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MINERALOGICAL AND PETROGRAPHICAL ANALYSIS OF COMPOSITE GLASS FIBER ADDED LIME MORTARS

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Abstract

Lime and brick dust are important and unique building materials commonly used in the construction of historical structures. Over time, different products and additives have been added to mortar mixtures to improve and modify certain characteristics. With the advancement of technology, composite materials have been developed when pure materials were insufficient, and their areas of use have expanded over time. The aim of this study is to contribute to the literature by adding materials such as glass fibers to lime mortar to create potential for new generation composite materials. The influence of these materials on the mineralogical structure of the produced mortars has been determined. Glass fiber materials exhibit high corrosion resistance and strength, making them resistant to outdoor conditions such as wind, temperature, and cold. They are widely used in the construction sector, especially in floorings. In this study, the mineralogical and petrographic analyses of lime mortar reinforced with composite glass fibers were examined. XRD analysis was performed for mineral composition and identification, FESEM analysis was used to define the morphology, microstructure, and chemical composition of the mortars, and EDS analysis was conducted to determine the chemical characterization of the mortars. As a result of these analyses, it was determined that glass reinforced mortars have a calcite mineral structure containing a high amount of Ca, C, O, Si elements, as well as Mg, Al, and small amounts of Fe, K, S elements.

Keywords: glass fiber, FESEM-EDS, lime mortars, XRD

1. Introduction

Expenses are adhesive materials used for the components of a structure to be connected and function as a whole [1]. These materials are inorganic construction materials formed by mixing and kneading aggregate materials such as sand and gravel with different proportions of clay, gypsum, lime, and cement to obtain a plastic consistency. Additionally, mortars can be created with non-cementitious materials such as lime. Horasan refers to the process of grinding and pulverizing it into a powder form and combining it with bricks, tiles, pottery, and fired clay. Horasan mortars are obtained by mixing lime and water in specific proportions. In some applications, sand is added to the mortar, while in others, pea-sized fragments like brick and tile shards are added [1-2]. To ensure compatibility with the building materials, it is necessary to know the physical and chemical contents of the mortars. In studies conducted for this purpose, the morphology of $CaCO_3$ crystals during carbonation accelerated by 95% relative humidity, 20°C temperature, and 20% and 100% CO_2 concentration in lime plasters has been examined [6]. The mineralogical composition of mortar and plaster samples was determined by XRD [4-5]. It has been stated that if the pozzolanic material reacts with a portion of lime in pozzolan-added lime mortars under atmospheric conditions, carbonation and hydration will occur [6]. It has been revealed that the hardening of lime mortars depends on the CO_2 absorbed from the air, and the presence of water vapor causes the hardening of lime through the reaction between CO_2 and $Ca(OH)_2$. Many comprehensive studies on the hardening of lime mortars with different contents have indicated that the carbonation rate ranges from 80% to 90%, and the carbonation varies depending on the mineralogical structure, texture, used additives, and lime [7].

The properties of the lime plasters taken from Ördekli Hamam in Bursa, Saray and Beylerbeyi Hamams in Edirne, Hersekzade and Kamanlı Hamams in Urla district of Izmir, and Düzce Hamam in Seferihisar district of Izmir, which belong to the Ottoman Period, were examined in a study conducted by Uğurlu and Böke [18].

As a result of microstructure analyses conducted on Horasan plasters, it has been determined that there is a strong bonding between lime and brick, and that it has a structure rich in calcium, silicon, and aluminum elements. Consequently, the reaction of amorphous substances in the brick content with lime has resulted in the identification of products such as calcium silicate hydrate (CSH) and calcium aluminate hydrate (CAH) [16, 18].

Lime mortars were produced with different building materials according to the characteristics of each period. To improve the quality of these mortars, organic or inorganic additives were added to their composition [18]. Additives mixed with binders and fillers were used to enhance the properties of the mortars and provide them with new characteristics. The addition of additives to the mortars increases their durability and performance. Thanks to these additives, the mortars gain better adhesion properties and features such as waterproofing. A study conducted on mortar samples from Istanbul's Tahtakale Hamam revealed the use of flax fibers as an additive in fine plasters [4, 6, 9, 12-13]. Another study conducted on mortar and plaster samples from the Nuruosmaniye Mosque in Istanbul determined that some of the plasters contained fiber (flax fibers) additives with content ranging from 0.2% to 1%, and it was suggested to use these plasters in restoration works [1, 8].

Plant and animal fibers have been used to improve the properties of building materials in the past. However, with the advancement of technology and the inadequacy of pure materials, composite materials have been developed and their use has become widespread. In a study conducted by Kalkan and Gündüz [12], composite mortars were produced by using natural calcium sulfate anhydrite binder material, different sized brick fragments aggregates with CL80 class hydrated lime, and adding zinc stearate, fiber, and ash as additive materials. Composite materials are a type of material where two or more different components are brought together in a way that they do not dissolve into each other, resulting in desired properties. These materials generally consist of matrix, reinforcement, and filler components. They also hold an important place as they constitute a majority of the industrial demand. Composite materials can overcome the shortcomings of the newly obtained materials [14-15].

Among the most preferred composite materials used today are glass fibers, steel fibers, and polymer fibers [17]. Glass fiber is used as an ideal reinforcement element in polymer matrix composites due to its high tensile strength, good chemical resistance, electrical insulation, and cost-effectiveness [3]. Glass fibers are advantageous in terms of cost and do not show clumping effect in the mortar. Additionally, they have high durability, which is another advantage [10]. Glass fiber is resistant to outdoor conditions and shows resistance against wind, temperature, and cold, making it widely used in the construction sector, especially in flooring [10-11].

2. Material and Method

2.1. Material

Among the high-quality composite materials, glass fibers are the most important reinforcement construction materials with high strength values [14]. Therefore, in this study, two different mortar samples were produced to determine the morphological structure of mortar by producing glass fiber-reinforced lime mortars. In the glass fiber-reinforced mortar sample (Sample 1), 30% slaked lime (hydrated lime), 17% clay lime aggregate (0-4mm), 20.8% quartz sand aggregate (0-4mm), 5% yellow sand aggregate (0-4mm), 3.6% brick chips (3mm), 3.6% fly ash, 10% glass fiber, 10% water were used. In the glass fiber-reinforced mortar sample (Sample 2), 30% slaked lime (hydrated lime), 17% clay lime aggregate (0-4mm), 20.8% quartz sand aggregate (0-4mm), 5% yellow sand aggregate (0-4mm), 1.8% brick chips (3mm), 1.8% brick powder, 3.6% fly ash, 10% glass fiber, 10% dental resin were used [13].

2.2. Method

FESEM-EDS analyzes were performed using a scanning electron microscope to examine the glass fiber reinforced lime mortar samples. Scanning electron microscope (FESEM) analysis was conducted to determine the morphology, microstructure, and chemical composition of the mortar samples, allowing the surface of the sample to be imaged by emitting high-energy electron beams onto the sample.

The FESEM analysis of mortar samples was conducted at Sakarya University SARGEM Laboratory. Energy measurement was performed to determine the X-ray emissions sent to the sample in the EDS Analysis (Scanning Electron Microscopy Analysis). The chemical characterization of the mortar components was determined by analyzing the elemental composition of the samples. The EDS analysis of the mortar samples was carried out at Sakarya University Metallurgy and Materials Engineering Laboratory. XRD analysis was applied to determine the intact characteristics and degradation morphologies in the sample specimens. In this analysis method, the mineral composition of the powdered samples was determined, allowing for the identification of unseen or unidentified minerals. The measurement of the distances between the building blocks (atoms, ions, or molecules) in the crystal lattice of minerals was performed using X-ray measurements. The XRD analysis of the mortar samples was also conducted at Sakarya University Metallurgy and Materials Engineering Laboratory [13].

3. Findings and Discussion

3.1. Scanning Electron Microscopy Analysis (FESEM-EDS) Results

At the end of 90 days, 1 cm^3 piece was taken from glass reinforced lime mortar samples produced in two different contents and FEEM-EDS images of the samples were obtained at X100, X500 magnification. In the EDS analysis of the mortar sample with glass fiber reinforcement, labeled as Sample No. 1, it has been determined that it has a calcite mineral structure containing high amounts of Ca, C, O, Si elements, as well as Mg, Al, and very small amounts of K, S, Fe elements according to the EDS result table at X100 and X500 magnifications. (Figure 1) The EDS image at X100 magnification of the mortar sample with glass fiber reinforcement labeled as Sample No. 1, the EDS analysis result table (Table 1) for the same sample, the EDS image at X500 magnification of the mortar sample labeled as Sample No. 1 (Figure 2), and the EDS analysis result table (Table 2) for the mortar sample with glass fiber reinforcement labeled as Sample No. 1 are provided.

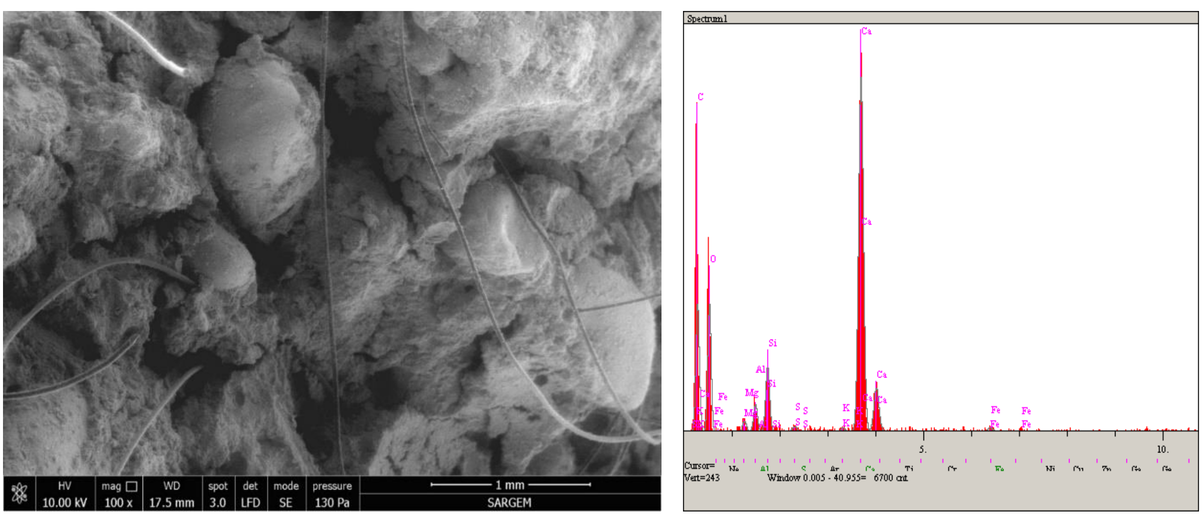

Fig. 1. FESEM-EDS images of the X100 magnified glass fiber reinforced mortar sample No.1.

Table 1. EDS analysis results table for Sample No.1 with glass fiber reinforced mortar.

Elt.	Line	Intensity (c/s)	Error 2-sig	Conc	Units	
C	Ka	70.11	5.294	17.943	wt.%	
O	Ka	65.32	5.110	38.052	wt.%	
Mg	Ka	6.12	1.564	0.685	wt.%	
Al	Ka	15.95	2.525	1.625	wt.%	
Si	Ka	40.40	4.018	3.833	wt.%	
S	Ka	3.62	1.202	0.354	wt.%	

K	Ka	2.17	0.931	0.241	wt.%	
Ca	Ka	270.64	10.401	36.253	wt.%	
Fe	Ka	2.86	1.069	1.015	wt.%	
				100.000	wt.%	Total

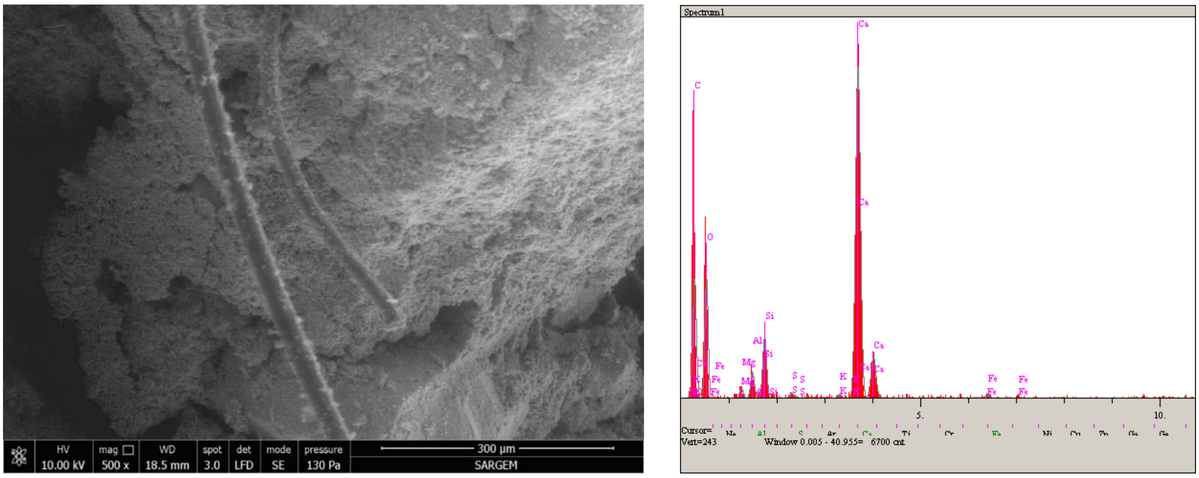

Fig. 2. FESEM-EDS images of the X500 magnified glass fiber reinforced mortar sample No.1.

Table 2. EDS analysis results table for Sample No.1 with glass fiber reinforced mortar.

Elt.	Line	Intensity (c/s)	Error 2-sig	Conc	Units	
C	Ka	19.86	2.818	5.789	wt.%	
O	Ka	96.69	6.218	44.509	wt.%	
Mg	Ka	4.59	1.355	0.459	wt.%	
Al	Ka	39.78	3.988	3.600	wt.%	
Si	Ka	92.70	6.088	7.961	wt.%	
S	Ka	4.33	1.316	0.390	wt.%	
K	Ka	4.70	1.370	0.481	wt.%	
Ca	Ka	299.74	10.947	35.913	wt.%	
Fe	Ka	2.86	1.070	0.898	wt.%	
				100.000	wt.%	Total

In the EDS analysis of Sample No. 2, it was concluded that it has a calcite mineral structure with high amounts of Ca, C, O, Si elements, as well as Mg, Al, and a very small amount of Fe element, according to the EDS result table at X100 and X500 magnifications.

The FESEM-EDS image at X100 magnification of Sample No. 2 with Glass Fiber Addition (Figure 3), the EDS analysis result table of Sample No. 2 with Glass Fiber Addition (Table 3), the FESEM-EDS image at X500 magnification of Sample No. 2 with Glass Fiber Addition (Figure 4), and the EDS analysis result table of Sample No. 2 are provided (Table 4).

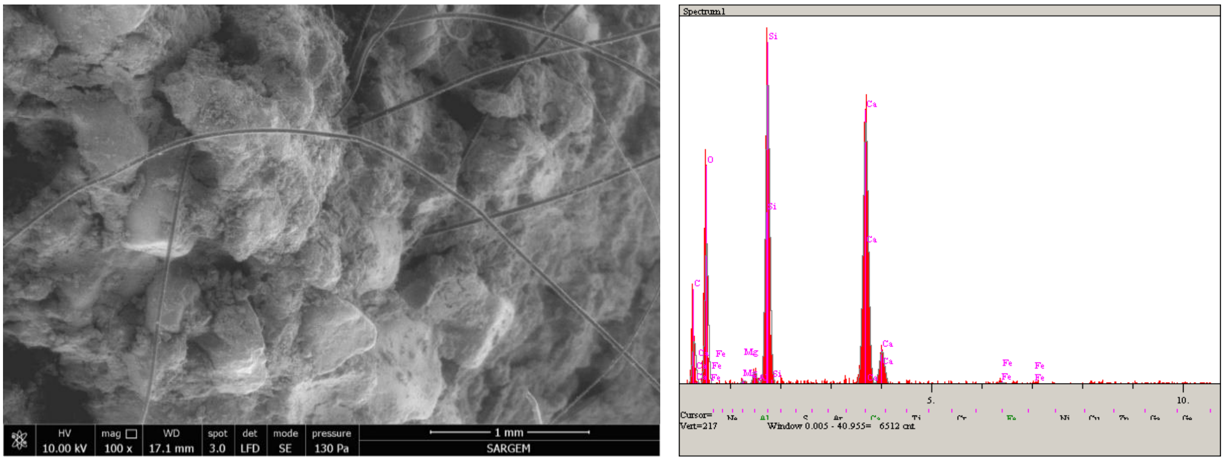

Fig. 3. FESEM-EDS images of the X100 magnified glass fiber reinforced mortar sample No.2.

Table 3. EDS analysis results table for Sample No.2 with glass fiber reinforced mortar.

Elt.	Line	Intensity (c/s)	Error 2-sig	Conc	Units	
C	Ka	23.08	3.038	10.672	wt.%	
O	Ka	71.55	5.349	39.289	wt.%	
Mg	Ka	2.08	0.913	0.245	wt.%	
Al	Ka	7.10	1.685	0.759	wt.%	
Si	Ka	186.90	8.645	18.821	wt.%	
Ca	Ka	197.34	8.883	29.421	wt.%	
Fe	Ka	2.05	0.906	0.793	wt.%	
				100.000	wt.%	Total

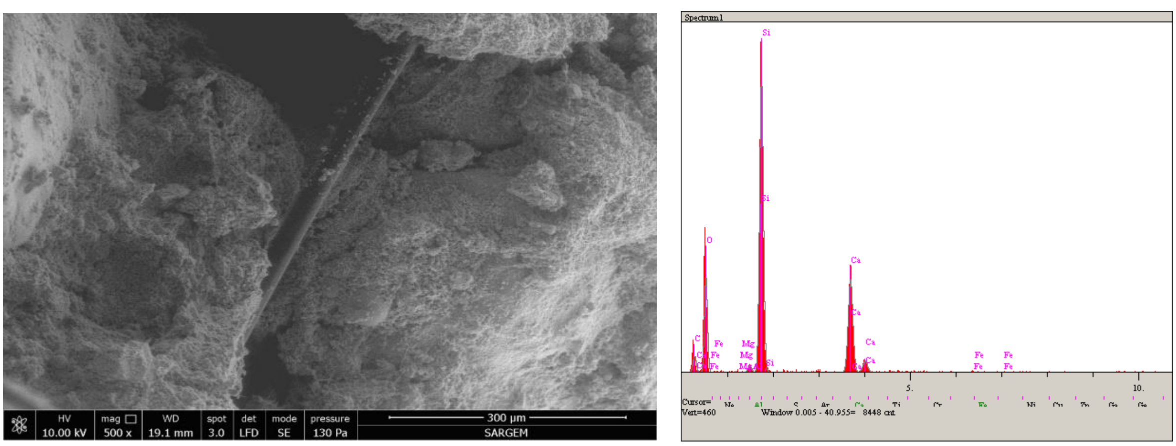

Fig. 4. SEM-EDS images of the X500 magnified glass fiber reinforced mortar sample No.2.

Table 4. EDS analysis results table for Sample No.2 with glass fiber reinforced mortar.

Elt.	Line	Intensity (c/s)	Error 2-sig	Conc	Units	
C	Ka	20.21	2.842	10.369	wt.%	
O	Ka	106.93	6.539	39.000	wt.%	
Mg	Ka	0.74	0.544	0.065	wt.%	
Al	Ka	8.87	1.883	0.712	wt.%	
Si	Ka	398.73	12.627	30.778	wt.%	
Ca	Ka	155.32	7.881	18.541	wt.%	
Fe	Ka	1.77	0.840	0.536	wt.%	
				100.000	wt.%	Total

3.2. XRD Analysis

In the X-ray analysis of glass-reinforced lime mortar samples with different contents, XRD analyses of the raw materials forming the mortar were performed. 1 cm^3 pieces taken from the samples at the end of 90 days of age were ground into powder, and the mineralogical structures of the samples were determined.

The X-ray diffraction pattern of the sample of mortar with glass fiber reinforcement No. 1 revealed the presence of calcite (C), silicate (Si), magnetite (iron oxide), and kyanite, which is an aluminum-rich aluminosilicate mineral. When examining the XRD graph of the mortar, it was determined that it is a material that contains a high amount of calcite, CaCO3 (calcium carbonate). The XRD result graph for the sample of mortar with glass fiber reinforcement No. 1 is shown in Figure 5.

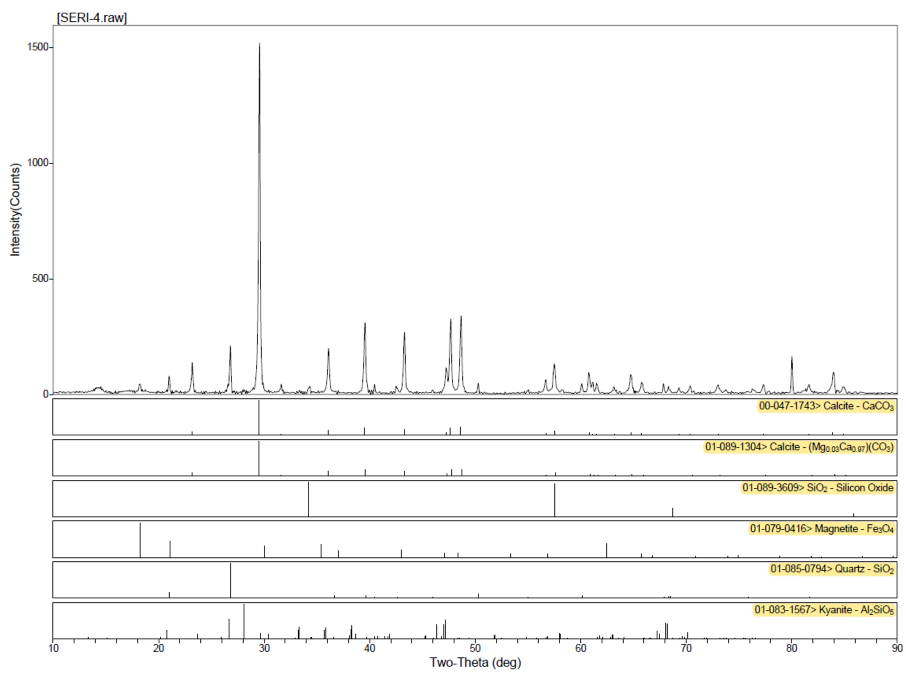

Fig. 5. XRD graph of the No.1 glass fiber reinforced mortar sample.

The diffraction patterns of sample 2, which is a cement mixture with glass fiber reinforcement, revealed the presence of calcite (C), quartz (Q), and magnetite (iron oxide) minerals. Upon examination of the XRD graph of the mixture, it was determined that it predominantly consists of calcite, $CaCO_3$ (calcium carbonate), followed by quartz and iron minerals. The XRD result graph for sample 2 of the cement mixture with glass fiber reinforcement is provided in Figure 6.

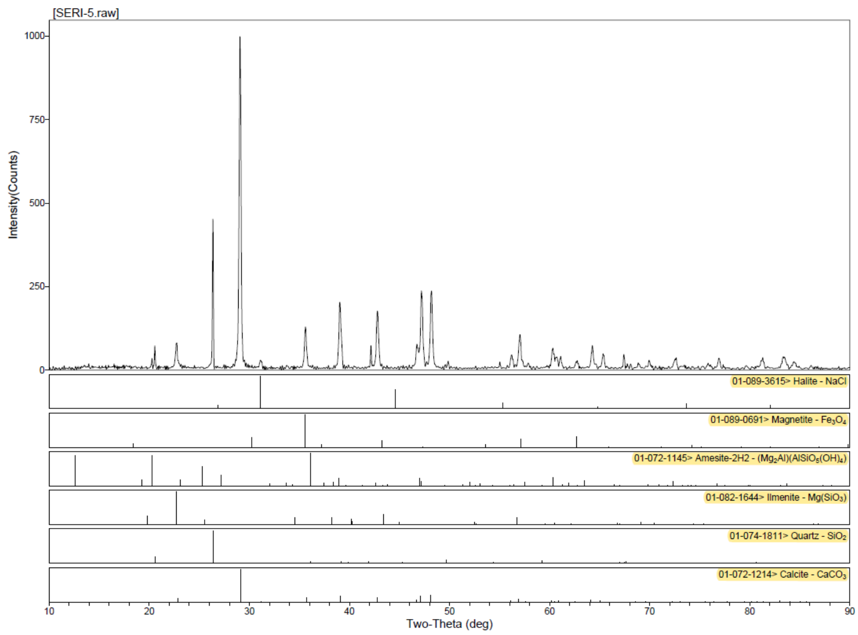

Fig. 6. XRD graph of the No.2 glass fiber reinforced mortar sample.

4. Conclusions

Composite materials are comprehensively characterized using a variety analytical techniques. XRD analyses are used to investigate the microstructure and morphology of composite materials, while scanning electron microscopy (SEM) is used to determine their chemical composition and distribution using energy dispersive spectroscopy (EDS) (Şahin et al., 2023). The microstructural properties of the mortar, as well as the mineralogical and petrographic analyses of the raw material composition, were determined using FESEM-EDS, and XRD analyses to identify the characteristic features, deterioration morphologies, and effects on the internal structure in the chemical application of the produced lime samples with glass fiber reinforcement.

The characteristic properties of the produced lime samples with glass fiber additives, their degradation morphologies, and the effects on the internal structure in chemical applications were determined through mineralogical and petrographic analyses. These analyses were conducted using various methods such as FESEM-EDS, and XRD analyses. Through these analyses, detailed information about the properties and internal structures of the produced lime samples with glass fiber additives could be obtained.

According to the evaluations made for mineralogical and petrographic analyses; after 90 days, 1 cm^3 pieces were taken from the glass fiber-reinforced mortar samples and X100, X500 magnified FESEM and X100, X500 magnified EDS images were obtained. In the FESEM images, insufficient adhesion of the glass fiber to the mortar resulted in the formation of voids. Sample No. 1 of the glass fiber-reinforced mortar was found to contain high levels of Ca, C, O, Si elements, as well as Mg, Al, and small amounts of K, S, Fe elements, indicating a calcite mineral structure. Based on these analysis results, the development of glass fiber-reinforced mortar and its improved use in different proportions will be guiding for restoration applications. These studies will be an important step towards achieving more durable and long-lasting results in the restoration process. Additionally, the use of glass fiber-reinforced mortar can reduce restoration costs and shorten the working time. Therefore, it is believed that these studies will introduce a new method and approach in the field of restoration. All of these will contribute to working more effectively and efficiently in restoration projects and to the preservation of structures.

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