



# THE EFFECT OF SEWAGE SLUDGE ASH REINFORCED ADHESIVE ON TENSILE STRENGTH OF SINGLE-LAP JOINT

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## ABSTRACT

This study aims to strengthen single lap joints with reinforcement sewage sludge ash (SSA). Micron size SSA was added to the epoxy adhesive at the rates of 0%, 5%, 10%, 15%, 20%, and 25% by weight. 10-layer glass fiber/epoxy composite laminates produced with a thickness of 2 mm were used as adherents. The effects of SSA particles on the epoxy adhesive were investigated by tensile test. The effects of 3 different bonding line thicknesses on the joints, 0.3, 0.5, and 0.7 mm, were also investigated. The optimum value was obtained at 0.5 mm bonding thickness. As a result, the shear strength of 20% by weight SSA added joints increased by 35% compared to the joints with pure epoxy.

**Keywords**: Sewage sludge ash, single lap joint, adhesive

#### 1. INTRODUCTION

Adhesively bonded joints are a good alternative to mechanical connections in many engineering applications, providing many advantages such as higher load transmission over conventional mechanical fasteners, uniform stress distribution due to no holes throughout the bonded region, low-stress concentration, connection of different materials. However, the joints in a structure are much weaker than other parts. Hence, it is necessary to increase the strength of the adhesive in order to increase the strength of the joint. Adding fillers to the adhesive is a common method to improve the mechanical properties of adhesive joints, in recent years.

Many studies have been conducted based on the use of different filler types to demonstrate the effect of these fillers on the mechanical properties of the bond. Many different fillers such as metallic nanoparticles, graphite nanofibers, carbon nanotubes, nano-clays, cellulose nanofibers, and nano-alumina particles have been used to improve epoxy resin properties [1-5].





Ghosh et al. [1] investigated the effect of  $30 \pm 5$  nm TiO<sub>2</sub> nanoparticles on the shear strength of copper plates. TiO<sub>2</sub> nano-particles of 5, 10, and 15% by weight were contributed to the adhesive. The lap-shear test results showed that the maximum shear strength was obtained in 10% by weight TiO2 nanoparticles were added.

Khoramishad and Razavi [6] proposed a new approach for adding metal fibers to reinforce adhesively bonded single-lap joints. The presence of metal fibers in the adhesive layer can increase the toughness of the adhesive joint and improve the stress distribution. It has been found that the incorporation of metal fibers into the adhesive matrix has a significant effect on the bond strength.

Kahraman et al. [7] attempted to alter the mechanical properties of adhesively bonded joints by adding an aluminum filler to an epoxy-based adhesive. The effects of the adhesive on the mechanical properties were investigated by using spherical aluminum particles with a diameter of less than 50  $\mu$ m in the epoxybased adhesive. The single-lap joint and shear tests were used as a standard test for comparison. Aluminum powder was used at the rate of 10%, 25%, and 50% of the total weight of the adhesive mixture. From the results, it was concluded that the use of aluminum filler in epoxy adhesive increases the mechanical strength of the bond. A cohesion error occurred even when 50% by weight of aluminum powder was used.

Zhai et al. [8] stated that combining  $Al_2O_3$  nanoparticles (average 80 nm diameter) with epoxy adhesive can significantly increase the tensile bond strength of the epoxy adhesive. The results showed that the addition of 2% by weight  $Al_2O_3$  nanoparticles to the adhesive increased the bond strength approximately 5 times.

Aydın [9] investigated the changes in the adhesion strength of the adhesives by adding two different sizes of multi-walled carbon nanoparticles in the range from 0.5% to 5% by weight to two different epoxy adhesives used for bonding metal surfaces. Depending on the surface condition, nanoparticle size, and adhesive type, an increase of up to 2%-3.5% in adhesion strength was observed.

Sadigh and Marami [10] performed tensile and compression tests to examine the effect of reduced graphene oxide (RGO) on the strength of adhesively bonded joints. When 0.5% by weight of RGO was added to the epoxy, the final tensile and compressive strengths of the cast samples increased by 30% and 26%, respectively. Since sewage sludge ash is a waste material, it has negative effects on the environment.

For this reason, it is used as a filler in certain proportions, especially in construction and chemical applications, to reduce the environmental effects of this waste material. Some studies in the literature, the effects of sewage





sludge ash on the mechanical properties of polymer composite materials were investigated [11-13].

It has been stated that the addition of fillers to epoxy resins plays an important role in improving various properties such as adhesion, thermal properties, electrical conductivity, and abrasion resistance in the literature. It is accepted that the large surface-to-volume ratio of nanoscale reinforcements plays an important role in improving mechanical properties.

At the same time, two ideas related to the reinforcement mechanism are presented in the literature. The first is that the formation of a nanostructure network of finely dispersed particles likely bonded to the polymer provides improvement. The second is that reinforcements occur due to the presence of a mechanically based interface observed in filled epoxies. Reinforcing epoxy resins with nanofillers has become necessary in engineering applications that require higher mechanical and thermal properties.

Considering the importance of the filler material, this study is based on the use of a new type of filler material with a different structure and a complex element composition than those used before. In this study, SSA microparticles in different mixing ratios were incorporated into the epoxy resin to examine their effects on the bond strength of single-lap adhesive joints and the maximum stress of these joints.

In addition, the effect of different bond line thicknesses on the bond strength was also investigated.

# 2. MATERIALS AND METHOD

#### 2.1. Materials

Sewage sludge ash (SSA) is basically a silty material containing some sandsized particles. It is a by-product of the combustion process of dewatered sewage sludge in a controlled combustion system. SSA is a polyphasic material consisting of a glassy phase ( $\approx$ 40%) and some crystalline minerals ( $\approx$ 60%).

The properties and size range of sludge ash depend on the type of incineration system and the chemicals involved in the wastewater treatment process. SSA microparticles which are used as a filler in the bonding process were obtained from Gaziantep Municipality Water and Sewerage Department (GASKI).

The components and their content ratio in the ash are shown in Table 1. MGS L285 epoxy resin and MGS L285 hardener used in a ratio of 100:40 was supplied from Dost Kimya.



Oxide	Percentage	Oxide	Percentage	Oxide	Percentage
P <sub>2</sub> O <sub>5</sub>	23.655	K <sub>2</sub> O	4.874	CuO	0.189
CaO	19.588	ZnO	2.096	$MnO_2$	0.188
SiO <sub>2</sub>	16.602	TiO <sub>2</sub>	1.079	NiO	0.068
SO <sub>3</sub>	8.533	C1	0.539	Br	0.063
MgO	8.221	Na <sub>2</sub> O	0.442	SrO	0.049
Fe <sub>2</sub> O <sub>3</sub>	7.461	$Cr_2O_3$	0.354	$ZrO_2$	0.036
Al <sub>2</sub> O <sub>3</sub>	5.727	BaO	0.206	PbO	0.031

**Table 1.** Components of SSA and their content percentage.

# 2.2. Preparation of Adhesive Material

GFRP composite plates were produced from 200  $\text{gr/m}^2$  glass fabric with dimensions of 250x350 mm in 10 layers. The thickness of the produced composite was measured as 2 mm.

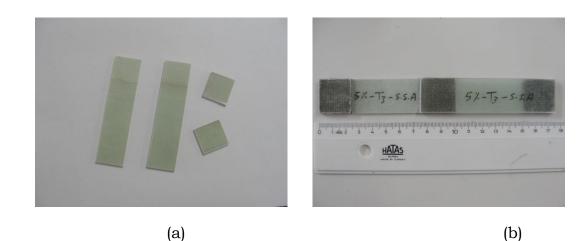
In order to prepare the surfaces of the GFRP plates for bonding, they were first sanded with P120D grade sandpaper, then acetone was used to remove chemical dirt and residues. GFRP laminates whose bonding surface was prepared were cut with a guillotine in dimensions of 100x25 mm for single-lap joints.

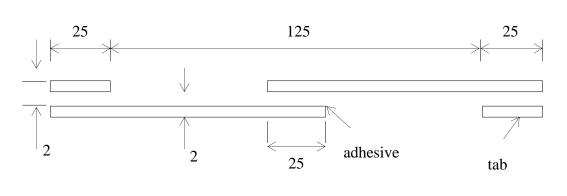
First, SLJs with different bond line thicknesses of 0.3, 0.5, and 0.7 mm were produced with GFRP substrates. The bond line thickness is provided by the metal material placed in between.

Figure 1 shows the specimen and schematic view of the single-lap joint. The tensile test was conducted by Shimadzu universal tensile testing machine according to ASTM D3165 – 07. The crosshead speed of the machine was 1 mm/min for all tests.









(c)

**Figure 1.** Single lap joint specimen. (a) Before assembling, (b) After assembling with the adhesive, (c) Schematic diagram with dimensions in (mm)

MGS H285 type hardener and L285 type epoxy resin were used in a 100:40 ratio modified with SSA particles in different proportions to bond GFRP composite substrates. These ratios, ranging from 5% to 25%, were considered as the total weight of the mixture.

Table 2 gives the epoxy adhesive and particle ratios used during sample preparation. In order to obtain a homogeneous particle distribution, SSA and epoxy resin were mixed with a fast mixer at 10000 rpm for 10 minutes. Then, the hardener was added to the mixture and mixed for another 5 minutes.





SSA	Epoxy	Hardener (%)	
(%)	(%)		
0	71.50	28.50	
5	67.93	27.07	
10	64.35	25.65	
15	60.78	24.22	
20	57.20	22.80	
25	53.63	21.37	

Table 2. The adhesive mixture components of each bonded joint specimen.

## **3. RESULTS AND DISCUSSION**

#### **3.1 Tensile test results**

The tensile test was performed on three fabricated single-lap adhesive bond samples. The values were then averaged. Maximum tensile force and displacements are given in Table 3.

**Table 3.** The average values of maximum force, displacement, and shearstress of SLJs under tensile loading

SSA ratio wt. %	Average tensile force (N)	Average displacement (mm)	Average shear stress (MPa)
0%	2362.02	0.92	3.78
5%	1801.36	0.55	2.88
10%	2299.96	0.87	3.68
15%	2735.07	1.20	4.38
20%	3225.51	1.27	5.16
25%	2616.50	1.03	4.19





The results showed that the shear strength of these joints increased with the addition of SSA. There was an unexpected decrease in the lap-shear stress values of the 5% and 10% by weight doped joints, and the measured values were lower than the pure epoxy joints.

The addition of 15% and 20% SSA to the epoxy increased the lap shear strength of the joints. The highest healing was found as 35% in SLJ with 20 wt.% SSA addition, compared to the pure epoxy joint. After maximum recovery, the lap shear strength of 25% SSA added joints decreased, but it was higher than that of unmodified joints.

The graph of tensile load vs. elongation according to different SSA ratios is given in Figure 2. The fact that the elongation amount of the SSA additive joints is higher showed that it is more ductile than the pure epoxy joints. The larger areas under the load-displacement curve of the SSA added samples showed that the SSA additive increased the toughness of the epoxy resin.

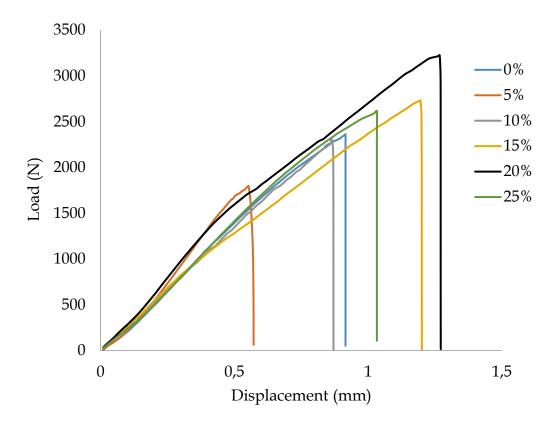


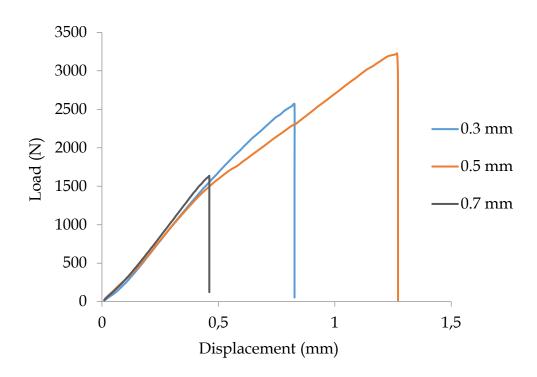
Figure 2. Load-displacement graph for tensile test of SLJs





# **3.2 Effect of Bond Line Thickness**

To investigate the effect of bond line thickness, single lap joints with various thicknesses of 0.3, 0.5, and 0.7 mm were fabricated. Since the highest shear strength is obtained in connections with 20% SSA additives, new connections were produced by keeping the 20% additive ratio constant in order to investigate the effect of different bond line thicknesses. Three specimens were produced for each bond line thickness and tested under tensile load. Average tensile loads, average elongations, and average shear stresses of the samples are given in Table 4. The load-displacement curves are shown in Figure 3.



**Figure 3**. Load- displacement graphs for tensile test of SLJs with different adhesive thicknesses

The results showed that the maximum lap shear strength was obtained when the bond line thickness was 0.5 mm. An optimum bond line thickness of 0.5 mm indicates that the stress distribution of the joint becomes uniform at this thickness.

However, a bond line thickness of 0.7 mm can cause high-stress concentration and high porosity (voids) and microcracks (due to residual stresses during curing) at the interface [14].





Adhesive thickness (mm)	Average tensile force (N)	Average displacement (mm)	Average shear stress (MPa)
0.3	2569.29	0.83	4.11
0.5	3225.51	1.27	5.16
0.7	1634.91	0.46	2.62

**Table 4.** The average values of maximum force, displacement, and shear stress of SLJs with different adhesive thickness under tensile loading

## **3.3 Macro Analysis of Fractured Surfaces**

Figure 4 shows the macro views of the fracture surfaces of the specimens after the lap shear test. Adhesive failures were observed on the fractured surfaces of pure epoxy and 5wt.% SSA added samples (Figure 4a and b). Adhesive failure occurs at the interfacial area of the adherents and the adhesive, with residual adhesive remaining at any position on the surface of only one of the adherents.

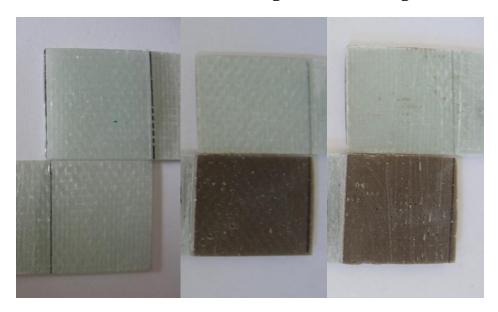
Adhesive failure results from a complete breakdown of the chemical bonds at the interfacial area. This type of failure indicates weak adhesive bonds, and thus pure epoxy and 5% SSA added joints have low strength. Adhesive failure on the surface of the 10% SSA-doped joints, along with light-fibre tear failures in a small area of the substrate surface, were also observed (Figure 4c). The presence of clean and sticky areas on both fracture surfaces of 15% or more SSA additives samples indicated that the failure mode type was mixed mode. Also, fibre imprints can be seen in these specimens. Mixed-mode failure consists of some adhesion failure and some cohesion failure. This failure happens when the interface is partially degraded. In fact, mixed-mode failure is just a transitional phase between adhesion and cohesion failure.

The SSA particles showed a generally positive effect, providing new mechanical pathways for load transfer between the substrates and the epoxy. However, decreases were observed in the shear strengths of 5% and 10% SSA added samples compared to pure epoxy joints. This reduction may be mainly due to insufficient surface roughness, it can also be caused by the fact that SSA is not dispersed homogeneously in the epoxy. The surface treatment is one of the most critical factors that influence the strength of the adhesive joint. But some other defects can be led to this low value, such as contaminants on substrate surfaces; the adhesive was cured before the joint design and the presence of voids at the interface [3, 15, 16]. Unlike the third, which is quite observable, the first two causes cannot be fully controlled and proven. These voids were resulted due to the air entrapment during specimen placement.





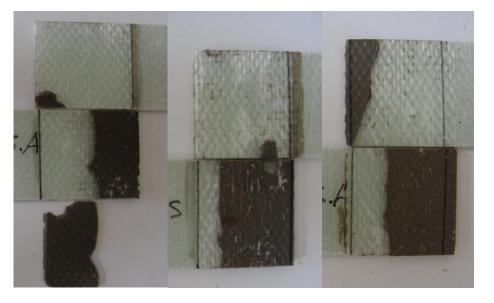
The average shear strength of these specimens could probably increase if these voids were eliminated [17]. The strength of the bond and the proportion of surface smoothness or roughness depend upon the level of degradation of the bond interface [18]. Mixed-mode type failures were observed on the fracture surfaces of samples containing 15% or more SSA additives after the lap-shear tests. As a result, this is great evidence that the incorporation of SSA particles into the epoxy plays an important role in improving the chemical bonds at the interface and thus increasing the bond strength.



(a) 0% SSA

(b) 5% SSA

(c) 10% SSA



(d) 15% SSA (e) 20% SSA (f) 25% SSA

Figure 4. Failure surfaces of SLJs subjected to shear loading





# 4. CONCLUSION

The effect of SSA on the mechanical properties of epoxy resin was investigated in this study. The experimental results showed that the inclusion of SSA in epoxy has a significant effect on the shear stress of adhesive lap joints. In the light of these results, it can be concluded the following:

- The use of SSA composite adhesive improves the lap shear strength of the joint. This could be caused by the effective transfer of stress between SSA particles and epoxy matrix which plays a significant role in enhancing the adhesion properties of the adhesive bonds. The shear strength of the adhesive joint increases with the increase of SSA content ratio up to 20%. This increase of joint strength is followed by a reduction in it with a further increase in the SSA content to 25%.
- The addition of SSA micro-particles efficiently improves the chemical compatibility of epoxy adhesive with glass/epoxy substrates and increases the surface wetting ability which plays a great role in the increase of shear strength of joints.
- The lap shear strength of the composite adhesive greatly depends on bond line thickness. The relationship between the shear strength of the adhesive mixture (SSA/epoxy) and the adhesive layer thickness is non-linear. At 0.5 mm of adhesive thickness, the test machine recorded higher shear stress than that of 0.3 mm thickness. After that, there was a decrease in shear stress by increasing the adhesive thickness to 0.7 mm.
- The addition of SSA made the epoxy more ductile than pure state this means more uniform stress distribution across the joint which results in higher strength.
- The use of SSA is an economical way to produce an epoxy-based adhesive with higher mechanical properties due to the low cost of SSA.

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