (1%, 2%, 4%, 8%, 16%) from the weight of cement. That result was compared to the blank sample result for Libda and Union Cement. Good results are obtained by enhancing the chemical properties of Libda and Union cement. Cement resistance to sulfates was studied for prepared Libda cement samples. The results show that sulfate resistance increases as the percentage weight of glass powder increases relative to the control sample. Sulphate strength was investigated for prepared Libda cement samples. Environmental problems have been taken into consideration as a serious situation in modern construction. The reuse and recycling of waste are seen as the only method of reducing the waste generated. However, applications continue to have many opportunities for improvement.
Keywords: Cement, West Glass Powder, Environment, Portland Cement Concrete, Sulfate Resistance.

Introduction

ASTM C 150 defines Portland cement as "a hydraulic cement produced by pulverizing clinkers consisting essentially of hydraulic calcium silicates, calcium aluminates, ferrite, and usually containing one or more of the forms of calcium sulfate as an internal ground addition". In addition, there can be some minor oxides as a result of the cement manufacturing due to the presence of some impurities in the raw material composition [1]. The clinker has a composition of 67% CaO, 22% SiO_2, 5% $A_{12}O_3$, 3% Fe_2O_3, and 3% other products. All of these components, composed together, form the main cement phases, namely, alite, belite, aluminate, and ferrite, which react with water to form hydrated cement products. The phase compositions in Portland cement are denoted by ASTM as tricalcium silicate (C_3S), dicalcium silicate (C_2S), tricalcium aluminate (C_3A), and tetra calcium alumino ferrite (C_4AF). In the clinker, minor phases in addition to the four principal phases can be found. Most significant of these are the magnesium oxides MgO, alkalis in the form of Na_2O, K_2O, and the sulfates SO_3. In addition, calcium sulfate is a retarder used to delay the setting time of cement paste during the hydration process, with approximately 5 % of calcium sulfate. There are different compositions of calcium sulfate, which are gypsum ($CaSO_4.2H_2O$), hemihydrates ($CaSO_4.½H_2O$), and anhydrite ($CaSO_4$) at 40 °C under dry conditions. Gypsum ($CaSO_4.2H_2O$) slowly dehydrates to hemihydrate ($CaSO_4.½H_2O$) or anhydrite ($CaSO_4$). Portland cement may be blended with glassy cementitious materials to form blended hydraulic cements [2].

Millions of tons of waste glass are being generated annually all over the world. Once the glass becomes waste, it is disposed of at landfills, which is unsustainable as this does not decompose in the environment [3]. In the 1960s, many studies were carried out to utilize the crushed waste glasses as an aggregate for cement concrete production [4]. However, a study that had been conducted to produce architectural exposed aggregate for concrete since 1963 found that concretes with glass aggregates were easily cracked [5]. Owing to the high disposal cost of waste glass and environmental regulations, the use of glass as cement concrete aggregates has again come under the attention of researchers in the last 20 years [6]. This aggregate was applied in road construction and also used for the production of glass tiles, wall panels, bricks, glass fiber, agricultural fertilizer, landscaping reflective beads, and tableware [7]. However, a deleterious alkali-silica reaction (ASR) may be induced in concrete for the high content of glass aggregate. ASR is a surface area-dependent phenomenon and creates a gel that swells in the presence of moisture, causing cracks and reduction of strength. The expansion could be reduced if the glass were ground to a particle size of 300µm or smaller. Finely ground glass powder is the main cause of the ASR reaction to occur.

Most recent work has concentrated on studying the feasibility of using waste glass powder as a partial replacement of cement. It can react with portlandite in hydrated cement to form C-S-H in increasing the strength and durability of concrete because of the high silica content in glass powder [8]. There is a rising enthusiasm for utilizing glass waste in concrete. These interests have been exacerbated by the large amount of glass waste existing from empty bottles, broken windows, and glass containers. On the off chance that such glass could be consumed in concrete, it would impressively diminish the transfer of glass and take

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care of some of the environmental issues. Utilizing glass as a development material is among the most preferred in light of the conceivably decreasing the cost of glass transfer and concrete production. This inclusive also decreased bond strength between the aggregate and the cement paste [9]. Glass can be found molded in numerous shapes, like packaging of containers (bottles, jars), flat glass (windows, windscreens), lamp glass (light globes), cathode ray tube glass (TV screens, monitors, etc), all of which have a partial life in the shape they are produced and require to be reused/second hand in order to prevent environmental issues [10]. Generally, the utilization of waste in place of natural one is one of the most magnificent approaches to make concrete sustainable. A large, enormous quantity of glass materials in the shape of waste is generated in the world [11].
The Cement industry is considered one of the most energy-intensive industries due to the huge amount of energy needed during the production steps and the high temperature used to burn the clinker, up to 1500 °C. The energy cost of the cement production accounted for approximately 40% of its variable total cost, 50–60% in some countries [12,13]. It has been estimated that the cement industry consumed 5% of the total industrial energy consumption all over the world in 2006 [14]. Recently, the energy consumption increased to 12–15% of the total industrial energy consumption in the entire world, and it is expected that the total energy consumption will increase with the total cement production rate [13].
The atmospheric CO_2 concentration increased from 280 ppm at the start of the Industrial Revolution to 368 ppm at the start of this century [15]. The high increase in the CO_2 concentration is believed to be responsible for the Earth's climatic changes and global warming [16]. One of the major industrial emitters of greenhouse gases, especially CO_2, is the cement industry. It was estimated that the production of each ton of clinker releases one ton of CO_2 [15]. The cement industry accounted for about 7% of the world's total CO_2 emissions [17]. Moreover, it was calculated that the European cement industry contributed about 4.1% of the total CO_2 emissions in the EU in 2007 [12], concluded that the global CO_2 emission from the cement industry will increase by more than 50% by 2030 due to the increase in the production rate of cement annually. While a previous study [18] reported that reducing the amount of clinker in the blended cement could be considered as one of the effective methods to reduce the CO_2 emissions. Using waste materials mixed with the ground clinker to produce cement could be able to reduce the CO_2 emission by 5% or as high as 20%, depending on the percentage of replacement. Also, it was found that the production of each ton of cement clinker consumes 1.5–1.7 tons of the earth's natural resources as raw materials; as an example, the Chinese cement industry consumes about 1.5 billion tons of limestone and clay annually [14].
Using waste material in cement and concrete production can save the earth's natural resources, save energy and reduce the cost of the production of cement and the price of cement, as well as reduce the greenhouse gases emission and reduce the environ- mental impact of the solid wastes, especially the waste glass because of the nonbiodegradable nature of glass materials. In Table 1, there are exemplary examinations introduced, in which waste glass powder was utilized as an eco-friendly substantial admixture.

Table 1. Exemplary studies covering the topic of using waste glass aggregate as an eco-friendly admixture.

Mixture Content	Main Findings	Analyzed Properties	References
Glass powder as a 10–25% cement replacement. Mixture type: Mortar	Increase in strength, reducing costs.	Flow test, compressive strength, cost analysis	[3]
Glass powder as 5–25% cement replacement Mixture type: Traditional concrete	Increase in chemical shrinkage, increase in heat of hydration, increase in compressive strength,	Chemical shrinkage, heat of hydration, absorption, compressive strength	[19]
Glass powder as 5–25% cement replacement. Mixture type: Traditional concrete	Increase of compressive and tensile strength, decrease of absorption, Increase of density,	Thermo-gravimetric analysis, strength (compressive and tensile), slump test, density, sorption	[20]
Glass powder as a 10% and 20% cement replacement Mixture type: Traditional concrete	Increase in compressive strength, porosity reduction,	Slump test, strength (compressive and flexural), porosity	[21]
Expanded glass as a 50% and 100% replacement of natural aggregate. Cement content was not reduced. Mixture type: Mortar	Density reduction, increase of water absorption, compressive strength reduction, heat transferring rate reduction	Flow test, density, water absorption, thermal insulation	[22]

The main idea of this research is to examine the possibility of using glass powder as a partial cement replacement , The experiments in this research work have been carried out primarily to prove that glass powder improves the chemical properties for cement and it is appropriate for several applications like the

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oil well cementation process, since the properties required for the cement used in well cementation process are: to be fast hardening, high resistance to corrosion and poor conductor to heat.

Materials and Methods

Locally produced materials were used in this study. Cement used in this study was the Libyan ordinary Portland cement produced by the Libda Cement Factory and the Union Cement Factory. The chemical properties were analyzed for the two cements as control samples, which have no glass powder added. Then, glass powder was added at 1, 2, 3, 4, 8, and 16% into each Libda and Union cement. The comparison of chemical properties between the cement control samples and the incorporated waste glass powder are presented in Tables 2 and 3 for Libda and Union cements, respectively.

Table 2. Chemical composition (%) of cement samples from the Libda cement factory by X-ray diffraction.

Cement Sample	SiO_2	AL_2O_3	Fe_2O_3	CaO	MgO	Na_2O	K_2O	SO_3
Blank sample	24.50	4.50	3.56	61.13	2.96	0.15	0.27	1.83
1% Glass Powder	25.27	4.49	3.65	61.82	2.95	0.49	0.26	1.82
2% Glass Powder	26.40	4.43	3.60	60.33	2.97	0.33	0.47	1.77
4% Glass Powder	27.41	4.32	3.58	59.20	2.87	0.20	0.45	1.73
8% Glass Powder	27.41	4.17	3.55	57.15	2.84	0.28	0.22	1.65
16% Glass Powder	36.80	3.84	3.39	51.92	2.73	0.30	0.18	1.50

Table 3. Chemical composition (%) for the Union cement samples by using X-ray Diffraction

Cement Sample	SiO_2	AL_2O_3	Fe_2O_3	CaO	MgO	Na_2O	K_2O	SO_3
Blank Sample	20.99	4.84	3.35	63.51	1.91	0.07	0.85	2.63
2% Glass Powder	21.31	4.64	3.35	62.60	1.92	0.12	0.83	2.47
4% Glass Powder	22.01	4.59	3.30	62.35	1.96	0.18	0.83	2.46
8% Glass Powder	22.01	4.40	3.24	61.21	2.05	0.25	0.80	2.32
16% Glass Powder	24.28	4.01	3.13	59.30	2.10	0.41	0.74	2.04

The cement sample that contains 1% of glass powder in the union cement samples showed small differences with the 2% cement replacement samples. When the ratio of percentages of aluminum oxide to ferric oxide is 0.64 or more, the percentages of tricalcium silicate, dicalcium silicate, tricalcium aluminate, and tetra calcium aluminoferrite shall be calculated from the chemical analysis as follows:

Tricalcium silicate $(C_3S)=(4.071*\%cao)-(7.600*\%sio2) - (6.718*\%Al_2O_3)-(1.430*\% Fe_2O_3)-(2.852*\%S03)$ (1)

Dicalcium silicate $(C_2S)=(2.867*\%sio_2)-(0.7544*\%C_3S)$ (2)

Tricalciumaluminate $(C_3A)=(2.650*\%Al_2O_3)-(1.692*\%Fe_2O_3)$ (3)

Tetra calcium aluminoferrite $(C_4AF)=3.043*\%Fe_2O_3$ (4)

When the alumina-ferric oxide ratio is less than 0.64, a calcium aluminoferrite solid solution expressed as (C_4AF+C_2F) is formed. No tricalcium aluminate will be present in cements of this composition. Dicalcium silicate shall be calculated as in Eq (2). Contents of this solid solution and of tricalcium silicate shall be calculated by the following formulas:

$(C4AF+C2F)=(2.100*\%Al2O3)+(l.702*\%Fe2O3)$ (5)

Tricalcium silicate $(C3S)=(4.071*\%CaO)-(7.600*\%SiO2)-(4.479*\% Al2O3)-(2.859*\%Fe2O3)-(2.852*\%SO2)$ (6)

In addition to the four main compounds, many minor compounds occur in the furnace. Two of the minor oxides, K_2O and Na_2O, known as alkalis in cement, are of some importance and expressed in terms of Na2O. These alkalis react mainly with aggregate active silica and produce so-called alkali-silica gel. It creates cracks.

Waste glass powder

The waste glass used was clear and had been purified of impurities. The glass specimens were washed and dried in a 100-degree oven for approximately 20 minutes. Pieces of the glass were crushed by a hammer to qualify it in the second stage of grinding using an electric grinding machine, as shown in Figure 1. Grinding was done in two steps at different speeds. Each step took 180 seconds, then sifted with a 90 µm laboratory sieve as shown in Figure 3-5, to prepare this glass powder for the next step. The fine glass powder was finally transferred and stored; it was ready for analysis and experimentation. Waste glass and natural sand have approximately the same physical properties, as shown in Table 4. The comparison between the properties of waste glass and the properties of natural sand shows that the absorption rate of waste glass is lower than that of sand by 14%, i.e., this means that concrete made up with glass as an aggregate has a lower absorption rate for water [23]. These properties make waste glass an interesting material to be used as an aggregate in the production of concrete.

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Figure 1. Grinding Machine

Table 4. Physical properties of waste glass and sand

Physical property	Waste glass	Sand
Specific gravity	2.19	2.57
Density (kg/m³)	1672	1688
Absorption (%)	0.39	2.71
Pozzolanic index (%)	80	-
Thermal Conductivity (W/m. °C)	0.96	2.05

Chemical analysis had been done by using an X-ray Diffraction device in the laboratory of the Union Arabic factory, and the results from that analysis are shown in Table 5.

Table 5. Chemical properties of glass powder by using X-ray Diffraction

Oxide Composition	SiO_2	AL_2O_3	Fe_2O_3	CaO	MgO	Na_2O	K_2O	SO_3
Glass Powder	83.52	0.72	0.42	12.3	2.12	9.61	0.24	0.26

Experimental procedure

Five cement samples from the Libda cement factory were mixed with different proportions of glass powder (1%, 2%, 4%, 8%, 16%) of the cement weight. And four cement samples from the Union cement factory at Zliten were mixed with different proportions of glass powder (2%, 4%, 8%, 16%) of the cement weight. Figure 2 shows Union cement factory blended samples with a blank sample of cement and glass powder. A sample of cement from the study was collected manually. Glass powder was added to the Libyan cement sample by placing the percentage weight in a hermetically sealed container. It was shaken in various ways for approximately 5 minutes. The mixing process was carried out in two steps: the manual step and the electrical grinder mixing step to ensure the homogeneity of the two materials. The mixture was sifted with the sieve mentioned in section 2.1 to ensure the consistency in particle size of the two materials.

The blended samples of glass powder and cement were prepared in the proportions indicated for comparison with the blank cement sample that contains no glass powder, before a chemical analysis was performed. The mixture was poured into cylindrical moulds and left to dry from moisture for a week. The samples were then immersed in a container filled with water for three weeks in order to increase the hardening process. Finally, the samples were left in the open air for a month to dry completely as shown in Figure 3, and become ready to dry from moisture.

Figure 2. Libda cement factory blended samples with blank sample and glass powder

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Figure 3. Cement samples after pouring

Experiment on the resistance of the cement mixture to sulfate attack

The experiment aims to measure the extent of the cement's resistance to sulfates, which is to determine the best additive ratio of glass powder to the cement that resists acids and sulfates compared to the cement sample that does not contain glass powder. This is based on the weight loss as a result of corrosion in the cement mixture samples by the medium present (Na_2SO_4). A solution of sodium sulphate was prepared at a concentration of 16% (because the sulfur is present in nature in proportions less than 16%, for the places such as buildings near sea water, which are usually very salty), after which the samples were prepared, cleaned and weighted, then immersed in the solution for 2 hours as shown in Figure 4. Then the samples were taken and dried by placing them in a dryer oven in the laboratory of the Libido cement factory for 4 hours, after which the weight of each sample was measured. Then the process was repeated, and in weight of each sample was measured to calculate the loss in weight for comparison with the resulting weight from the first stage, where the calculation of the corrosion by weight loss can be performed. A maximum solution of 16% Na2SO4 was prepared for testing.

Figure 4. Immersing of cement samples in Na2SO4 solution

Standard composition requirements

Chemical and mineralogical composition has been determined via ASTM C 150 Standard Specification for Portland Cement, which covers eight types. The types of cement that were focused on in this research are Type II, for general use, and Type V-For use when high sulfate resistance is desired. The respective standard chemical requirements for the two types are prescribed in Table 6 [1].

Table 6. Chemical and mineralogical component of cements, wt%.

Cement Type (ASTM C150)	II	V
Aluminum oxide (Al_2O_3), max, %	6.0	----
Ferric oxide (Fe_2O_3), max, %	6.0	----
Sulfur trioxide (SO_3), max, %	3.0	2.3
Tricalcium aluminate (C_3A), max, %	8.0	5.0
($C_4AF + 2(C_3A)$), max, %*	----	25

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The limit on the sum ($C_4AF + 2(C_3A)$), in Table 6, provides control on the heat of hydration of the cement, determining the extent to which high sulfate-resistant cement can be produced by adding glass powder material. These results are compared to standard ASTM C 150 type V prescriptions. Tricalcium aluminate C_3A has been reduced by adding glass powder to meet the required specifications of less than 5%. C_3A values decreased in Libda cement samples after adding 8% of glass powder, as shown in Table 7. C_3A values decreased to the limits of sulfate-resistant cement specifications in samples named Union Factory after the addition of 16% of glass powder, as shown in Table 8.

Table 7. Chemical Standard Requirements ASTM C 150 for Libda cement factory samples

Cement Type	Spec. limit Type II	Blank sample	1% Glass Powder	2% Glass Powder	4% Glass Powder	Spec. limit Type V	8% Glass Powder	16% Glass Powder
Aluminum oxide (Al_2O_3), max, %	6.0	4.50	4.49	4.43	4.32	NA	4.17	3.84
Ferric oxide (Fe_2O_3), max, %	6.0	3.56	3.65	3.60	3.58	NA	3.55	3.39
Sulfur trioxide (SO_3), max, %	3.0	1.83	1.82	1.77	1.73	2.3	1.65	1.50
Tricalcium aluminate (C_3A), max, %^{B}	8.0	5.91	5.73	5.66	5.40	5.0	5.0	4.46
($C_4AF + 2(C_3A)$), max, %^{C}	A	22.64	22.56	22.142	21.68	25	20.79	19.23

Table 8. Chemical Standard Requirements ASTM C 150 for Union Cement Factory samples

Cement Type	Spec. limit Type II	blank sample	2% Glass Powder	4% Glass Powder	8% Glass Powder	Spec. limit Type V	16% Glass Powder
Aluminum oxide (Al_2O_3), max, %	6.0	4.836	4.641	4.587	4.404	NA	4.009
Ferric oxide (Fe_2O_3), max, %	6.0	3.354	3.323	3.298	3.239	NA	3.131
Sulfur trioxide (SO_3), max, %	3.0	2.629	2.469	2.459	2.319	2.3	2.039
Tricalcium aluminate (C_3A), max, %^{B}	8.0	7.142	6.676	6.576	6.190	5.0	5.000
($C_4AF + 2(C_3A)$), max, %^{C}	NA	24.250	23.263	23.188	22.237	25	20.183

Where: A = Not applicable.

High sulfate resistance

Cement samples (0%,1%,2%,4%,8%,16%) were weighed (0%,1%,2%,4%,8%,16%) before immersed for 2 hours in the Na2So4 solution, and then dried in the oven for 4 hours and weighed again, and the process was repeated as shown in Table 9.

Table 9. Weight results from the resistance to sulfates experiment

Cement sample	Weight Before Immersing (g) WBI	First stage weight (g) FSW	Second stage weight (g) SSW
Blank sample	151.450	148.490	146.313
1%	152.120	149.173	147.005
2%	153.730	150.857	148.743
4%	154.300	151.606	149.621
8%	155.510	153.007	151.159
16%	156.100	153.810	152.118

It was noted from this experiment longer the that the immersion in the solution, the greater the amount of weight loss, If the weight loss continued, the cement samples would have been diminished, but the corrosion only continued to a certain extent in which the cement sample forms a layer on the perimeter of the sample to prevent the continuity of corrosion.

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Table 10. Percentage of weight loss for cement samples

Cement sample	**W1= WBI - FSW**	**W2 = FSW - SSW**	**weight loss of the first stage% = (W1/WBI)×100**	**weight loss of the Second stage% = (W2/FSW)×100**
Blank sample	2.96	2.177	1.954	1.466
1%	2.947	2.168	1.937	1.453
2%	2.873	2.114	1.869	1.401
4%	2.694	1.985	1.746	1.309
8%	2.503	1.848	1.610	1.208
16%	2.29	1.692	1.467	1.100

Results and Discussion

Effect of cement replacement on silica

The effect of cement replacement on silica content is shown in Figures 5 and 6. The percentage of silica increases by increasing the proportion of added glass powder. This is due to the importance of silica in the compounds as the main components of Tricalcium silicate C_3S, Dicalcium silicate C_2S, which represents (70-80) % of cement strength. The increase requires very high heat in order to incorporate silica into clinker formation reactions. Thus, an increase in temperature could lead to damage to the coating of the kiln used in the processing.

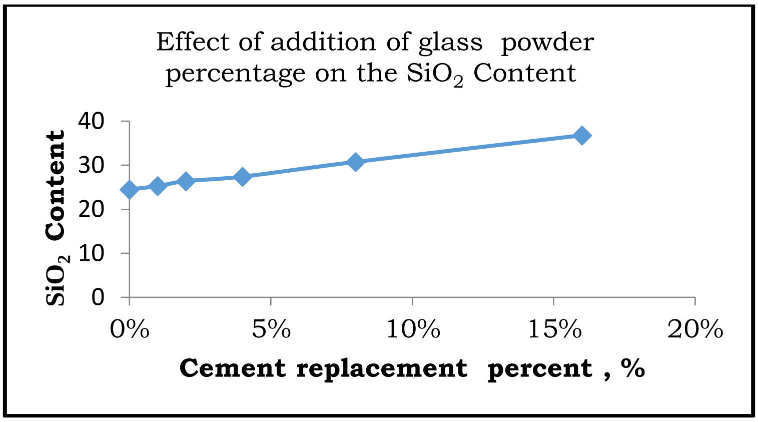

Figure 5. Libda Cement Factory samples

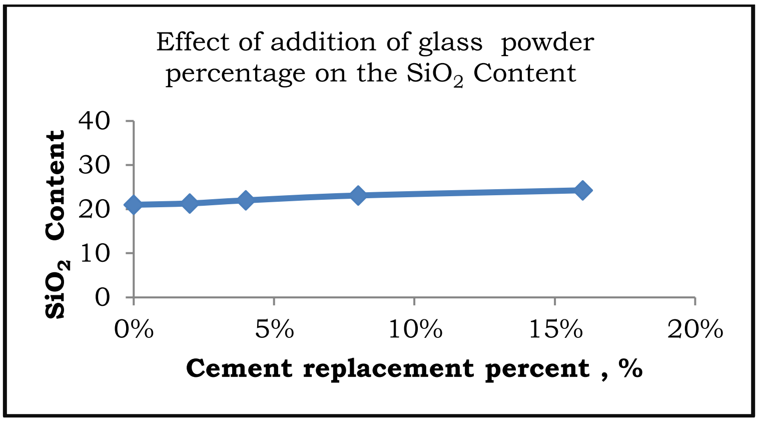

Figure 6. Union Cement Factory samples

Effect of cement replacement on

The effect of cement replacement on alumina content is shown in Figures 7 and 8. The alumina percentage decreases as the proportion of added glass decreases since alumina affects tricalcium aluminate C_3A according to equation (3). Therefore, the alumina ratio should be as low as possible because it reacts with sulfates to cause crashes and cracks in the construction. This is why it is expected that cement with a low alumina content to be highly anti-corrosion by sulfates.

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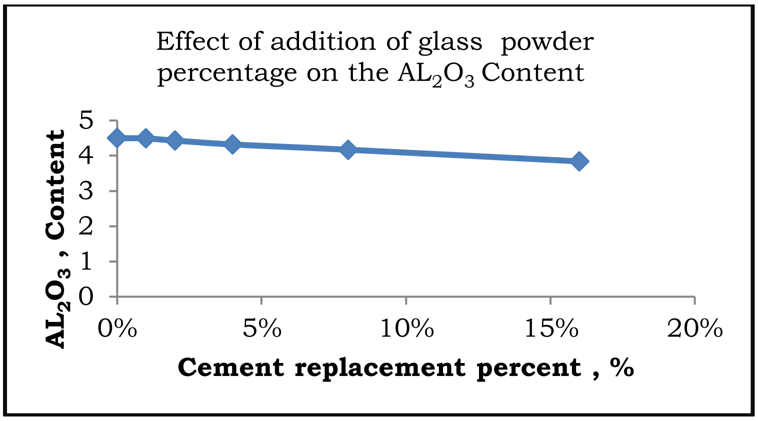

Figure 7. Libda Cement Factory samples

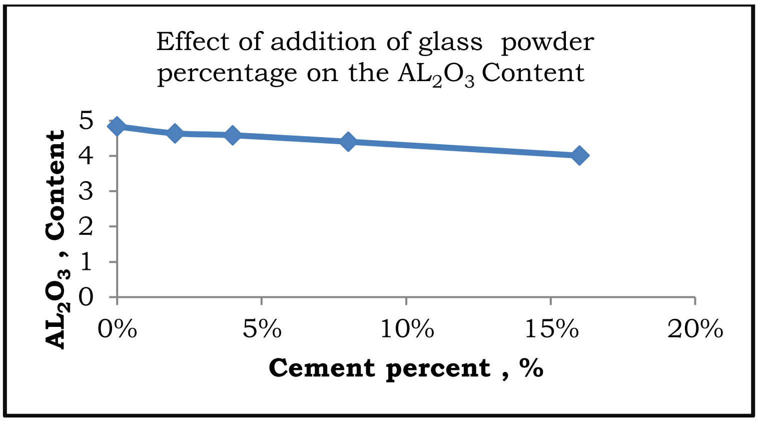

Figure 8. Union Cement Factory samples

Effect of cement replacement on ferric oxide

The effect of cement replacement on ferric oxide content is shown in Figures 9 and 10. The proportion of ferric oxide is limited in sulphate-resistant cement. The C_4AF percentage described in the equation (4), plus twice the C_3A percentage shown in the equation (3) is not more than 25% for sulphate-resistant cement. As for ordinary cement, the percentage is unlimited, but the high increase in ferric oxide leads to the production of clinker difficult to grind, thus increasing the cost of cement processing.

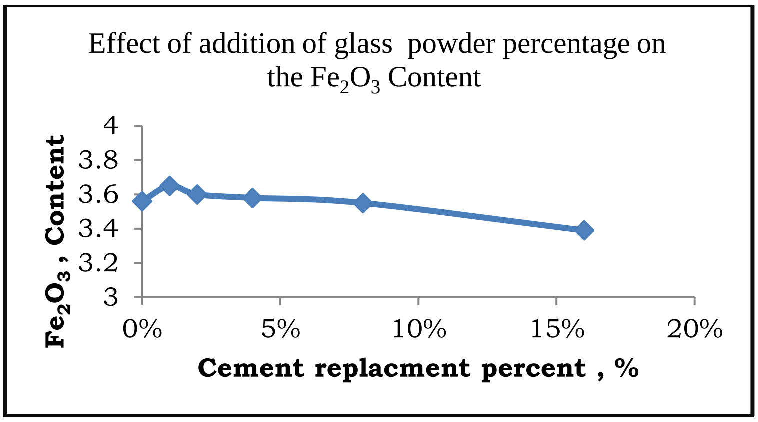

Figure 9. Libda Cement Factory samplesEffect of cement replacement on magnesium oxide

The effect of cement replacement on magnesium oxide content is shown in the Figures 11 and 12. Since magnesium oxide slowly reacts with water, it leads to the formation of magnesium hydroxide and causes volumetric expansion. The interaction of a sufficient an excess amount of water inside the concrete leads to damage. This is known in cement technology as unsoundness. There is also another problem caused by the interaction of magnesium oxide with carbon dioxide for the formation of magnesium carbonate, which causes concrete to crack. Therefore, it expected that shorter lifetime for a building that contains a high magnesium oxide content.

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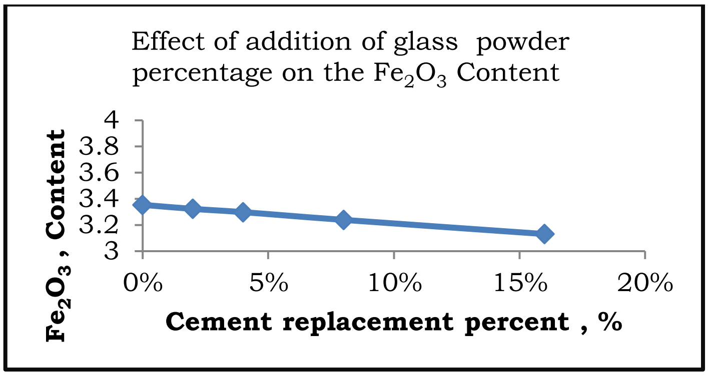

Figure 10. Union Cement Factory samples

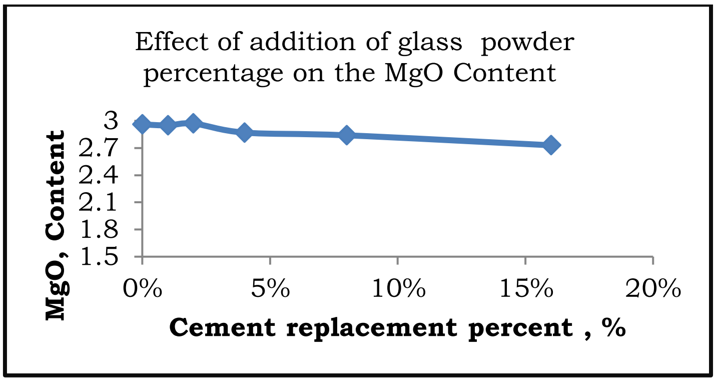

Figure 11. Libda Cement Factory samples

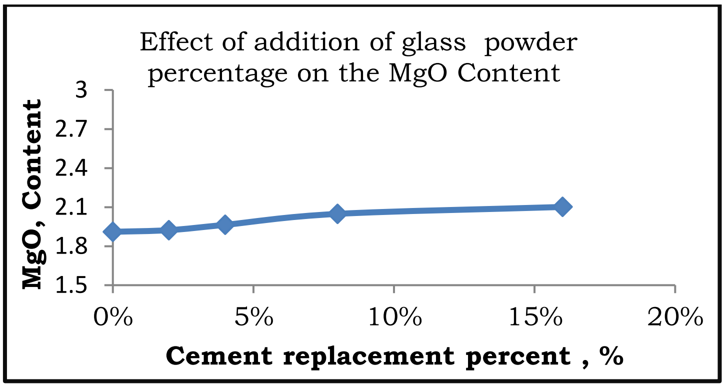

Figure 12. Union Cement Factory samples

Effect of cement replacement on weight loss

The effect of cement replacement on physical properties is shown in Figure 13. The result shows that the weight loss gradually decreases with the increase of glass powder. The obvious benefits of waste glass powder admixtures are the reduction impact sulfate attack by partial replacement of the Portland cement. replacing the Portland cement reduces the presence of compounds such as C_3A that cause ettringite formation.

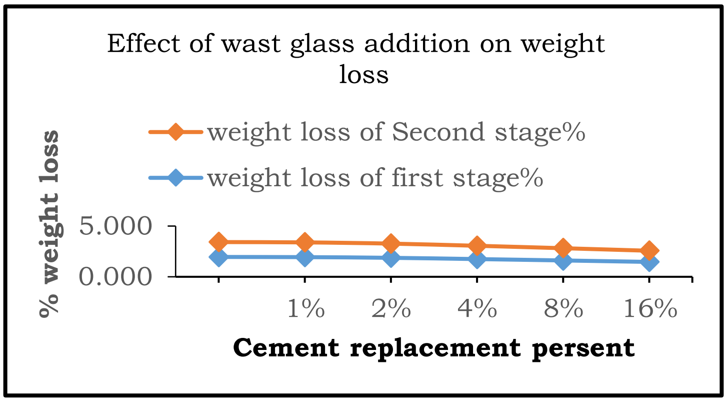

Figure 13. Weight loss (vs) Mixing rates for samples

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Conclusion

The study demonstrates that waste glass powder can be a valuable additive in cement production, improving both chemical and physical properties while offering economic and environmental benefits. By optimizing the replacement percentage, cement manufacturers can enhance sulphate resistance, reduce waste, and minimize carbon emissions, contributing to more sustainable construction practices. Further research could explore the ideal glass powder ratios for different cement applications to maximize efficiency and performance.

Conflict of interest. Nil

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