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An Investigation On the Use Of Geopolymer As An Alternative Adhesive For Aluminum Panels

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Abstract: One of the improving application areas of geopolymers is their using as an alternative assembly of different materials. This study presents the results of an experimental study on making aluminum panels by using geopolymer as an alternative adhesive to replace typical organic polymers. We prepared a metakaolin-based geopolymer having a composition of $Na_2O•Al_2O_3•4SiO_2•11H_2O$. Aluminum alloys (Al 3003), 7 and 15 mm in high and (Al 5005) were used as core and plate materials of the panels, respectively and their surface was cleaned for adhesion bonding according to ASTM D 2651-01. Shear test samples were cut and then geopolymer resin was applied to the cleaned surface of aluminum core and plate by hand. Following hydrothermal sealing, the samples were then cured at 50 °C in an open-air furnace for 36 h under clamp fixed. Aluminum-geopolymer interface and geopolymer bond were examined by SEM-EDS in terms of chemical composition. Ambient temperature mechanical test results revealed that the aluminum-geopolymer bonds have mean shear strength of (1.406 ±1.01 MPa) and (0.133 ±0.07 MPa) for 7 and 15 mm samples, respectively.

Geopolimerin Alüminyum Panellere Alternatif Yapıştırıcı Olarak Kullanımı Üzerine Bir Araştırma

Anahtar Kelimeler
Jeopolimer,
Yapıştırıcı,
Aliminyum paneller

Öz: Jeopolimerlerin gelişen uygulama alanlarından biri de farklı malzemelerin birleştirilmesinde alternatif olarak kullanılmasıdır. Bu çalışma, tipik organik polimerlerin yerini alacak alternatif bir yapıştırıcı olarak jeopolimer kullanarak alüminyum paneller yapmaya yönelik deneysel bir çalışmanın sonuçlarını sunmaktadır. $Na_2O•Al_2O_3•4SiO_2•11H_2O$ bileşimine sahip metakaolin bazlı bir jeopolimer hazırladık. Panellerin çekirdek ve plaka malzemesi olarak sırasıyla 7 ve 15 mm yüksekliğinde alüminyum alaşımları (Al 3003), (Al 5005) kullanılmış ve yüzeyleri ASTM D 2651-01'e göre yapışma için temizlenmiştir. Kayma testi numuneleri kesilmiş ve daha sonra alüminyum çekirdeğin ve levhanın temizlenmiş yüzeyine elle jeopolimer reçine uygulanmıştır. Hidrotermal sızdırmazlığın ardından, numuneler daha sonra 50 °C'de bir açık hava fırınında 36 saat süreyle kelepçe altında kürlendi. Alüminyum-geopolimer arayüzü ve geopolimer bağı SEM-EDS ile kimyasal bileşim açısından incelenmiştir. Oda sıcaklığındaki mekanik test sonuçları, alüminyum-jeopolimer bağlantılarının 7 ve 15 mm'lik numuneler için sırasıyla (1.406 ±1.01 MPa) ve (0.133 ±0.07 MPa) ortalama kayma direncine sahip olduğunu ortaya koydu.

1. Introduction

Geopolymers (GPs) are a class of inorganic polymeric, new ceramic-like X-ray amorphous materials composed of cross-linked alumina (AlO_4^-) and silica (SiO_4) tetrahedra to form polysialates, with a charge balancing cation (Na^+, K^+, Cs^+, etc.). The term geopolymer, also known as an alkali-activated aluminosilicate, was coined by Joseph Davidovits in the 1970s [1]. Their processing includes dissolution

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and polycondensation of any natural or industrial silicoaluminate sources in the presence of caustic alkali solutions, and subsequently a network form at room temperature. Along with these, geopolymers have been investigated for years as an alternative binder to cement, especially due to their less carbon footprint production and thus contributions to the circular economy [2]. By controlling the initial composition, a wide variety of properties and characteristics can be gained for GPs and they find a lot of applications area such as refractory applications, as precursors to high technology ceramics [3-5], coating and adhesive [6, 7]. In particular ceramic-like characteristics, including high-temperature resistance, make geopolymers interesting for fire resistant coating applications [8, 9]. Even if the adhesion strength is low, they were first used as coating material in aircraft cabin where even a few minutes are crucial, following it has been reported that they can be a promising coating material on metal [10-12] or concrete [13] for similar aim such as automobiles, naval structures and builds [14,15]. These studies have encouraged the use of geopolymers for bonding different materials such as metal or polymer. Bell et al. [16] reported < 1 MPa shear strength in steel in their study of joining special steel and aluminum alloys, alumina and borosilicate glasses with geopolymer. Considering that the bonding material and its surface preparation, including alkali choice as well, affects the bond properties, this study is quite limited and further studies would be made by applying GPs to joints of different metal alloys in different alkaline conditions [17].

Panel structures are a method for consolidating light metal alloys such as aluminum and have begun to appeal to a wide range of uses. In applications (space shuttles, aircraft, boats, and construction industry) that required maximum rigidity and strength with minimum weight, the sandwich panels are the most promising materials [18-20]. They are application areas that require high mechanical strength in which the energy resulting from impact is absorbed. A core is placed between the plates to obtain additional lightweight structures. Polymer-bismaleimide and epoxy are used as adhesive for bonding between the plate and the core [21-23]. However, these adhesives have low operating temperatures; bismaleimide 260 °C, epoxy 180 °C, vinyl phenolic 130 °C, and their densities are 1.75g/cm^3, 1.47g/cm^3,1.28 g/cm^3, respectively. GPs itself are inorganic polymers and of relatively lower density (1.4 g/cm^3) and in particular much higher fire resistance even around 1200 °C than the other organic polymers [24, 25]

This study aims to investigate the use of GPs as an alternative adhesive to the above organic polymers for the preparation of aluminum panels. In addition, the application of geopolymers, which can be produced from a waste aluminosilicate source at room temperature, in an alternative field would also contribute to a sustainable approach [26]. Hot pressing, vacuum bag and suitable mold methods are utilized to make aluminum panels [27]. Hot pressing is the heating of pre-impregnated resin plates and joining them with adhesive. Vacuum bag production is the assembly of complex-shaped parts, usually heated in the oven, by applying negative pressure. Production in a suitable mold is the joining of heated equipment by curing in an oven under mechanical pressure, with the possibility of temperature and pressure control. As indicated above organics require a high-temperature process sometimes multi-step, GPs, however, need only be cured once at relatively low temperatures (50 °C) to complete their curing process [16]. As for mechanical characterization, shear tests are of critical importance in determining the adequate mechanical strength of joints using cemented or geopolymer adhesive [28, 29]. Room temperature shear strength and the microstructure of plate-core interfaces were made to characterize the GP-bonded aluminum panels.

2. Material and Method

Aluminum alloys (Al 3003), 7 and 15 mm in high and (Al 5005) were used as core and plate materials of composite panels, respectively. Sulfuric acid (Sigma-Aldrich) and sodium dichromate (Sigma-Aldrich) were used in the surface cleaning solution of these alloys. Metakaolin (Al_2O_3•$2SiO_2$) obtained by 6 h calcination of kaolin from Eti Maden, Turkey, at 900 °C and fumed silica, with an average particle size of 0.007 µm (Sigma-Aldrich) and NaOH pellets (Sigma-Aldrich) was used for geopolymer binder preparation.

The shear test specimens were cut from the aluminum sheet and the cell in the size of 80x50x15 mm and 80x50x7 mm and their surface was cleaned for adhesion bonding according to ASTM D 2651-01 [30]. The cleaning protocol for aluminum surfaces is shown in Table 1. Sulfuric Acid-Sodium Dichromate solution was applied to the surfaces in the following order. Aluminum surfaces were washed with acetone to remove oil and grease residues from the surface. The cleaned surface plates were immersed in the Sulfuric Acid-Sodium Dichromate solution given in Table 1 at 66-71°C for 12-15 minutes. Then, immersion was carried out for 1-2 minutes at 40 °C in tap water and 30 minutes in pure water at 60 °C. While preparing the solution, the components were first poured with 60 % of one liter of water and mixed, and then the remaining water was added.

Table 1. The cleaning protocol for aluminum surfaces

Constituents	**Amounts in 1 liter water (g)**
Sulfuric Acid	287.9-310
Sodium Dichromate	28-67.3
Aluminum Alloy	1.5

As for geopolymer binder preparation, a sodium silicate solution (water glass) with a composition of ($NaOH.2SiO_2.11H_2O$) was first made by dissolving the required amount of NaOH pellets and then fumed silica in deionized water. The solution was magnetically stirred overnight to ensure that the silicon had completely dissolved. Following metakaolin was mechanically mixed with the sodium silicate solution to obtain the resulting geopolymer binder with a composition of ($Na_2O.Al_2O_3.4SiO_2.11H_2O$). Subsequently, the homogenous geopolymer slurry was applied to the prepared aluminum surface by hand and cured at 50 °C in an open-air oven for 36 h under clamp-fixed and sealing conditions.

Surfaces of starting aluminum were examined with an optical microscope (Nikon ECLIPSE LV150N) and the interface properties of assembled panels were characterized by a scanning electron microscope (SEM) equipped with energy dispersive spectroscopy (EDS) analysis (Quanta, 450). The shear strength of the samples was made by Shimadzu mechanical testing machine using a 100 kN load cell at 0.5 mm/min. Five specimens were tested for each average value and their results were given with standard deviation calculations.

3. Results

The surface treatment and preparation methods required for adhesive bonds depend on the specific nature of each material. The preparation of the aluminum plate surfaces used in this study is a crucial factor affecting the bonding capability of the panels as well. Sulfuric acid-sodium dichromate solution was chosen as an effective, controllable and safe method for surface preparation of the aluminum alloys. In Figure 1, optical images of the aluminum plate before and after surface preparation are given. Unlike starting surface in Figure1a, Figure 1b clearly shows a cleaned surface that was free of layers without oxide, paint, chemical residues, oil, etc., which will affect the full contact between the adhesive and the surfaces. If these layers, called "Weak Boundary Layers", are not cleaned sufficiently, a homogeneous bonding will not occur. The method is based on the dissolution of the initial oxide layers on the surface and the formation of a new thin oxide layer as a result of etching with the prepared solution. This new layer is thin, reticulated, porous and has ridges, which is sufficient to provide mechanical interlocking between the adhesive and the oxide surface [31, 32].

Figure 2 shows the SEM images of the polished surface and XRD spectra of the adhesive collected from the joint after 36 hours of drying at 50 °C. A homogeneous gepolymerization was observed in the microstructure in general from the SEM images of the adhesive in Figure 2a. However, the drying cracked nature of geopolymers originated from the dehydration, carbonaceous compounds formed because of the reaction of carbon dioxide in the atmosphere with free Na on the surface of the adhesive and some agglomerated regions came from unreacted solid aluminosilicates (metakaolin) [33, 34]. These are inevitable and there is no method developed so far to overcome these issues [35].

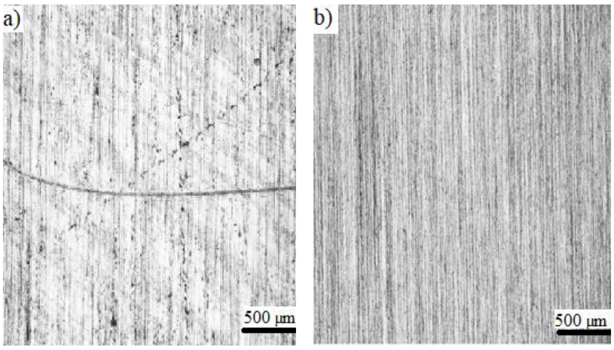

Figure 1. Optical micrographs of aluminum panels; a) starting and b) cleaned surfaces.

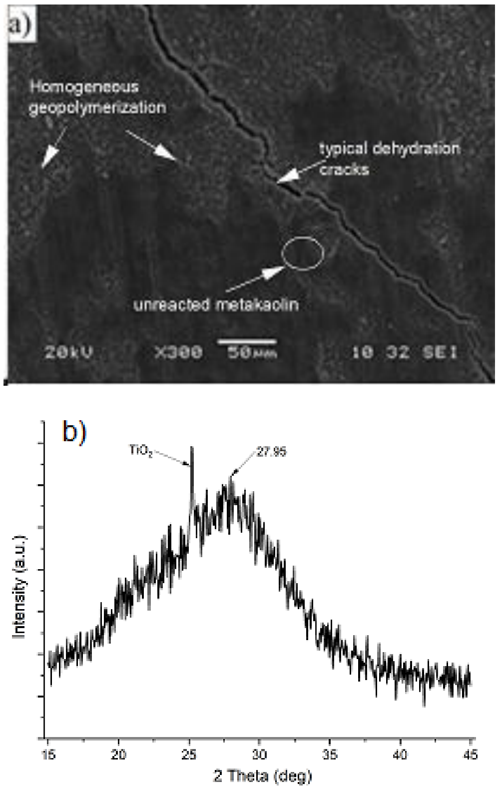

Figure 2. a) SEM image of the polished surface and b) XRD spectra of the adhesive collected from the joint after 36 hours of drying at 50 °C.

This broad 2θ peak around 28º in Figure 2b indicates that the desired geopolymerization has occurred in the adhesive with typical TiO_2 traces on the broad hump [3]. Related to a little amount of metakaolin, which has a peak 2θ peak around 22º seen on the SEM micrograph, no peak detected belongs to metakaolin on XRD. A simplified methodology for the geopolymerization is the dissolution of metakaolin, and this causes to alumina and silicate types to emerge, poly-condensation and precipitation to form an inorganic, polymeric network [36].

The SEM micrographs of the assembled aluminum panel are shown in Figure 3. It is hard to clearly distinguish the aluminum-geopolymer interface since they have very close atomic number contrasts. But it is well known that geopolymer itself includes a porous structure ranging from micro to nano size [37].

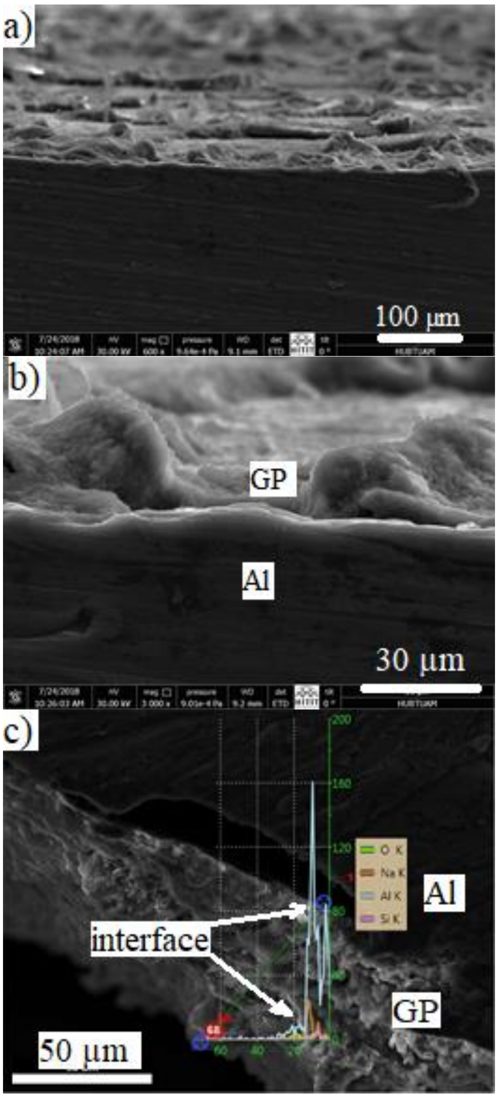

Figure 3. SEM micrographs of the aluminum panel assembled with geopolymer; a) general b) high magnification and c) aluminum-geopolymer interface through line scan.

And also with the help of the general image in Figure 3a geopolymer side and aluminum side were labeled on high magnification image in Figure 3b. Subsequently a line scan EDS analysis was made from the Al side to the GP side to catch the interface chemistry exactly and shown in Figure 3c. The aluminum side was stressed with high-intensity Al peak and the weak Na, Al, Si, O peaks ascribed to the geopolymer side. Following the intense peak of aluminum, diffusion of Si and O peaks, mainly Na, which is the geopolymer component, is observed in a line of 10 microns from approximately 10th microns to 20th microns. Even in this limited distance, it means that diffusion takes place between the aluminum and the geopolymer and the interface is formed. This situation can be explained that the geopolymer thinks that the Al is part of the recipe and may incorporate it to form a geopolymer, leaving porosity at the interface.

Figure 4 shows photographs of shear strength measurements during and after the test. To make more accurate measurements, a more axial pulling force is applied to the bond with the silicon block placed on the plates in Figure 4a. After the shear test, the panel material components have undergone different types of deformations during the test period. In particular, the high distortions that occur in the direction of shear in the core aluminum (Al 3003) material can be taken as evidence of the presence of adhesion at the interface in Figure 4b. Geopolymer was well adhesive on the plate surface and the core material has a smaller surface area than the cover material, but this has contributed to its adhesion by being embedded in the geopolymer paste.

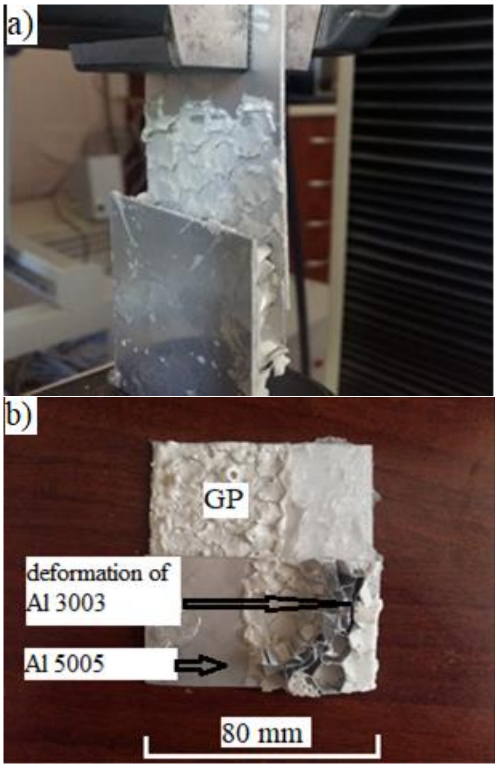

Figure 4. Photographs of shear strength measurement; a) during the test, b) deformation on the core aluminum after the test.

Ambient temperature mechanical test results revealed that the aluminum-geopolymer bond has mean shear strength of (1.406±1.01 MPa) and (0.133±0.07 MPa) for 7 and 15 mm samples, respectively. In a study on bonding 6061-T6 Al with geopolymer, the shear strength of the joint was reported as < 1 MPa [16]. In another study, the shear stresses of the joints obtained by adhering the geopolymers prepared by using different activators from different aluminosilicate sources to the concrete substrate were investigated and the shear strength was measured approximately starting from 2 MPa, depending on the starting precursors [38]. Consistent with comments done on the interface images in Figure 3, when the geopolymer slurry was applied to the treated aluminum surface, a remarkable reaction took place and a highly porous interface occurred because aluminum is one of the constituents of geopolymer. Although an interface is formed between the aluminum-geopolymer in a short time with a rapid reaction, the mechanical properties are limited due to the porous nature of the interface formed. Nevertheless, the results of the shear test are comparable to previous studies.

4. Discussion and Conclusion

Geopolymers having a composition of $Na_2O•Al_2O_3•4SiO_2•11H_2O$ were applied between aluminum alloys (Al 3003) and (Al 5005) to make composite panels. Their surfaces were successfully cleaned for sufficient adhesion bonding according to related standards. SEM-EDS results showed that an interface had occurred about 10 μm in length. Ambient temperature mechanical test results revealed that the aluminum-geopolymer bond had mean shear strength of (1.406±1.01 MPa) and (0.133±0.07 MPa) for 7 and 15 mm samples, respectively. Although comparable results have been obtained with previous studies, geopolymer coating can be suggested to apply on aluminum, especially in terms of fire, where mechanical strength is of secondary importance.

Acknowledgment

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Declaration of Ethical Code

In this study, we undertake that all the rules required to be followed within the scope of the "Higher Education Institutions Scientific Research and Publication Ethics Directive" are complied with, and that none of the actions stated under the heading "Actions Against Scientific Research and Publication Ethics" are not carried out.

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