https://doi.org/10.46810/tdfd.1569404



Investigation of Waste Mineral Wool in Geopolymer Production

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(Received: 17.10.2024, Accepted: 03.12.2024, Online Publication: 30.12.2024)

Keywords Geopolymer, Alkaline activation, Mineral wool, Rock wool, Glass wool Abstract: Mineral wools are widely used insulation materials in the construction industry; however, their non-recyclable nature poses an environmental challenge. In this study, mineral wool wastes were sustainably utilized by grinding them into powder and activating them with Na₂SiO₃ and NaOH solutions. During the production process, different silica modulus ratios of Na₂SiO₃ (2.0, 2.5, and 3.0) were examined, and the optimal ratio was determined to be 2.5. The mechanical properties of the samples were evaluated after curing at various temperatures (25°C, 50°C, 75°C, and 100°C), with the maximum compressive strength of 59.2 MPa observed in glass wool samples. Thermal curing enhanced compressive strengths of the samples stabilized after a curing period of 90 days. These findings demonstrate the feasibility of recycling mineral wool wastes into high-performance materials and highlight the significant role of thermal curing in enhancing mechanical properties.

Atık Mineral Yünlerin Jeopolimer Üretiminde Kullanımının İncelenmesi

| Anahtar Kelimeler | Öz: Mineral yünler inşaat sektöründe yaygın olarak kullanılan yalıtım malzemeleridir; ancak | | | | |
|---------------------|--|--|--|--|--|
| Jeopolimer, | geri dönüştürülemeyen yapıları çevresel bir sorun teşkil etmektedir. Bu çalışmada, mineral | | | | |
| Alkali aktivasyonu, | yün atıkları toz haline getirilip Na2SiO3 ve NaOH çözeltileri ile aktifleştirilerek sürdürülebilir | | | | |
| Mineral yün, | bir şekilde değerlendirilmiştir. Üretim sürecinde farklı Na2SiO3 silika modülü oranları (2.0, | | | | |
| Taş yünü, | 2.5 ve 3.0) incelenmiş ve optimum oran 2.5 olarak belirlenmiştir. Numunelerin mekanik | | | | |
| Cam yünü | özellikleri çeşitli sıcaklıklarda (25°C, 50°C, 75°C ve 100°C) kürlendikten sonra | | | | |
| | değerlendirilmiş ve en yüksek basınç dayanımı 59,2 MPa ile cam yünü numunelerin | | | | |
| | gözlenmiştir. Termal kürleme, cam yünü bazlı numuneler için özellikle 75°C'de bas | | | | |
| | dayanımını artırmıştır. Ayrıca, numunelerin basınç dayanımları 90 günlük bir kürleme | | | | |
| | süresinden sonra stabilize olmuştur. Bu bulgular, mineral yün atıklarının yüksek | | | | |
| | performanslı malzemelere geri dönüştürülmesinin uygulanabilirliğini göstermekte ve termal | | | | |
| | kürlemenin mekanik özelliklerin geliştirilmesindeki önemli rolünü vurgulamaktadır. | | | | |

1. INTRODUCTION

Ordinary cement production is a main contributor to CO₂ [1]. An alternative activity to reduce CO₂ emission from ordinary Portland cement production is geopolymerbased binder materials development [2]. Geopolymerization is the dissolution of aluminosilicate in an alkaline condition, and result a three-dimensional network between amorphous and semi-crystalline [3]. Sources of aluminosilicate can be natural [2-4] or industrial by product [5-7]. NaOH, Na₂SiO₃, KOH and K₂SiO₃ are mainly used as alkaline solutions. In general, geopolymers show better mechanical performance [8, 9]. Buildings account for about 33% of global energy consumption and about 30% of CO₂ gas, in addition approximately than 1/2 of buildings' energy consumption is by building heating and cooling [10]. So there are an interest in finding and production of new binding materials containing Phase Change Materials to improving and decreasing energy consumption. There are some studies on these topics [11-13]. The conversion of waste and byproduct materials to cementing materials

helps conserving the environment and existing resources. Geopolymers considered as principal alternative to ordinary cement and can significantly reduce harmful gas emissions and greatly reduce the high energy consumption in cement industry [3, 14, 15]. Geopolymers can be prepared from aluminosilicate-based or kaoliniterich industrial waste materials with an alkaline solution [16, 17]. Geopolymers show better mechanical performance [18-20]. In cold countries, structures are exposed to frost. This is a major problem in terms of durability [21, 22]. The effect of freeze-thaw cycles on the mechanical properties of cementitious and some geopolymer binders has been studied in many investigations [23-27]. Rock and glass wools are the most used insulation materials in the building industry [28]. These wastes are produced by demolition. European countries produced 2.3 million tons waste mineral wool and will increase to 2.5 million tons by 2020 [29]. Rock and glass wool wastes are non-recyclable material. Since these natural or industrial geopolymer base materials can be used in many industrial by-product materials, CO2 emission reductions of up to 80% compared to Portland cement can be achieved [30]. Mineral wools have acceptable chemical composition for geopolymerization (Table 1).

 Table 1. Rock and glass wool Chemical composition

 [31]

| Component | SW New | SW Old | GW New | GW Old |
|--------------------------------|--------|--------|--------|--------|
| | [%] | [%] | [%] | [%] |
| CaO | 18.2 | 16.6 | 7.9 | 7.3 |
| SiO ₂ | 39.4 | 44.1 | 61.3 | 61.9 |
| Al ₂ O ₃ | 15.9 | 14.3 | 2 | 3.3 |
| Fe ₂ O ₃ | 9.8 | 5.5 | 1.4 | 1.2 |
| Na ₂ O | 1.3 | 1.2 | 16.3 | 16 |
| K ₂ O | 0.5 | 0.3 | 1 | 0.8 |
| MgO | 11.4 | 14.7 | 2 | 2.9 |
| P_2O_5 | 0.1 | 0 | 0.2 | 0 |
| TiO ₂ | 1 | 0.2 | 0.1 | 0 |
| SO ₃ | 0.1 | 0 | 2 | 0.3 |
| Cl | 0 | 0 | 0.1 | 0.1 |
| LOI 550°C | 4.3 | 2.6 | 9.4 | 8.8 |

In terms of amorphous feature which increases their reactivity, they are completely amorphous and this is seen with the help of X-ray and XRD results [32, 33]. This study justifies the feasibility of rock and glass wool as geopolymer base material. UPV and compressive strength tests results provided main measurement for this feasibility.



Figure 1. Comparison of chemical compositions of various alkali binder based materials [34]

The chemical compositions of mineral wool compared to other alkali active binders are shown in Figure 1.

In the study by Weil et al. two geopolymer mixtures were prepared to examine the CO₂ emissions that cause global warming. At the end of this study, they stated that geopolymers derived from fly ash and slag emitted less CO₂ than normal cement [35]. Energy consumption in geopolymer cements production is approximately 40% less than normal Portland cement [36]. It is stated that the amount of Si/Al is important factor in the geopolymer production [37]. Torgal et al. [38] reported that the main materials that can be activated with alumina and silicate based alkalis are kaolinite clay, metakaolin, combining of fly ash and metakaolin in different ratios, combining of slag and metakaolin in different ratios and mixtures of slag and red mud. They conducted Ca-Si and Ca-Al based experiments on these materials and measured the hydration development with XRD and infrared rays. Kong et al [39] exposed metakaolin and fly ash to high temperatures together and made them more active. They stated that this was due to the high amount of alumina and silica in fly ash. They determined that metakaolin had an amorphous structure at high temperatures (approximately 800°C) and transformed into an activated aluminosilicate. The effects of aggregate, plasticizer and temperature on geopolymer cement were investigated in the study. It was stated that as the sample sizes increased, the compressive strength decreased due to thermal cracks. If the aggregate grain diameters were smaller than 10 mm, the shelling was more common. It was thought that this phenomenon could be prevented if it was larger than 10 mm. It was stated that the superplasticizer additive reduced the strength in geopolymer concretes and did not have a significant contribution to the total workability.

In the study conducted by Malolepszy 2009, it was stated that Na₂CO₃ is suitable for activating slags containing large amounts of C₂MS (M: alkali metal). It was stated that NaOH is a good activator for slags containing large amounts of C2AS. The activation of different systems with NaOH, Na₂CO₃ and Na₂OSiO₂ was investigated by Krivenko (1992). It was stated that Na₂SiO₃ (sodium silicate or glass water) is a very effective activator [40]. Allahverdi et al., [41] prepared geopolymer cement using pumice type natural pozzolan around Taftan Mountain and combinations of NaOH and Na₂SiO₃ as activators. Three different silica moduls were prepared by adding sodium hydroxide to sodium silicates. Three different geopolymer cement systems were formed with sodium oxide contents weight. Water/cement ratio was taken as 0.36, 0.40 and 0.44. As a result of the study; they stated that Taftan pozzolan can be activated by using NaOH and Na₂SiO₃ in appropriate proportions; it can be converted into geopolymer cement formation providing appropriate workability and 28-day compressive strength of 63 MPa. It was explained that natural pozzolans can be activated and geopolymer cement can be produced by using a mixture of sodium silicate and sodium hydroxide in certain proportions as alkali activators. In the literature, early strength, acid resistance, sulfate behavior, shrinkage of geopolymers has been investigated, especially on fly [42-46]. Energy consumption ash. in the

geopolymerization is almost 40% less than the energy needed ordinary Portland cement [36].

Atis et al. [47] investigated the use of a new binder that would activate slag without using Portland cement in their studies. Compressive strengths, flexural tensile strengths were measured and drying shrinkage in a 6-month period was examined. At the same time, the hydration development of the samples was examined. It was stated that the setting start and end times were earlier in liquid sodium silicate and sodium hydroxide-activated cements compared to normal Portland cement, and the cements activated with sodium carbonate were the same as normal Portland cement. It was stated that with the increase in the silica modulus, the effect of liquid sodium silicate on gaining final strength and flexural tensile strength was higher. It was stated that the mortars produced with sodium silicate and sodium hydroxide-activated slags were more brittle, and the behavior of the mortars produced with sodium carbonate was similar to normal Portland cement.

Komljenovic et al [48] investigated the microstructure properties of fly ash (Class F) geopolymers. They stated that the most important parameters in the alkali activation method are activator properties and density, while the important parameter in fly ash is fineness. They stated that the compressive strength of fly ash geopolymers (<43µm) is generally high. The best results were obtained by using sodium silicate solution. It was stated that the compressive strength is largely dependent on the Si/Al ratio. Anuar et al [49] used NaOH and Na2SiO3 mixed as alkaline liquid in their studies. In this study, geopolymer concrete samples were used in two different molars (8M and 14M sodium hydroxide NaOH). 3, 7, 14, 21 and 28 day compressive strengths were tested in laboratory conditions. They stated that the best result for compressive strength was obtained by 14M NaOH.

A major environmental challenge for the construction industry is the non-recyclability of mineral wool waste. In recent years, efforts have focused on recycling these wastes and developing environmentally friendly binder systems. This study aims to transform mineral wool waste (glass wool and rock wool) into sustainable materials. The waste materials were processed using the alkaline activation method and investigated at different silica modulus ratios (2.0, 2.5, and 3.0). Additionally, the effects of thermal curing conditions (25°C, 50°C, 75°C, and 100°C) on the mechanical properties were examined. The primary objective of the study is to identify the optimal parameters for converting mineral wool waste into high-performance materials. The findings not only contribute to sustainable material design but also provide an environmentally friendly solution for addressing the issue of mineral wool waste.

2. MATERIAL AND METHOD

2.1. Material

Mineral wools were obtained from waste mineral wools that had completed their service life at Bingöl University education facilities. These mineral wools were ground in the Los Angeles Device (Figure 2) and then made ready for use with the help of a ring grinder.



Figure 2. Grinding stages of mineral wool

Mineral wools consisting of rock wool and glass wool were ground and made ready for use in geopolymer production (Figure 2).

2.2. Alkaline Solution Production Method

According to the results obtained from the preliminary experiments, different mixtures of glass water and sodium hydroxide solutions were used for alkaline solution production in this study and project. In alkaline solution production, Na₂SiO₃/NaOH mixtures were prepared at 3 different ratios. Compressive strength and UPV tests were carried out for geopolymers produced at three different ratios. Our Na₂SiO₃/NaOH ratios were selected as 3, 2.5 and 2 (Figure 3).



Figure 3. Produced samples (a) rock wool, (b) glass wool

2.3. Experimental Procedure

The compressive strength of the geopolymer composites after curing was determined according to ASTM C109. UPV was performed according to the principles specified in ASTM C597-16. Experiments were carried out with three specimens from each mixture group. The average of 3 sample results for each mixture group was used.

3. RESULT AND DISCUSSION

3.1. Compressive Strength and Ultrasonic Pulse Velocity Tests

Compressive strength and ultra sound tests were performed on samples produced from glass wool and rock wool. The material and molarity used were taken into account when coding the sample. For example, when coding for C3, the first letter of the glass wool in the mixture and the molarity ratio were used. Our samples were produced in 3x3x3 cm³ molds and their 3, 7, 14, 28 and 90 day compressive strengths and UPV values were measured. The 1-day compressive strength of our samples was determined to be very high. According to the results, it was determined that the samples produced from glass wool were more advantageous in geopolymer production. In other words, higher strength geopolymer samples can be produced by using glass wool. Considering this situation, the experiments were continued on glass wool. The compressive strength and UPV test results related to glass wool are presented in Figures 7, 8. In all three molarity cases, the one-day compressive strengths of the samples were higher than 60 MPa. However, in the 3 Molarity and 2 Molarity usage cases, the compressive strengths decreased to 30.04 MPa and 25.80 MPa, respectively.



Figure 4. Results of glass wool based samples (a) compressive strengths and (b) UPV

In the 2.5 molarity case, the compressive strength was obtained as 59.20 MPa. The 90-day compressive strengths in the C2 and C3 coded samples decreased by approximately 65% and 50%, respectively (compared to the one-day compressive strengths). Compared to the change in the C2 and C3 coded samples, the compressive strength in C2.5 was less than 10% (Figure 4a). When the UPV test results are examined; In the 90-day

measurements, there was an approximately 25% increase in the UPV value in the C2.5 coded sample, while there was a decrease of approximately 15% and 10% in the C2 and C3 coded samples, respectively (compared to the oneday UPV values). In the first 7-day measurements, an increase in UPV values is observed for all molarity conditions. However, there was a decrease in the UPV value of the 28-day sample (Figure 4b).

Compressive strength and UPV test results for rock wool are presented in Figures 5. In all three molarity cases, there was a decrease in the compressive strength of the samples up to the 7-day curing period. The 90-day compressive strengths for T3, T2.5 and T2 were determined as 21.70 MPa, 20.01 MPa and 23.40 MPa, respectively. The decrease in 90-day compressive strengths for T3 and T2.5 was approximately 40%, while it was approximately 50% for T2 (Figure 5a). When the UPV test results are examined; In the 90-day measurements, there was an approximately 3% increase in the UPV value in the T2 coded sample, while there was a smaller decrease of less than 1% in the C2 and C3 coded samples (compared to the one-day UPV values). In the first 7-day measurements, a decrease in UPV values is observed for all molarity conditions (Figure 5b).



Figure 5. Results of rock wool based samples (a) compressive strengths and (b) UPV

It is clearly seen in Figure 4a and Figure 5a that the 7 and 28-day compressive strengths of the samples did not become stable. On the other hand, when Figure 4a and Figure 5a are examined, it is seen that the compressive strengths of the samples became stable at the end of 90 days. Therefore, in the study, the 90-day compressive strengths were taken into account when determining the molarity ratio with the rock wool and glass wool to be used. When the 90-day compressive strengths are taken into account, the sample using the C2.5 coded glass wool

with 2.5 molarity gives the best compressive strength. In the continuation of the study, 2.5-molarity mixtures were prepared under three different curing temperatures (25 °C, 50 °C and 100 °C) to determine the effect of the curing temperature on the compressive strength.

In the study of Yadollahi et al. [50] it was stated that the increase in silica modulus increases the compressive strength. Similarly, in our study, it was observed that the compressive strength values obtained for 2.5 silica modulus were higher than 2 silica modulus in the samples using glass wool. However, a reverse situation was detected in the samples where stone wool was used.

3.2 Compressive Tests at Different Curing Temperatures

In the continuation of the studies, the compressive strengths of the 2.5 molarity samples produced from glass wool at 25 °C, 50 °C and 100 °C cure temperature conditions were determined. In addition, the experiments conducted at 75 °C were repeated against any doubts. The results are presented graphically in Figures 11-13. Since the necessary hardening did not occur in the samples kept in the mold at 25 °C for 24 hours, their 1-day strengths did not yield results. For this reason, their 3, 7, 14, 28 and 90-day compressive strengths were examined. However, since there was no problem in the initial cure conditions of 50 °C and 100 °C, their 1, 3, 7, 28 and 90-day compressive strengths were examined.



Figure 6. Compressive strengths of 2.5 molarity samples produced from glass wool (a) at 25-50°C and (b) at 75-100°C curing conditions

When the compressive strengths of the samples produced from glass wool under 75 °C curing conditions were examined, the highest compressive strength was reached in the 3-day samples. However, there was a decrease in the compressive strength in the 7 and 28-day samples (Figure 6b).

When the compressive strengths of the samples produced from glass wool were examined under 25 °C curing conditions, the highest compressive strength was reached in the 28-day samples. The compressive strength took its lowest value in the 3-day samples. There was an increase in compressive strength in direct proportion to the curing time (Figure 6a).

In samples produced from glass wool under 50 °C and 100 °C cure conditions, compressive strengths increased in direct proportion to the cure time. While the first day compressive strength of samples produced at 50 °C was 39 MPa, the 28-day compressive strength was determined as 65.278 MPa. While the first day compressive strength of samples produced at 100 °C was 90.945 MPa, the 28-day compressive strength was determined as 91.037 MPa. When the results were examined, it was seen that the samples produced under the initial cure conditions of 100 °C reached the best compressive strength. However, as the cure time increased, it was seen that the compressive strength of samples produced at 75 °C cure temperature was better.

Figures 6a and 6b show that curing temperature and curing time have a significant effect on compressive strength. At low temperatures (25 °C and 50 °C), the compressive strength increased up to 28 days, while a decreasing trend was observed after 28 days. At higher temperatures (75 °C and 100 °C), although the strength was high on the first day, fluctuations occurred depending on the curing time. Especially at 75 °C, the strength increased again at the end of 90 days, while at 100 °C, the strength decreased on the 3rd day and then recovered. This shows that both temperature and time-dependent chemical processes have complex effects on material properties.

4. CONCLUSION

This study demonstrated that mineral wool waste, including glass wool and rock wool, can be effectively utilized as raw materials for alkali-activated materials. The results revealed that the chemical and mineralogical composition of these wastes makes them highly suitable for alkali activation. The results are listed below:

- The mechanical properties of the specimens were significantly improved by thermal curing. The highest compressive strength (59.2 MPa) was obtained in glass wool specimens cured at 75°C. This shows that a suitable curing temperature improves the strength by increasing the density of the binder matrix.
- The effect of silica modulus ratios (2.0, 2.5 and 3.0) on mechanical properties was investigated and the optimum silica modulus ratio was determined as 2.5. This ratio balanced the amount of silica

dissolved, ensuring adequate polymerisation and increasing the homogeneity of the binder matrix.

- Glass wool samples exhibited higher mechanical properties compared to stone wool. This was attributed to the higher SiO₂ content in glass wool and a structural composition more favourable to alkali activation.
- It was observed that the compressive strength values stabilised at the end of the 90-day curing period. This shows that the long-term mechanical performance is reliable and the effects of curing time decrease with time.
- This study has shown that mineral wool waste can be utilised to produce sustainable materials for the construction industry. The recovery of wastes both reduces the environmental burden and provides an opportunity for the development of new binder systems.

Although the findings of the study prove that mineral wool wastes are applicable in the production of construction materials, their long-term durability and performance under different environmental conditions need to be investigated. Additionally, the effects of chemical and structural properties of different waste sources (e.g. old mineral wool and new mineral wool) on performance should be investigated in detail.

Acknowledgement

This work has been supported by Bingöl University Scientific Research Projects Coordination Unit under project number MMF.2020.00.001. We would like to thank BAP unit for their support.

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