

Article

Synthesis and Evaluation of Geopolymer Mixtures Containing Chronologically Aged Basic Oxygen Furnace Slags

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Abstract: Applying industrial by-products as a substitution for conventional construction materials (natural resources) is a superior solution for the environment in terms of waste management and reduction in greenhouse emissions and for the construction industry in terms of cost and expenditure. Applying basic oxygen furnace slag (BOFS), one of the metallurgical industry by-products, as a construction material can be a high-potential and promising idea. However, the utilization of BOFS in construction applications is considerably limited because of its inherent characteristics leading to volumetric expansion behavior caused by the chemical reaction between free lime (f-CaO) and water. This study used geopolymer technology to stabilize the expansive behavior of chronologically aged BOFS aggregates. The compressive strength, expansion behavior, and drying shrinkage characteristics of a normal ordinary Portland cement (OPC) mixture and a geopolymer mixture containing siliceous river sand and chronologically aged BOFS aggregates were investigated. The test results showed that the compressive strength of geopolymer mixtures containing chronologically aged BOFS aggregate achieved 64.02 MPa, and the expansion behavior of geopolymer mixtures was improved compared with normal OPC mixtures containing the same BOFS aggregates, reaching 0.02% and 0.44%, respectively. However, due to the air-curing method, geopolymer mixtures had higher drying shrinkage values than normal OPC mixtures. Therefore, further studies should be conducted to investigate how to control the drying shrinkage of geopolymer mixtures containing chronologically aged BOFS aggregate.

Keywords: basic oxygen furnace slag; geopolymer; compressive strength; expansion; drying shrinkage



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1. Introduction

The rapid growth of economics and infrastructure worldwide has led to the dramatic use of concrete since it is one of the most durable and strong construction materials. The core component of concrete is the ordinary Portland cement (OPC) that binds together all aggregates and is mainly responsible for strength development. However, the production of OPC causes significant environmental issues by consuming many natural resources and liberating tons of greenhouse gas. Specifically, producing one ton of OPC releases one ton of carbon dioxide [1]. Moreover, the hydration process always going on during strength development in the concrete is also accompanied by the release of greenhouse gasses, leading to global warming [2]. Thus, cement manufacturing is responsible for 5–7% of global carbon dioxide (CO₂) emissions [3].

Since the world continues to deal with significant environmental issues [1], developing an alternative material to OPC has become extremely important. Geopolymer arrives as a

replacement for conventional concrete. It is considered the third generation of cement after lime and OPC [4].

Geopolymer is an inorganic polymer material synthesized through activating alumina (Al)–silica (Si) sources with alkali or, in some rare cases, with acidic activators (phosphoric acid or phosphate) [5]. As an alumina–silica source, metakaolin, kaolinite, clays, zeolite, fly ash, ground granulated blast slag, or other Al–Si-rich cementitious materials might be used [6]. Even though metakaolin is one of the most widely used materials for synthesizing geopolymer, geopolymer composites can include both natural raw materials (clays and volcanic tuff) and waste materials (concrete waste, rubble, glass, rice husk ashes, palm oil, etc.) [7,8]. Sodium and potassium-based materials including sodium hydroxide (NaOH), sodium silicate (Na_2SiO_3), potassium silicate (K_2SiO_3), and potassium hydroxide (KOH) are widely used to activate alumina–silica materials [9]. The term “geopolymerization” was first introduced by Joseph Davidovits in 1978 and was described as a cementitious material with ceramic properties [10,11]. Although the exact mechanism of the geopolymerization process is not thoroughly investigated, there are prominent proposed mechanisms to explain its setting and hardening [12]. It is a complicated process containing three basic steps: (1) rapid dissolution of Al and Si elements under hydroxide actions, (2) reorientation and diffusion of dissolved species monomers, and (3) a polycondensation process that forms a three-dimensional network structure containing a Si–O–Al–O or Si–O–Al–O–P– linkage [13–17].

The geopolymers have amorphous microstructures. Geopolymers’ properties are influenced by the type and subsequent amount of raw materials and optimized mixture proportion (design) [18]. It was proven by many researchers all over the world that properly designed geopolymer concrete subjected to steam curing demonstrated superior engineering properties compared with conventional OPC concrete [19–23].

Unlike ordinary hydraulic cement, the geopolymer does not require moisture or water curing. Air or steam curing is generally used to initiate geopolymerization [24]. Since the manufacturing of a geopolymer mixture does not require high energy and uses residual materials, geopolymer is considered an ecologically friendly green material [25]. Low cost and low greenhouse gas emissions are also among the advantages of geopolymers. Moreover, geopolymers have higher resistance to fire compared with OPC. At high temperatures, its chemical structure remains stable and does not undergo breakdown, unlike OPC hydration products [26].

According to the governmental report of the Republic of Kazakhstan, from October 2020, about 31.6 billion tons of industrial waste have been accumulated, 70% of which constitutes ash. Moreover, approximately one billion tons of industrial waste are annually produced in Kazakhstan [27]. Thus, applying industrial by-products as a substitution for conventional construction materials or non-renewable natural resources is a superior solution for the environment in terms of waste management and for the construction industry in terms of cost and expenditure [28].

Steel slags, the by-products of the metallurgy industry, can be used as an aggregate for geopolymer concrete since they have a sufficient chemical composition. Depending on the furnace type, generated slags might be categorized mainly into three groups: blast furnace slag (BFS), basic oxygen furnace slag (BOFS), and ladle furnace slag (LFS). Among these three types of slags, more than 100 million tons of BOFS are produced annually by steelmaking and mining industries worldwide [29]. Of the total slag generated in China, 70% is BOFS [30]. The accumulation of BOFS in landfills causes severe environmental problems such as land occupation and contamination of soil and water sources. Therefore, the utilization of slag has drawn the research community’s attention. The utilization of BOFS is well managed in developed countries. Generally, BOFS is used as aggregates in road construction and additives in cement production [31–33]. Since cement and concrete are products in the highest demand in the construction industries worldwide, using BOFS as a construction material, considering its mechanical and energy-saving properties, can be one of the practical and beneficial solutions to existing pollution and resource-saving problems.

The chemical composition of BOFS might vary due to several external factors during manufacturing depending on iron ores and admixtures, steelmaking, and cooling methods. Typically, along with main components such as CaO, SiO₂, Al₂O₃, and Fe₂O₃, BOFS contains high amounts of free calcium (f-CaO) and free magnesium (f-MgO) [34–37]. The f-CaO and f-MgO occur through the process treated by blowing oxygen to remove carbon and other elements in hot molten iron. These free oxides expand during hydration, causing internal pressure, surface protrusion, and cracks [38–40]. This expanding propensity of BOFS limits the utilization of BOFS as construction materials. Therefore, the stabilization of BOFS is a key factor for alternative aggregates used in construction applications.

One of the methods to use BOFS aggregates as construction materials is to apply for the mineral carbonation, which is based on the reaction of CO₂ with metal oxide-bearing materials (CaO, MgO, and Ca(OH)₂) to form insoluble carbonates (CaCO₃ and MgCO₃). This process is an emerging thermochemical solid-looping process for CO₂ capture and CO₂-related energy conversion and storage [41–43]. Moreover, on top of trapping CO₂ in a geochemically stable form, the mineral carbonation process can also bring benefits such as stabilizing leaching and structural integrity of BOFS, enabling further valorization of waste BOFS materials considering reduced waste treatment, landfilling costs, and the production of marketable products.

Another promising innovative solution to utilize BOFS aggregate is the geopolymer mixture. During the geopolymerization process, complex chemical reactions occur, and the Si-O-Al framework is formed. SiO₄ and AlO₄ tetrahedra are connected through the O₂ atoms in it. Such a geopolymer structure provides improved mechanical properties such as high compressive strength, durability, and low permeability. Moreover, the geopolymer framework contains high free SiO (f-SiO) contents. The reaction of silica with f-CaO and f-MgO ends up with the formation of wollastonite (CaSiO₃) and enstatite (MgSiO₃), which are volumetrically stable products [44–47]. Thus, the expansion of BOFS aggregates in geopolymer mixtures would be minimized or eliminated, and it might be a sustainable and innovative alternative composite concrete that is superior to conventional OPC concrete.

It is worthwhile to think about creating a study to look into the possible use of BOFS produced in Kazakhstan as construction materials and their application, starting with sustainable development and the reuse of waste resources. Therefore, this study aims to utilize BOFS in a geopolymer mixture as an aggregate source and develop an optimum user-friendly mix design of BOFS geopolymer mixtures without expansion problems. This study compares the performances of a normal OPC mixture with geopolymer mixtures containing BOFS aggregates. This study used fresh (unaged) and chronologically aged BOFS aggregate, ground granulated blast furnace slag (GGBFS), ASTM class F fly ash (FFA), and alkali-activated solutions to create geopolymer mixtures. Compressive strength, expansion characteristics, drying shrinkage, and microstructural analyses were conducted to investigate the possibility of utilizing BOFS as an aggregate for the geopolymer mixture. The effect of the aging duration of the BOFS aggregate on compressive strength and expansion behavior was investigated.

2. Research Objective and Scope

The main objective of this study was to investigate the mechanical and swelling properties of geopolymer mixtures containing fresh (unaged) and chronologically aged BOFS aggregate. As presented in Figure 1, the experimental program of the study contains the following steps: phase I, a full-scale essential material characterization of fresh and chronologically aged BOFS, and phase II, an evaluation of the fresh and hardened properties of geopolymer mixtures with fresh and chronologically aged BOFS aggregates. Evaluations and comparisons of the engineering properties of normal OPC and geopolymer mixtures were provided.

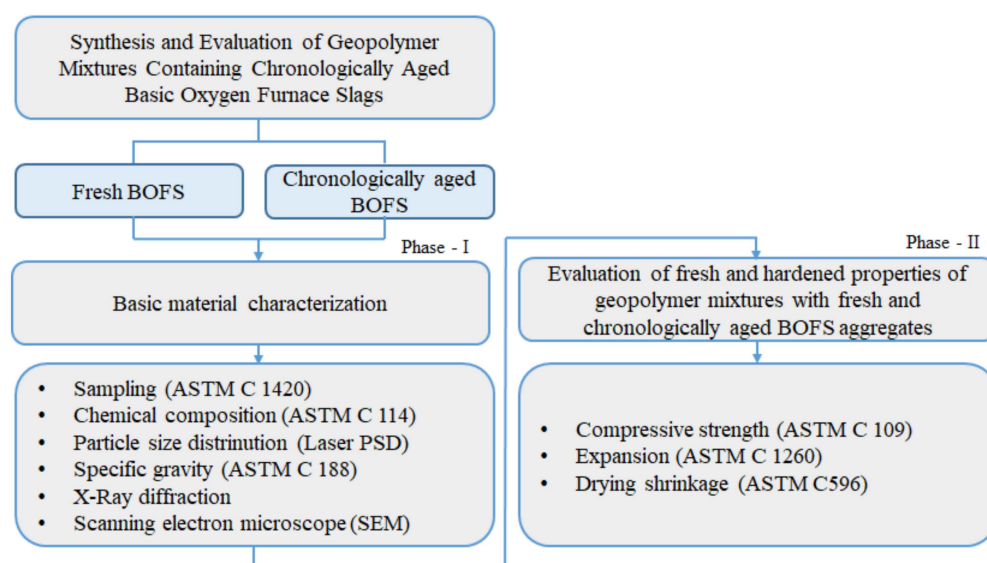


Figure 1. Diagram of the experimental program. Standards [48? ?–51].

3. Experimental Programs

3.1. Sampling of Stockpiled BOFS Materials

In this study, materials that were used as aggregates were classified mainly into two groups: (1) natural river sand for the control group and (2) the BOFS aggregate group. The BOFS was obtained and provided by JSC ArcelorMittal Temirtau, one of Kazakhstan’s biggest steelmaking and mining companies. Depending on the aged (storage) conditions, the BOFS used in this study was classified as fresh and chronologically aged. The chronologically aged BOFS materials were subjected to natural aging (weathering) for many years, approximately from 1 to 30 years. As illustrated in Figure 2, according to the location, excavated stockpiled BOFS materials were divided into three types: top (1–10 years), middle (11–20 years), and bottom (21–30 years). The fresh BOFS material (approximately less than one month) referred to slag that was subjected to natural cooling. Based on slag types, the following mixture proportions were prepared: a natural river sand mixture, fresh BOFS samples, a top BOFS mixture, a middle BOFS mixture, and a bottom BOFS mixture.



Figure 2. BOFS aggregate: (a) Field stockpiled BOFS with different ages. (b) Bottom BOFS.

3.2. Material Characteristics

Siliceous river sand, fresh and chronologically aged (stockpiled) BOFS aggregate, FFA, and GGBFS were used in order to cast geopolymer mixtures. The chemical compositions of all materials were investigated through an X-ray fluorescence (XRF) spectrometer by a PANalytical machine (Axios FAST, Malvern, UK). Some differences existed between the oxide compositions of the fresh and stockpiled BOFS. Specifically, the fresh BOFS was generally

characterized by lower Al_2O_3 , SiO_2 , Fe_2O_3 , and SO_3 and higher CaO and MgO than those in the stockpiled BOFS. Both the river sand and BOFS aggregate were sieved with different fractions and maintained with the same gradation to produce uniform geopolymer specimens for each test (2.36 mm of 10%, 1.18 mm of 25%, 600 μm of 25%, 300 μm of 25%, and 150 μm of 10%). Bulk chemical analyses and aggregate properties of river sand and BOFS are presented in Table 1. In the bulk chemical analysis, the calcium content determination included the calcium that was part of the compounds and “free” calcium ions present.

Table 1. Chemical compositions and aggregate properties of river sand and BOFS aggregates.

Chemical Compositions									
	CaO	SiO ₂	Fe ₂ O ₃	MgO	Al ₂ O ₃	P ₂ O ₅	MnO	TiO ₂	Cr ₂ O ₃
River sand	12.01	64.46	4.26	1.51	11.38	0.39	0.44	0.37	-
Fresh BOFS	40.28	8.03	36.93	2.63	0.61	2.75	5.35	0.94	0.39
Top BOFS	41.17	22.83	19.64	6.67	3.22	2.68	1.97	0.39	0.39
Middle BOFS	44.02	19.79	19.00	7.33	2.19	3.75	2.18	0.39	0.43
Bottom BOFS	27.73	33.76	10.57	6.49	16.60	1.47	1.10	0.66	0.27
Aggregate Properties									
	Specific Gravity (%)	Absorption Capacity (%)		Size of Aggregate (Sieve Number)			Mass (%)		
River sand	2.68	2.87		2.36 mm (#8)			10%		
Fresh BOFS	3.33	5.08		1.18 mm (#16)			25%		
Top BOFS	2.91	3.28		600 μm (#30)			25%		
Middle BOFS	2.86	5.27		300 μm (#50)			25%		
Bottom BOFS	2.60	10.22		150 μm (#100)			15%		

Moreover, the mineralogical analysis was performed using an X-ray diffraction (XRD) system using Rigaku SmartLab equipment (Tokyo, Japan), and the results are plotted in Figure 3. The powder 45 micron-sized samples were step-scanned from 10 to 70° (2 θ) in increments of 0.01°. The raw data obtained from the investigation were analyzed through the Jade 6.5 software. The XRD patterns of the BOFS slag samples were significantly complex. It was shown that the most intense peaks were developed at 2-theta values of 21.5° and 30.5°. The XRD pattern of the BOFS aggregate showed that the fresh BOFS aggregate presented lime (CaO) and periclase (MgO), while other chronically aged BOFS aggregates did not show lime. Chronically aged BOFS aggregate’s crystalline phases mainly included portlandite (Ca(OH)₂), quartz (SiO₂), and calcite (CaCO₃). Portlandite presented in both fresh and aged BOFS, which was expected because BOFS contained lime (CaO). Free lime converted to Ca(OH)₂ in the presence of water.

In this study, three types of cementitious materials were used as binders. The FFA and GGBFS were used as binders for the geopolymer mixture, whereas OPC was used for the reference mixture created with OPC. The specific gravities of OPC, FFA, and GGBFS were defined as 3.15, 1.87, and 3.05, respectively, according to the ASTM C188 Standard Test Method for Density of Hydraulic Cement [49]. The chemical compositions of these binders are presented in Table 2.

The particle size distributions (PSDs) of the binder materials were performed using a laser scattering analyzer Mastersizer 3000 (Malvern, UK), which measured particle size distributions from 0.01 μm up to 3.5 mm. The mean values of D₁₀ (μm), D₅₀ (μm), and D₉₀ (μm) for FFA were 1.19, 5.4, and 16.1, while the mean values of D₁₀ (μm), D₅₀ (μm), and D₉₀ (μm) for GGBFS were 0.804, 2.66, and 7.07.

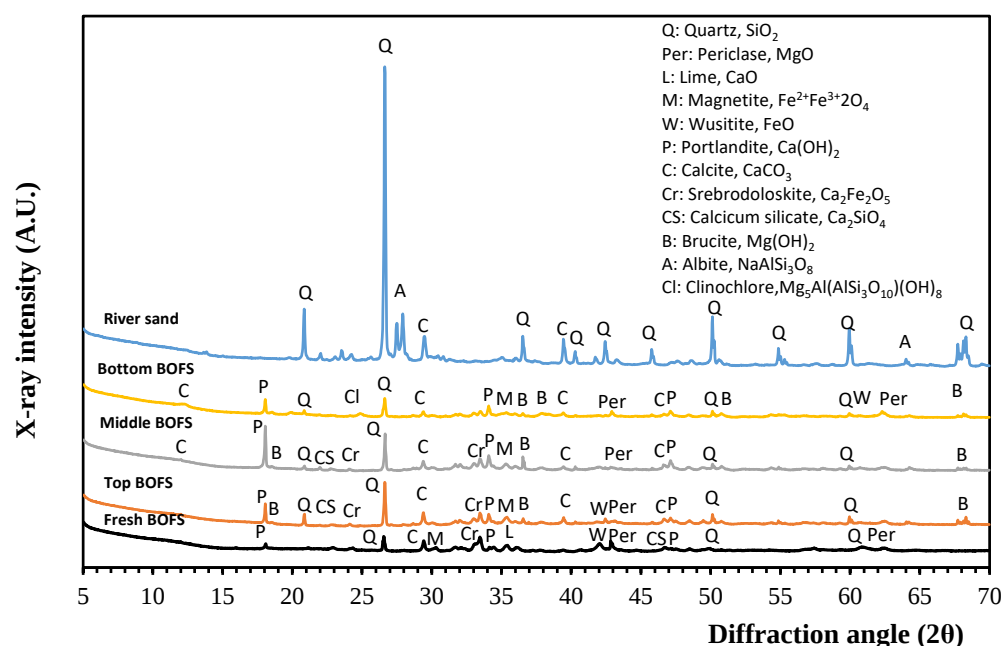


Figure 3. X-ray diffraction patterns of river sand and BOFS aggregates.

Table 2. Chemical compositions of cementitious materials.

Chemical Compositions (%)									
	CaO	SiO ₂	Al ₂ O ₃	Fe ₂ O ₃	SO ₃	MgO	K ₂ O	TiO ₂	MnO
OPC	69.19	17.41	4.36	2.96	2.61	2.05	0.95	0.20	0.12
FFA	1.86	65.34	24.85	4.01	0.31	0.53	0.68	1.10	0.07
GGBFS	40.51	32.25	11.40	0.33	2.00	10.46	0.88	1.41	0.31

3.3. Mixture Proportions and Sample Preparation

The volumetric mix design method was applied for both OPC and geopolymer mixtures. While the water to cement ratio was maintained at a constant 0.4 for OPC mixtures, the alkali-activated solution (AAS) to binder ratio was kept at 0.4 for geopolymer mixtures. This was because decreasing the w/c or AAS/b below 0.4 led to a non-workable consistency for the mixtures [54]. For all geopolymer mixtures, the GGBFS to (FFA + GGBFS) ratio was kept at 0.5, and the Na_2SiO_3 to NaOH ratio was also kept constant at 2.5 by mixing sodium silicate (Na_2SiO_3) and 12 M sodium hydroxide (NaOH) solution for all mixtures. Finally, the sand to binder ratio was kept at 0.3. Generally, the increase in the concentration of NaOH led to the growth of compressive strength [55]. However, the increase in the NaOH concentration beyond 12 M caused a negative effect on the geopolymerization process, which resulted in a reduction in the compressive strength [56]. At the concentration of 12 M NaOH, the workability of the mixture was also satisfied [57]. Therefore, the provided values were established for mixture design.

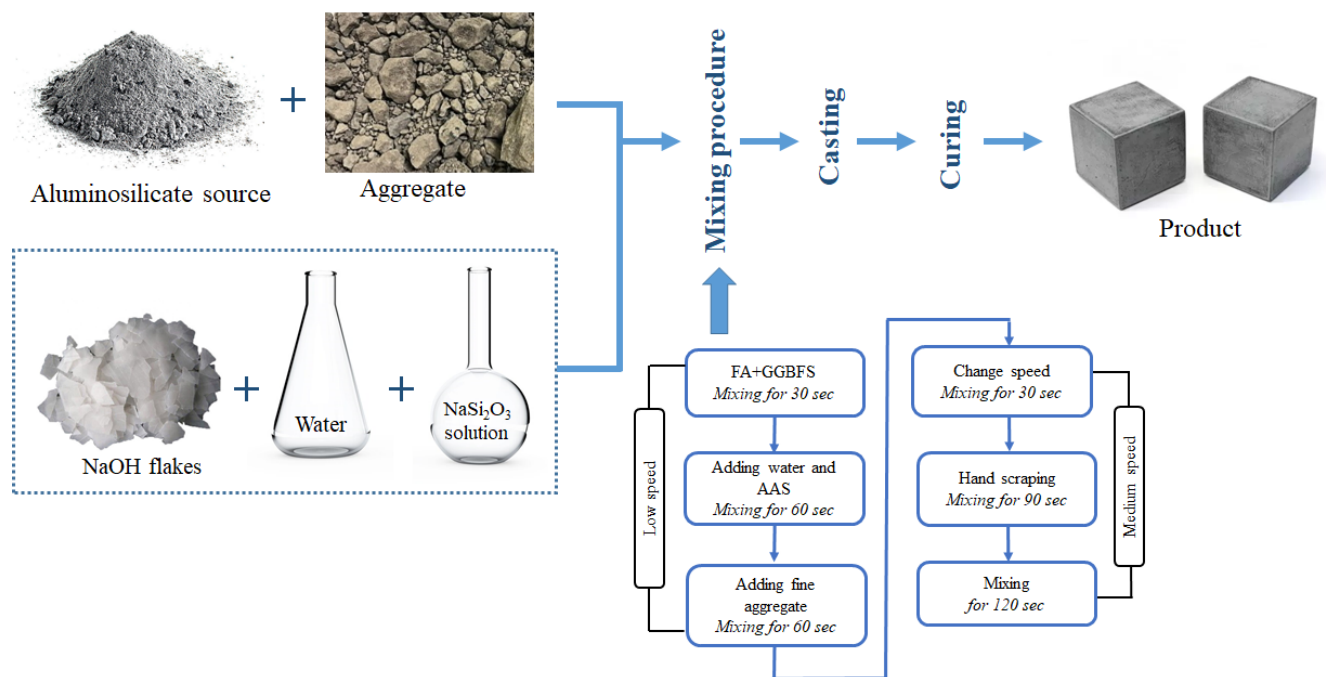
In the case of a normal OPC mixture, each material's specific gravity and water absorption capacity were considered as a mixture proportion design. The mixture design of the OPC and geopolymer mixtures is represented in Table 3.

Figure 4 schematically summarizes the procedures of mixing, casting, and curing samples for geopolymer mortar. The mixing procedure for the normal OPC mixture was performed according to the ASTM C305 Standard Practice for Mechanical Mixing of Hydraulic Cement Pastes and Mortars of Plastic Consistency [58].

Table 3. Mixture design of normal OPC and geopolymer mixtures.

OPC Mixtures (kg/m ³)						
	Water	Cement	River sand	BOFS		
River sand-N	222.0	555.0	1608.8	-		
Fresh BOFS-N	222.0	555.0	-	2001.8		
Top BOFS-N	222.0	555.0	-	1748.1		
Middle BOFS-N	222.0	555.0	-	1719.6		
Bottom BOFS-N	222.0	555.0	-	1563.7		

Geopolymer Mixtures (kg/m ³)						
	Water	FFA	GGBFS	River sand	BOFS	AAS
River sand-G	122.6	522.3	522.3	313.4	-	417.8
Fresh BOFS-G	125.4	534.5	534.5	-	320.7	427.6
Top BOFS-G	123.7	527.3	527.2	-	316.3	421.7
Middle BOFS-G	123.5	526.2	526.2	-	315.7	420.9
Bottom BOFS-G	122.1	520.4	520.4	-	312.3	416.3

**Figure 4.** Mixing procedures for geopolymer mixture.

However, since there is no established specification on the preparation of a geopolymer mixture, the following mixing procedure was applied to gain a homogenous geopolymer mixture. The sample preparation started with dry mixing sufficient amounts of GGBFS and FFA in a Hobart mixer for 30 s. Then, water and AAS solution were added simultaneously and mixed for one minute. The AAS was prepared 24 h before casting. After adding an entire quantity of river sand or BOFS at oven-dry conditions, mixing was continued for one more minute. The next step was changing the mixing speed from low (136 rpm) to medium (580 rpm), and the mixing was continued for one minute. After a minute was over, the mixer was stopped, and hand scraping was started, focusing on the walls and bottom part of the bowl. Hand scraping lasted 1.5 min, and then mixing at medium speed continued for two minutes. This provided mixing procedure ensures homogeneity.

The ready mixtures were cast into greased molds right after being tested for flowability and air content. Depending on the conducted test type, samples in various shapes were prepared. All samples were air-cured under controlled room temperature (23 ± 2 °C) and

humidity (70~80% RH) until each test period except for bar samples that were cured in the air only for the first day and after were submerged in water or 1 M NaOH solution at 80 °C [40].

3.4. Test Methods

The compressive strength of geopolymers and normal OPC mixtures was evaluated according to the ASTM C109/C109M Standard Test Method for Compressive Strength of Hydraulic Cement Mortars [52] on four air-cured cubic specimens with dimensions of 50 × 50 × 50 mm at the rate of 0.125 MPa/s. Strength measures were taken on the 7th and 28th days of strength development.

The expansion behavior of the geopolymer mixture was examined according to the ASTM C1260, “Standard Test Method for Potential Alkali Reactivity of Aggregates (Mortar-Bar Method)”, by measuring the length change of bar samples with dimensions 25 × 25 × 285 mm [53]. The first group of samples was submerged in water and put in an oven that provided 80 °C of temperature. The measurements were taken every day for the first up to 10 days, then once in three days. The second group of samples was submerged into 1 M NaOH solution to investigate the resistance to the alkali-silica reaction.

The 25 × 25 × 285 mm bar samples were prepared for the shrinkage test that was conducted following the ASTM C596 Standard Test Method for Drying Shrinkage of Mortar Containing Hydraulic Cement [48]. The samples were placed horizontally in the room maintained at a temperature of 23 ± 2 °C and a relative humidity of 50 ± 4%. Four samples were tested from each group, and the average of these four tests was taken as the drying shrinkage rate. The length change measurement was taken with 3- or 4-day intervals until 56 days, then once in 7 days up to 147 days. Also, the weight change of the samples was recorded simultaneously with the drying shrinkage measurement.

A length comparator device measured the bar length for expansion and drying shrinkage specimens. The expansion or shrinkage of bar specimens is calculated by Equation (1):

$$L = \frac{(L_x - L_i)}{G} \times 100, \quad (1)$$

where L = change in length at x age, %; L_x = comparator reading of specimen at x age minus comparator reading of reference bar at x age (mm); L_i = initial comparator reading of specimen minus comparator reading of reference bar at that same time (mm); and G = nominal gauge length (250 mm).

Finally, the morphological evaluation of materials was investigated through scanning electron microscope (SEM) images using the JSM-IT200 InTouchScope™ SEM (JEOL, Astana, Kazakhstan). Since OPC and geopolymer mixtures are not electrically conductive, the samples were coated with Au with a thickness of 20.0 nm by Magnetron Sputtering System—LAB-18 equipment.

4. Test Results and Discussions

4.1. Compressive Strength

The compressive strength for the normal OPC mixture (a) and the geopolymer mixture (b) is represented in Figure 5. With the exception of a normal bottom mixture, all cases showed strength increases over time. It is well known that basically, due to the hydration reaction between water and cement, hydration products, especially C-S-H gel that is responsible for the strength of cement-based materials, are produced and positively affect compressive strength development over time. The exception in the bottom-N mixture might be explained by the presence of $\text{Ca}(\text{OH})_2$ in the bottom BOFS, which initiated microcracks inside the sample and resulted in low strength at 28 days.

Figure 5 also presents a negative correlation between the strength and duration of the natural weathering of aggregates. The mixture containing the longer-aged BOFS aggregate showed a lower compressive strength, which was theoretically logical. Because BOFS was exposed to more prolonged natural weathering, it contained more calcite (CaCO_3) and portlandite ($\text{Ca}(\text{OH})_2$). These minerals negatively affected the strength development.

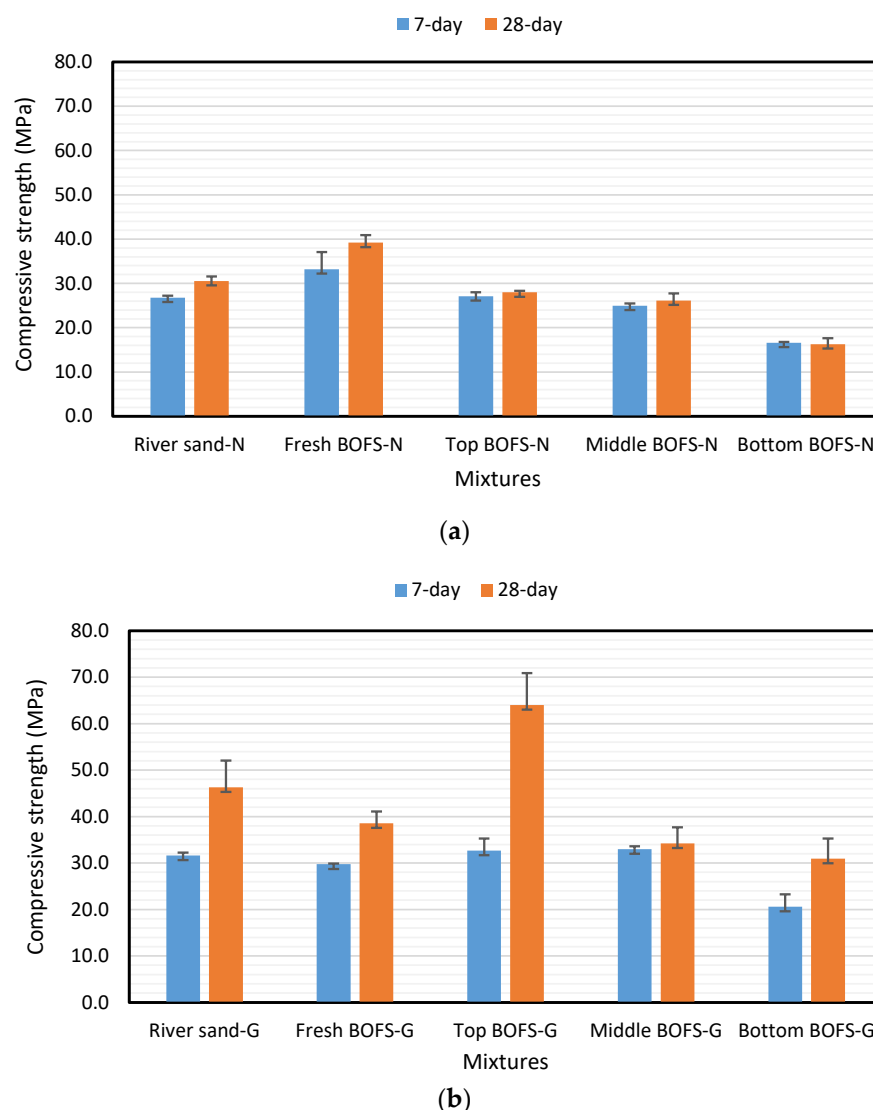


Figure 5. Compressive strength of (a) normal mortar mixtures and (b) geopolymer mixtures.

As shown in Figure 5, the compressive strength of geopolymer mixtures was higher than that of normal OPC mixtures except for the mixture having fresh BOFS. The compressive strength of geopolymer mixtures in comparison with normal OPC mixtures was higher by at least 5–10 MPa at 7 days. The strength difference between the normal OPC and geopolymer mixtures became increasingly notable at 28 days. The compressive strength of the geopolymer samples was higher than that of the OPC mixtures on average at 22.2 MPa. The compressive strength of geopolymer concrete depends on many factors such as alumina source, raw materials composition, and AAS [59]. The gel phase to undissolved Al–Si particle ratio also affects the compressive strength. In a low-calcium or calcium-free binder case, the main hydration product is N–A–S–H gel that has a three-dimensional structure, and this hydration product is responsible for strength development in the geopolymer system [60,61]. Hence, it might be concluded that the compressive strength of the geopolymer might be adjusted to a satisfactory level.

Interestingly, the normal mixture containing fresh BOFS had slightly higher compressive strength than the geopolymer mixture with the same fresh BOFS. For example, while the compressive strengths of fresh BOFS-N at 7 and 28 days were 33.2 MPa and 39.2, those of fresh BOFS-G were 29.7 MPa and 38.6 MPa. This result may be due to higher drying and chemical shrinkages of the geopolymer mixture having fresh BOFS. It should be noted that fresh BOFS has a higher water absorption capacity than the other BOFS, and the

geopolymer mixture typically provides higher chemical shrinkage than the normal cement mixture, resulting in microcracking inside the specimen.

4.2. Expansion Characteristics of Mortar Bar

4.2.1. Expansion in the Water

Figure 6 presents the test results of accelerated mortar bar expansion for OPC (a) and geopolymer (b) mixtures submerged in water. Regardless of the mixture type, the expansions of the river sand, top BOFS, middle BOFS, and bottom BOFS mixtures were significantly low. The maximum expansion of these mixtures was no more than 0.03%, which meant that these aggregates were non-reactive under water-submersed conditions. It should be noted that since the top BOFS, middle BOFS, and bottom BOFS were subjected to natural weathering for a long time in the presence of CO_2 , f-CaO and f-MgO reacted with it and formed stable CaCO_3 and MgCO_3 . It explained the insignificant expansion of those samples in the water. The formation of CaCO_3 and MgCO_3 was supported by the XRD results described in the earlier section (Figure 3).

However, the fresh BOFS mixture (fresh-N) performed oppositely. This mixture sharply increased up to 5 days, and all four specimens of the fresh BOFS mixture were broken at 9 days, which showed 0.433% expansion. This result was attributed to the fact that f-CaO and f-MgO existing in the fresh BOFS mixture reacted with H_2O and formulated $\text{Ca}(\text{OH})_2$ and $\text{Mg}(\text{OH})_2$. These hydration reactions increased the volume, and additional stresses appeared in the samples. These stresses eventually led to crack derivation and breakage of the fresh BOFS mixture matrix [40,62].

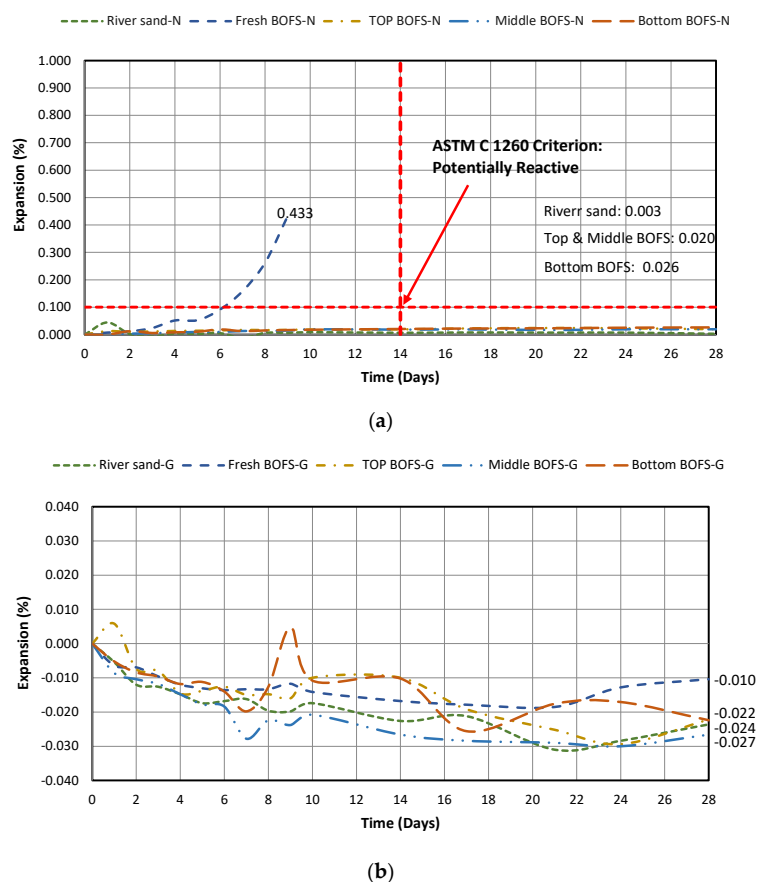


Figure 6. Expansion behavior of (a) normal OPC mixtures and (b) geopolymer mixtures submerged in the water.

Interestingly, all geopolymer mixtures showed no expansion regardless of aggregate type, even for the fresh BOFS mixture (Figure 5b). It might be explained by the fact

that free oxides (f-CaO and f-MgO) in the BOFS reacted with free silica that came from AAS and formed stable calcium silicate compounds (CaSiO_3 and MgSiO_3). Therefore, geopolymerization techniques eliminated expansion in the fresh BOFS mixture. Even though there were some fluctuations in the graph, the top, middle, and bottom BOFS mixtures had similar expansion patterns and low expansion behavior. These fluctuations occurred due to negative expansion, i.e., shrinkage. Shrinkage under water-submerged conditions might be induced by the difference in the physical and chemical properties of the reactant and the product of the geopolymerization reaction such as porosity, density, chemical bonding condition, etc. [63].

4.2.2. Expansion in 1 M NaOH Solution

Figure 7 illustrates the results of the accelerated mortar bar expansion test in 1 M NaOH solution using the ASTM C 1260 accelerated mortar bar test method. The C 1260 is a classic test method and one of the most commonly used methods because the test result can be obtained within as few as 14 days. Aggregate having a mean bar expansion of 0.1% or less at 14 days is considered non-reactive in the alkali-silica reaction. Between 0.1 and 0.2%, the aggregate is indicative of potentially reactive. Aggregate with a larger than 0.2% expansion is considered reactive. As presented in Figure 7a, irrespective of aggregate type, the expansion of all OPC mixtures was between 0.1 and 0.2% at 14 days, which classified potentially reactive aggregate. When the test period was extended to 28 days, the expansion of all OPC mixtures reached even more than 0.2%, which meant reactive aggregate for the alkali-silica reaction (ASR) and indicated inadmissible volume expansion.

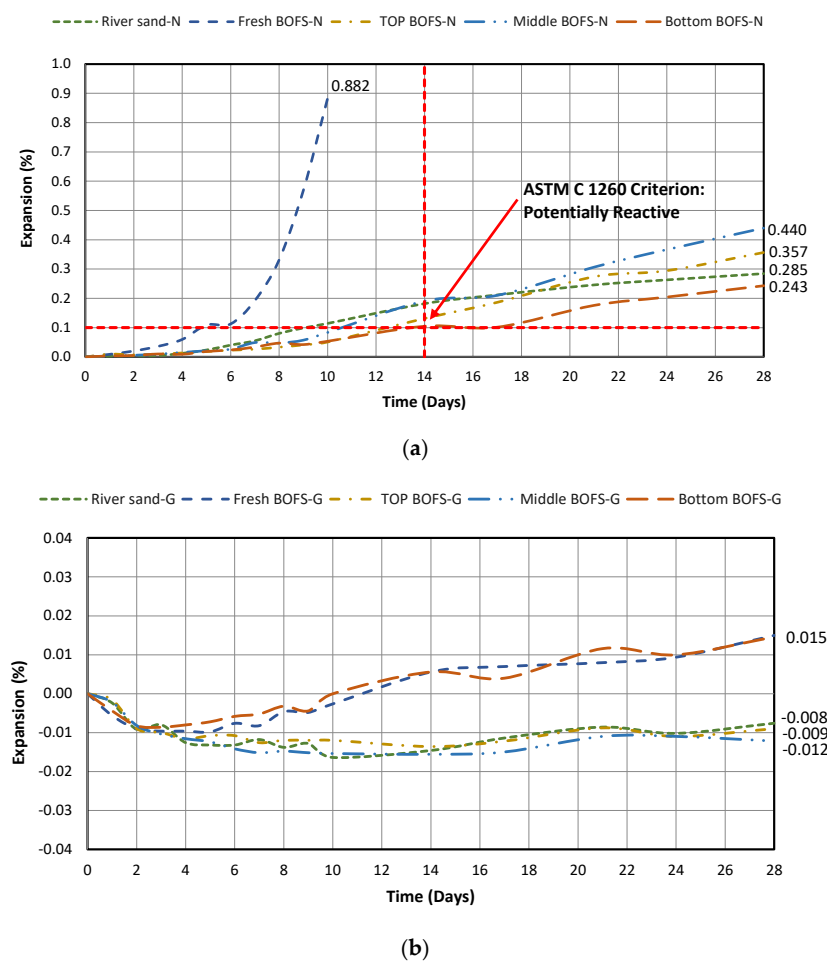


Figure 7. Expansion behavior of (a) normal OPC mixtures and (b) geopolymer mixtures submerged in 1 M NaOH solution.

The following expansion might have been caused by more Ca(OH)_2 and ASR gel formation. Since abundant OH^- ions in 1 M NaOH solution were available, these ions reacted with calcium ions (Ca^{2+}) released from the BOFS, resulting in more Ca(OH)_2 formation and volumetric instability in the normal OPC mixture matrix.

Moreover, it is well known that ASR is initiated as the attack of the surface layer of aggregate by hydroxyl ions (OH^-), and silanol groups (Si-OH) on the aggregate surface are broken down by OH^- into SiO^- molecules. The released SiO^- molecules attract alkali cations (Na^+ or K^+) in pore solution with Ca(OH)_2 , forming an alkali-silica gel around the aggregate. The alkali-silica gel takes in water, expanding and exerting an osmotic pressure against the surrounding paste or aggregate. When the expansionary pressure exceeds the tensile strength of the cementitious material mixture matrix, cracks on/in the mixture matrix occur [64,65]. Therefore, Ca(OH)_2 and high OH^- ion concentrations are necessary to induce ASR expansion. It should be noted that the BOFS aggregate contains silica components, irrespective of BOFS type (Table 2). The continuous formation of Ca(OH)_2 due to the reaction of f-CaO and water and the high concentration of 1 M NaOH solution accelerate the ASR of normal OPC mixtures with river sand and BOFS aggregate. It is also mentioned that siliceous river sand used in this study was classified as potentially reactive and had 0.19% at 14 days.

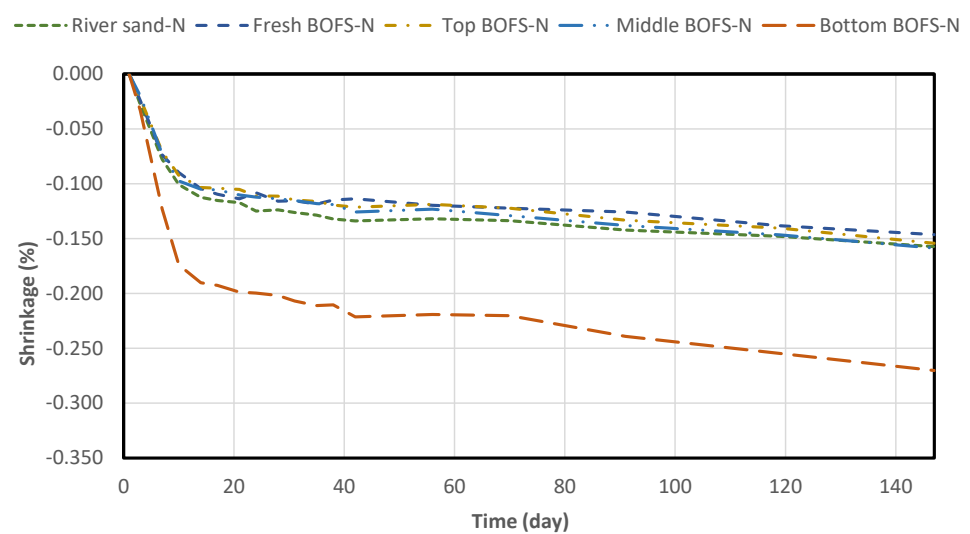
Oppositely, all geopolymer mixtures showed slight expansion, although the mixture containing fresh BOFS aggregate had relatively higher expansion than the other mixtures. As stated previously, silicon (Si) released from the geopolymer mixture matrix reacted with f-CaO and f-MgO in BOFS aggregate and formed stable products (CaSiO_3 and MgSiO_3), eliminating the expansion due to the formation of Ca(OH)_2 .

Moreover, controlling ASR expansion in geopolymer mixtures containing siliceous river sand and BOFS aggregates can be explained as follows: in the geopolymer mixtures made of FFA and GGBFS, the majority portions of alkalis in pore solution that include alkalis coming from 1 M test solution are consumed by geopolymerization process to convert them to cementitious binders. This is called the dilution effect of alkali concentration and fixation of alkali to the hydration process. Therefore, only the remaining alkalis participate in forming the ASR gel, which is definitely less than those of normal OPC mixtures [66,67]. Furthermore, reactive silica components in both sand and BOFS aggregate also participate in the geopolymerization process, which causes the engagement of alkalis in binder and pore solution and silica compounds. This reaction produces non-expansive lime-silica gel. Therefore, applying geopolymer technology for the mixture containing BOFS aggregate seems to have a positive effect on controlling ASR expansion because none of the geopolymer mixtures exceed the threshold value of ASR based on the C 1260 test.

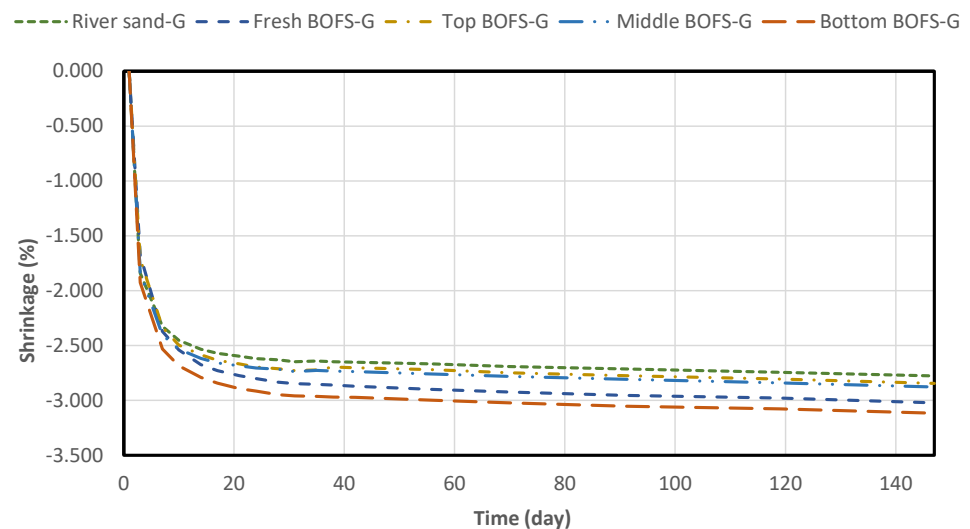
4.3. Drying Shrinkage and Mass Change

Drying shrinkage, specifically at an early stage, is one of the critical factors affecting concrete durability [68]. It is a time-dependent deformation caused by water loss from capillary pores due to hydrostatic tension and might induce critical crackings in the material matrix [69,70]. Figure 8 shows the drying shrinkage results of normal OPC mixtures (a) and geopolymer mixtures (b). The drying shrinkage of normal OPC mixtures occurred sharply for the first 2 weeks and then proceeded gradually until 40 days. After that, the graph trend gradually stabilized and achieved maximum change, which was approximately -0.156% at 147 days of testing. Among all normal OPC mixtures, the bottom BOFS mixture had the highest shrinkage, which was about two times more than the other OPC mixtures. It should be noted that the bottom BOFS aggregate had a more extended aging period. As a result, it became more porous, contained higher amounts of calcite (CaCO_3), and had a high water absorption capacity. These factors caused a higher drying shrinkage than that of the other mixtures. This result was supported by the low compressive strength of the bottom BOFS mixture, which had lower density and more pores. This explained the higher weight change in the bottom BOFS mixtures. Since more internal water evaporated, significant weight loss was observed from the graph.

Figure 8b illustrates the drying shrinkage of geopolymer mixtures. The shrinkage rate in the geopolymer mixtures was relatively high during the early ages, approximately up to 28 days, and after this age, the drying shrinkage rate gradually went down. By the end of the testing period, regardless of BOFS type, all geopolymer mixtures shrank approximately at similar levels, ranging from 2.77 to 3.11%. In fact, the evaporation of internal free water in the mixture under environmental conditions caused drying shrinkage that was dominated by macroporous and mesoporous structures in the mixture. Free water from the macropores first evaporated due to the low capillary pressure (tension) inside pores, which corresponded to a significant and rapid initial drying shrinkage lasting up to 10 days [71,72]. These characteristics were matched with test results in Figure 8b. Since the continuous water evaporation led to less water in the macropores, the water in the mesopores started to evaporate gradually. After 10 days, the drying shrinkage behavior in geopolymer mixtures was connected to continuous contraction due to significant capillary pressure.



(a)



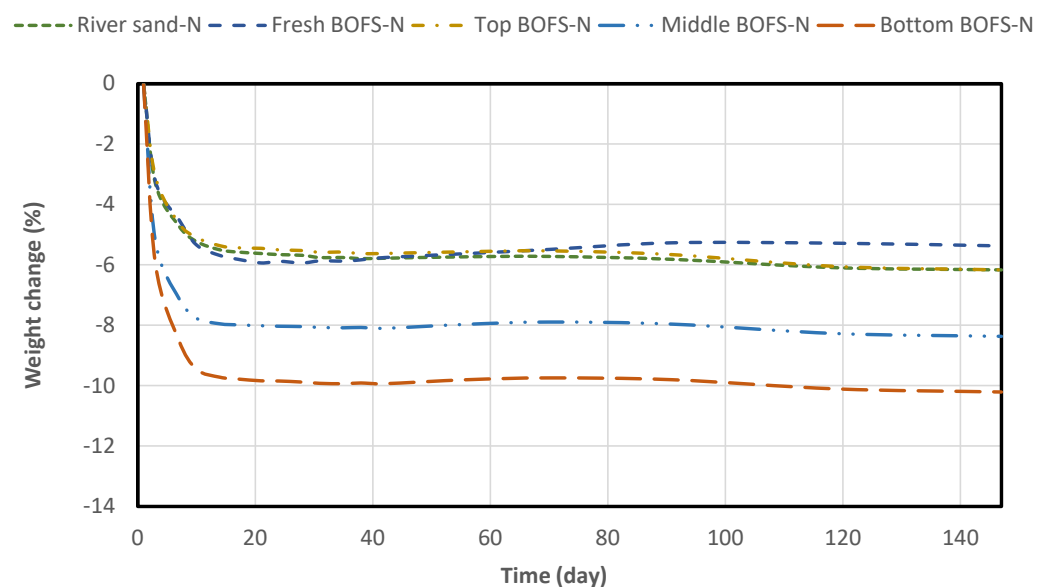
(b)

Figure 8. Drying shrinkage of (a) normal OPC mixtures and (b) geopolymer mixtures.

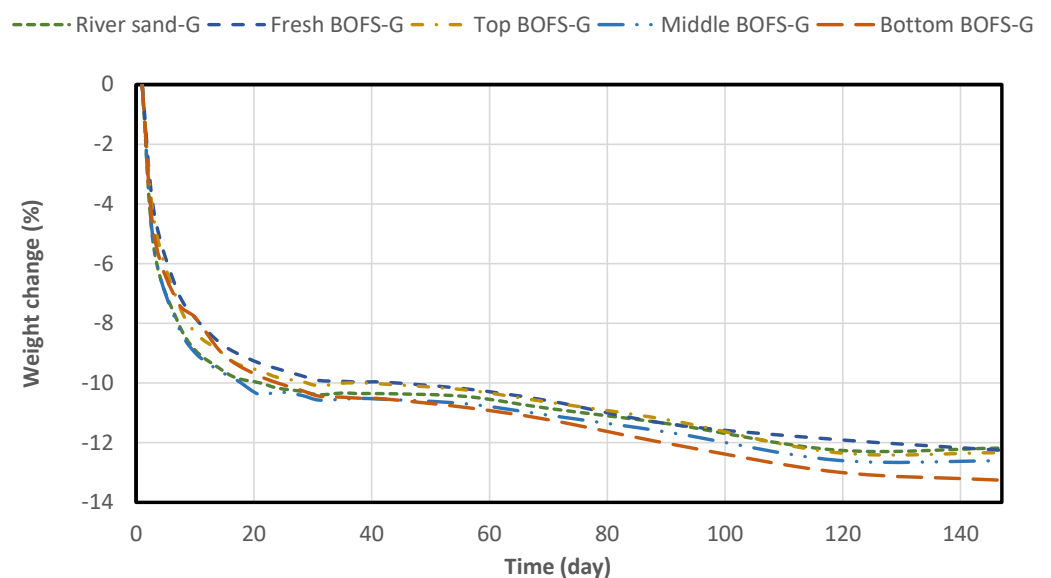
Generally, compared with normal OPC mixtures, geopolymer mixtures had significantly higher shrinkage, which is typically one of the main drawbacks of air-cured geopolymer mixtures at room temperature [73]. This drawback warrants exploration, especially

in assessing its implications for practical engineering. The early-stage chemical shrinkage also notably contributed to the geopolymer mixture's overall drying shrinkage, requiring careful consideration in real-world engineering scenarios. Therefore, sealing the surface of the specimen using an impervious plastic sheet, applying a curing compound, or using wet covering at an early age may reduce such a large shrinkage in the geopolymer mixture.

Figure 9 displays the weight change of normal OPC mixtures (a) and geopolymer mixtures (b) during drying shrinkage. Compared with geopolymer mixtures, normal OPC mixtures sharply lost weight in the first 10 days, after which it stabilized. Contrariwise, geopolymer mixtures showed rapid weight loss up to 28 days and then had gradual weight loss up to the end of the testing period. Interestingly, similar to the shrinkage behavior, the normal OPC mixture with bottom BOFS aggregate displayed the highest weight loss, reaching about 10% at 147 days. However, this result was less than the weight loss of all geopolymer mixtures that had approximately analogous values of 13%.



(a)



(b)

Figure 9. Weight change of (a) normal OPC mixtures and (b) geopolymer mixtures.

4.4. Scanning Electron Microscopy (SEM) Analysis

Figure 10 presents scanning electron microscopy (SEM) images of a normal OPC mixture containing bottom BOFS aggregate, which was submerged into 1 M NaOH solution at 80 °C. Solid crystallized products were identified with strong Mg and Ca peaks followed by minor Si, Fe, Mg, K, C, and Na peaks from the energy dispersive X-ray spectroscopy (EDS) analysis, among other complex mineralogical compositions. As stated previously, during the hydration process, the reaction of free calcium oxide (f-CaO) with water induced the formation of $\text{Ca}(\text{OH})_2$, leading to excessive expansion and cracks in the specimen. Therefore, many $\text{Ca}(\text{OH})_2$ formations having plate-like hexagonal shape crystal structures were observed. Moreover, rosette-type ASR products were also identified [74]. The EDS analysis clearly showed that these ASR products had Na, Ca, and Si compositions. The formation of ASR gel in the normal OPC mixture with bottom BOFS aggregates corresponded to the expansion characteristics in Figure 6a. Also, BOFS aggregates having higher iron oxide (FeO or Fe_2O_3) contents supported by element analyses based on EDS were observed.

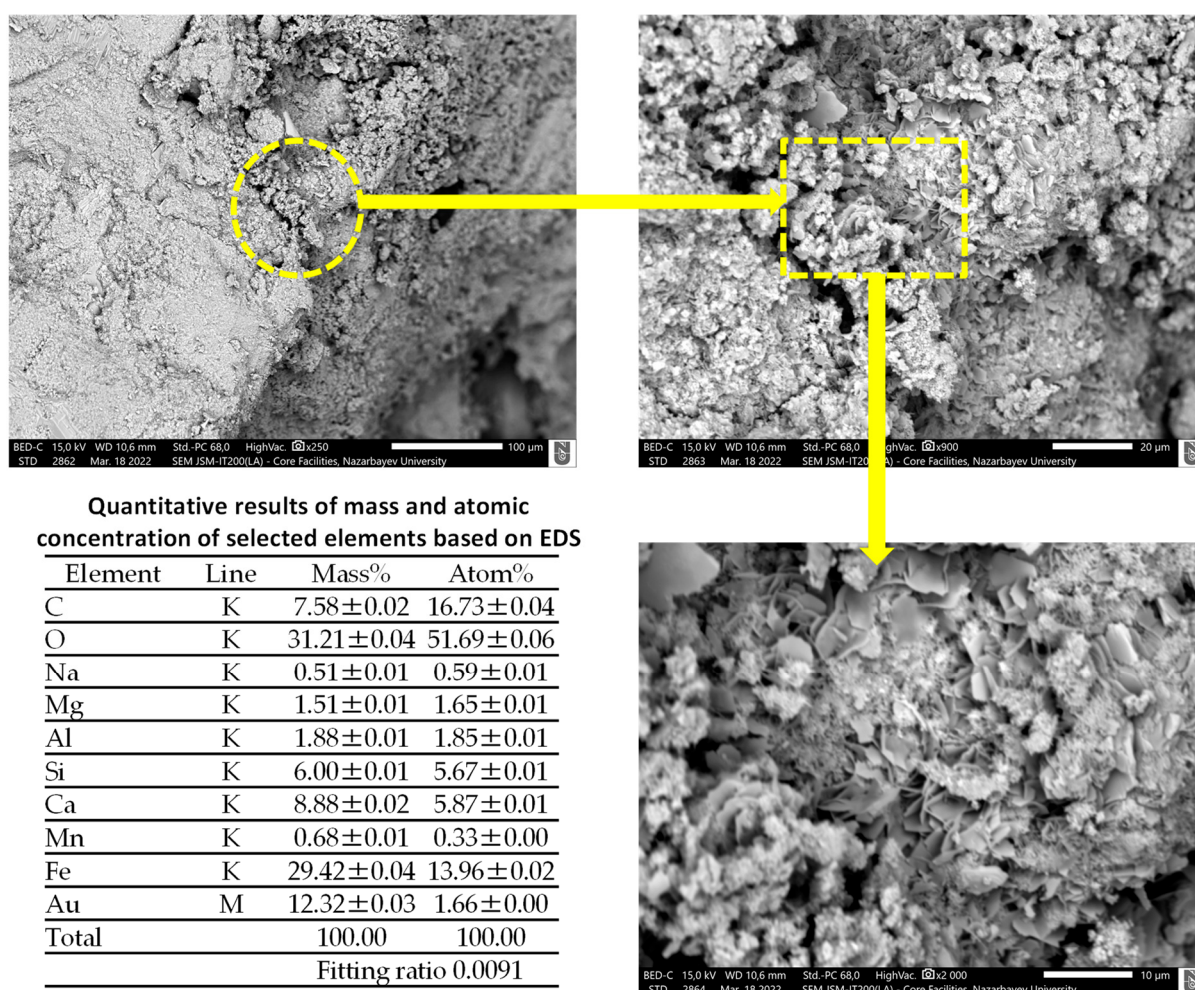


Figure 10. SEM/EDS images for fracture surface of normal OPC mixture containing bottom BOFS aggregate submerged into 1 M NaOH solution.

The SEM images of the geopolymer mixture with bottom BOFS aggregate submerged into 1 M NaOH solution at 80 °C are shown in Figure 11. In a geopolymer mixture with the same bottom BOFS aggregate, ASR products were absent or slightly present. The dense structure of calcium silicate hydrate (C-S-H) and calcium aluminate silicate hydrate (C-A-S-H) were identified instead of $\text{Ca}(\text{OH})_2$ and ASR gel. Fernandez-Jimenez and Puertas [75] reported that most alkalis are fixed in the geopolymerization products as C-S-H or C-A-S-H with low Na and

Al in the geopolymer mixture. These products are characterized by a low calcium to silica ratio (C/S) and high alumina content, providing low expansion characteristics. The EDS analysis clearly supports these hydration products. This SEM/EDS analysis corresponds to the low expansion characteristics of geopolymer mixtures presented in Figure 7b.

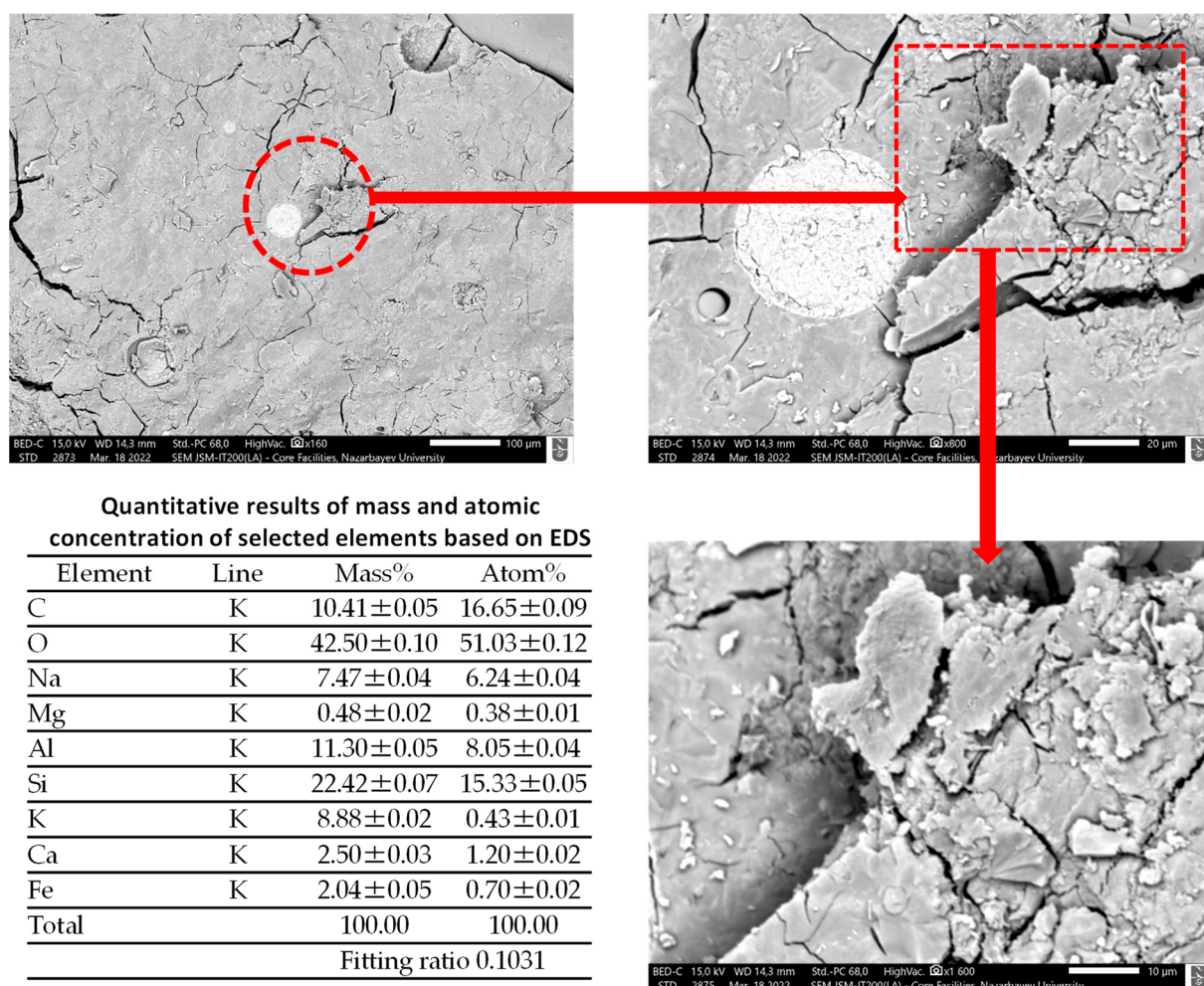


Figure 11. SEM/EDS images for fracture surface of geopolymer mixture containing bottom BOFS aggregate submerged into 1 M NaOH solution.

5. Discussion

This study examined the feasibility of chronologically aged BOFS as an aggregate component in the geopolymer mixture. BOFS mainly consists of CaO, SiO₂, Al₂O₃, FeO, and MgO with other minor components. The oxide chemical compositions of BOFS are highly varied and change from batch to batch in the same plant depending on raw materials and furnace conditions. The variation range of the components in BOFS is 30–55% of CaO, 8–20% of SiO₂, 1–7% of Al₂O₃, 10–35% of FeO, and 5–15% of MgO [37,41,76]. In this study (Table 1), regardless of age, all used BOFS aggregates had weight percentages for major components similar to those of previous studies.

Moreover, BOFS had similar mineral compounds to raw cement, such as dicalcium silicate (C₂S), tricalcium silicate (C₃S), tetracalcium aluminoferrite (C₄AF), and other mineral compounds such as olivine [(Mg,Fe)₂(SiO₄)], merwinite [Ca₃Mg(SiO₄)₂], wüstite (FeO), CaO · Fe₂O₃, the RO phase (CaO-FeO-MnO-MgO), and CaO. Although C₂S, C₃S, and C₄AF compounds in BOFS mainly contribute to the cementitious properties, BOFS's crystalline structure due to the slow cooling solidification process makes BOFS less reactive than OPC [37,76]. The fresh BOFS aggregates used in this study seemed similar to those in the

previous studies, but all chronically aged BOFS aggregates also present CaCO_3 due to the natural mineral carbonation process.

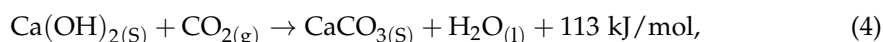
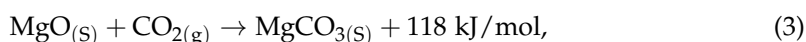
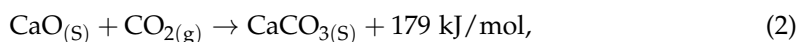
Furthermore, the presence of f-CaO and f-MgO in BOFS is more susceptible to expansion [41]. The residual free lime from the raw material and precipitated lime from the molten slag due to the injection of high-pressure oxygen are the primary resources, while the over-burned slag is attributed to f-MgO. As stated earlier, the presence of potentially high amounts of f-CaO and f-MgO limits the utilization of BOFS as an aggregate source in OPC concrete: f-CaO and f-MgO react with water at a low hydration rate and induce Ca(OH)_2 and Mg(OH)_2 , leading to significant volume expansion [77–79].

Figure 12 presents normal OPC mixtures containing fresh BOFS aggregate submerged in water at 80 °C. As explained above, due to Ca(OH)_2 formation, localized expansion occurred on the surface and inside OPC mixtures, making them crack. This visual observation of specimens matched the expansion results in Figure 6a.



Figure 12. Normal OPC mixtures containing fresh BOFS aggregate submerged in water at 80 °C.

However, chronically aged BOFS aggregates used in this study naturally suffered from the mineral carbonation process. This is an emerging thermochemical solid-looping process that indicates the reaction of CO_2 with metal oxide-bearing materials to form insoluble carbonates, with calcium and magnesium being the most attractive metals [43,80]. The volume instability of BOFS due to its inherent high f-CaO, f-MgO, or Ca(OH)_2 content in the presence of water and the oxidation of the metallic content was minimized by the formation of thermodynamically stable and insoluble products (CaCO_3 or MgCO_3) as shown in Equations (2)–(4). The XRD analyses presented in Figure 3 support the mineral carbonation of all chronically aged BOFS aggregates.



As presented in Figure 6a, all OPC mixtures containing chronically aged BOFS aggregates showed less than 0.03% expansion. This proved that the f-CaO, f-MgO, or even Ca(OH)_2 component might be converted to CaCO_3 or MgCO_3 , which was innocuous and stable to the volume expansion.

Using BOFS aggregate in the geopolymer mixture also can minimize the volume instability of the BOFS. During the geopolymerization process, Si gel and Al gel are produced on the solid particle surface, and thus, they form the Si-O-Al framework. SiO_4 and

AlO_4 tetrahedra are linked to each other by sharing all O_2 atoms. These exothermic and complex geopolymer reactions contain large amounts of free silicon (f-SiO_2). This f-SiO_2 can react with f-CaO and f-MgO in the BOFS aggregate, and hence, the formation of stable compounds such as wollastonite (CaSiO_3) and enstatite (MgSiO_3) takes place, thereby inhibiting the expansion of BOFS aggregates as shown in Equations (5) and (6) [44,81].

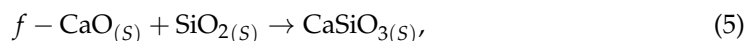


Figure 13 presents geopolymer mixtures containing fresh BOFS aggregate submerged in water at 80°C . The visual observation of specimens does not show any cracks on the surface of the specimens. This result corresponds to no expansion, illustrated in Figure 6b. As explained above, the reaction between f-SiO_2 , f-CaO , or f-MgO forms a stable CaSiO_3 or MgSiO_3 to prevent volumetric expansion in the geopolymer mixture. Moreover, although the geopolymer mixtures containing fresh BOFS aggregate have relatively higher expansion than the other mixtures, all geopolymer mixtures show slight expansion even when all mixtures are submersed in 1 M NaOH solution at 80°C . Therefore, using mineral carbonation and geopolymer technology together can provide the synergistic effect on minimizing the volumetric expansion of BOFS due to the formation of Ca(OH)_2 or Mg(OH)_2 and expand the utilization of BOFS as one of the ingredients in the concrete mixture.



Figure 13. Geopolymer mixtures containing fresh BOFS aggregate submerged in water at 80°C .

6. Conclusions

This study investigated the principal differences between normal OPC and geopolymer mixtures with chronologically aged BOFS aggregates. Several fundamental properties, such as compressive strength, expansion, and drying shrinkage behavior, were investigated. Based on the test results, the following findings can be drawn:

- Generally, geopolymer mixture samples showed higher compressive strength. However, it was affected by many factors, such as the appropriate ratio of GGBFS to FFA and AAS concentration, etc. The mixtures with aged BOFS aggregate had lower compressive strength regardless of mixture type. It might be explained by the fact that the free oxides in the aggregate were already consumed and formatted portlandite that was not involved in strength development but might produce expansion in the mixture.
- Regardless of the test conditions (water and 1 M NaOH submersion at 80°C), a normal OPC mixture containing fresh BOFS aggregate demonstrated the highest expansion compared with the mixtures with chronologically aged BOFS aggregates since fresh

BOFS had the highest amount of free oxides and reactive silica that could induce the formation of $\text{Ca}(\text{OH})_2$ and ASR gel.

- Implementing geopolymerization technology can reduce the expansion behavior under both water and 1 M NaOH test conditions. Even the expansion of the mixture containing fresh BOFS aggregate mixture can be stabilized.
- The drying shrinkage and weight loss of the geopolymer mixture were more intensive than those of the normal OPC mixture, irrespective of aggregate type. In geopolymer mixtures with BOFS aggregate, aging duration did not significantly affect the reduction in drying shrinkage.
- SEM/EDS analysis supported the expansion characteristics of the mixtures containing BOFS aggregate.

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