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The Effect of Organic Fillers on the Mechanical Strength of the Joint in the Adhesive Bonding

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Abstract: The most important advantages of adding additives to adhesives are increasing the bonding strength and reducing the adhesive cost. The desire to reduce costs as well as the need for environmentally friendly and health-friendly products have paved the way for the recycling of waste materials and the use of cheaper natural materials as additives. In this study, mussel, olive pomace, and walnut powders in different ratios (5%, 15%, and 30% by weight) and in different sizes (38 and 45 µm) were added to an epoxy adhesive. The steel materials were joined in the form of single-lap joints by using the obtained adhesives with additives. These joints were subjected to the tensile test and the strengths of these joints were examined. SEM images of the bonding interface were taken, and the distribution of the powders was examined. When the powder size was 45 µm, bond strengths increased in all additive ratios compared to the pure adhesive, while for 38 µm powders, the strength value increased only at the 5% additive ratio. In joints with 45 µm powder additives, the strength increased by up to 38% compared to the pure adhesive, while this rate was determined as 31% for 38 µm.

Keywords: adhesive bonding; organic filler; waste material; the single lap joint; mechanical strength

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1. Introduction

Although both adhesive materials and the application methods have changed over the years, adhesive bonding is a traditional method that has been used since ancient times to join various materials [1]. There are many factors that must be taken into account in order for a bonding application to be considered successful. Since the sectors in which adhesives are used are quite different from each other, it is important to use a strong, economical adhesive appropriate for the application purpose [2]. Additionally, adhesive materials have the potential to be developed according to their intended use. Adhesive materials are very diverse since they are made of plastic and rubber material groups [3]. Moreover, due to the combinations of the properties and amount of organic and inorganic additives in nano or micron size that can be added into additives, the types of new composite adhesives and their application areas are constantly increasing [4–11].

When the literature is examined, it is seen that there are various studies conducted by adding ceramic/glass [12–14], metal [15–18], and plastic-based [19–21] additives into adhesive materials. These studies have focused on reducing the processing cost; increasing the mechanical strength; improving the viscosity, electrical, and/or thermal conductivity; and improving the water/moisture absorption properties [22]. By adding materials found in nature or produced in the laboratory into adhesives, the researchers aim to increase the life expectancy of the joint and its resistance to the forces that the joint is exposed to.

Difficulties in supplying raw materials and increased product costs are serious problems worldwide. The increase in raw material costs and the need for environmentally friendly and biodegradable products have also affected the adhesive industry. In the countries that do not have adhesive manufacturers or whose adhesive suppliers are dependent on foreign countries, it becomes a much bigger problem to reach these products.

The fact that natural (bio-based) additives have advantages such as ease of availability, low cost, and being environmentally friendly support the interest in studies on the additives and their effects on the bonding process. Materials such as rye, wheat, walnut shell and wood flours, flour, soybean powder, wood powder, and bark powder can be organic additives. Similarly, agricultural industrial waste materials such as palm kernel and starch material are specimens for such additives [23]. Some organic materials are used as composite additives [24], and some are used in the form of fibers in the adhesive [25,26].

Kumar et al. [27] investigated the mechanical properties of particle-filled composites produced using biowaste horn powder (HP) and epoxy resin. The HP particles and matrix were mixed and molded in an appropriate ratio and cured at room temperature to produce the specimens. The properties of the samples such as tensile strength, tensile modulus, elongation percentage at break, flexural strength, flexural modulus, impact strength, and microstructure were investigated.

Alireza Akhavan-Safar et al. [28] examined the effects of date palm fibers on the mode I fracture energy of adhesives. For this purpose, they added fibers collected from four different parts of a date palm tree (bunch, rachis, petiole, and mesh) to the adhesive in three various weight ratios (2%, 5%, and 10%). The results showed that date palm fibers had the ability to increase the tensile fracture energy of adhesives. It was also found that the mode I fracture energy of the adhesive reinforced by 10% weight of rachis fiber was 7.6 times higher than that of the pure adhesive. The same authors, in another study [29], improved the static strength of the bonded joints by factors such as the type and size of natural fibers/particles, alkali treatment, and weight ratio. Fibers collected from a date palm tree were added to the adhesive in different weight ratios (2%, 5%, and 10%), in short fiber (0.5–2 mm) and long fiber (30 mm) sizes. They found that the strength of single-lap joints reinforced by 2% weight of rachis fiber treated with 6% by weight NaOH solution increased by 140%.

Barbosa et al. [30] used natural micro cork particles ranging in size from 125 to 250 mm to increase the ductility of a brittle epoxy adhesive. The amount of cork varying between 0.5% and 5% in weight was added to Araldite 2020 epoxy adhesive and the effect of the amount of cork particles on the joint was investigated. As a result of the evaluation conducted using tensile tests, it was seen that higher adhesive ductility and joints containing 1% cork had higher bond strength.

The reuse of waste materials both prevents the pollution of natural resources (soil, water, etc.) and can provide new high value-added products at affordable costs. When the literature was reviewed, no study was found on the reintroduction of waste materials into production and adding them to adhesives as reinforcement.

In this study, waste mussel, olive pomace, and walnut shells were added to an adhesive material in different ratios (5%, 15%, and 30% by weight) and in different sizes (38 and 45 µm) after being recycled. Then, they were used as an adhesive in single-lap joints for the experimental investigation of joint strength. Finally, the tensile test was applied to examine the joint strength and the effects of the additives on the joints were interpreted.

2. Materials and Methods

2.1. Materials and Properties

DX51D+Z galvanized steel material (EN 10346:2015) with dimensions of $100 \times 25 \times 1.5$ mm was used for the experimental study. The chemical compositions of the test samples are given in Table 1, and their mechanical properties are given in Table 2.

Araldite 2015 Huntsman was used as the adhesive material (an intermediate-stiffness epoxy adhesive). The properties of the adhesive are given in Table 3.

Table 1. Chemical composition of steel (% by weight).

C	Mn	P	S	Si	Al	Cu	Ti
0.06	0.3	0.019	0.022	0.02	0.032	0.04	0.002

Table 2. Mechanical properties of the steel.

Hardness	Yield Strength (MPa)	Tensile Strength (MPa)	Elongation at Break %
56 HRB	319	409	25

Table 3. Mechanical properties of Araldite 2015 [31].

Young's modulus (MPa)	1850 ± 0.21
Poisson ratio	0.33
Tensile yield strength (MPa)	12.63 ± 0.61
Tensile strength (MPa)	21.63 ± 1.61
Shear modulus (MPa)	560 ± 0.21
Shear yield strength (MPa)	14.6 ± 1.3
Shear strength (MPa)	17.9 ± 1.8

Three different types of powders, namely mussels, olive pomace, and walnut powders, in two different sizes of 38 µm and 45 µm were used as additives. The grinded powders of 38 µm and 45 µm are given in Figure 1 as olive pomace, walnut, and mussel, respectively. For mussel powders, waste mytilus galloprovincialis shells were used. A hardness of 3.5 Mohs to 4.0 Mohs is acceptable for mussel shells [32].

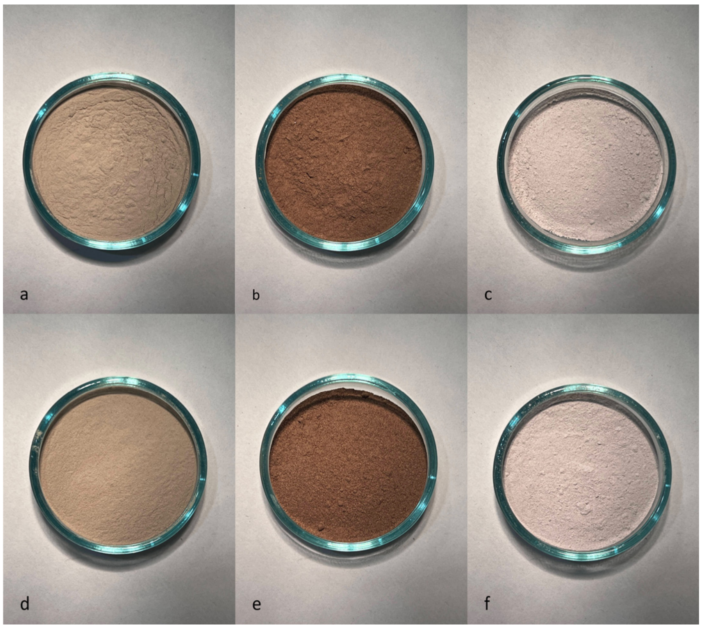


Figure 1. Ground 38 µm (**a**) olive pomace, (**b**) walnut, and (**c**) mussel powders and ground 45 µm (**d**) olive pomace, (**e**) walnut, and (**f**) mussel powders.

Calcium carbonate is a commonly used filler in polymer material. While the chemical composition of mussel shells contains 95.7% CaO, this ratio is 99.1% in commercial $CaCO_3$. Since mussel shells contain a similar amount of CaO as commercial $CaCO_3$, it is appropriate to be used as an additive material [33,34].

Olive pomace was obtained from a company operating in the Aegean region. Walnut shells were also collected from people who consumed walnuts. Olive seeds and walnut shell are lignocellulosic in chemical structure. Olive pomace contains 40% cellulose and 19% lignin in its structure [35]. Lignin content in walnut shells is around 30% [36]. Walnut Sheel has a specific gravity of 1.2–1.4 and a hardness of 3–3.5 MOH [37,38].

After the waste materials were dried in the oven, they were ground in a ring mill and sieved in a sieve shaker. The amounts of organic additives added to the adhesive were determined as 5%, 15%, and 30% by weight.

Infrared spectra were recorded on a Perkin–Elmer Spectrum 100 FTIR spectrophotometer with an attenuated total reflection (ATR) accessory featuring a zinc selenide (ZnSe) crystal at room temperature.

Since walnuts and olive pomace are organic structures, -OH and -CH groups are observed (3300 cm^{-1} OH and 2900 cm^{-1} CH). For organic powders, the OH groups can be hydrogen bond-promoting groups. Likewise, alkene groups (C=C) in the 1610s, amine groups in the 1230s, and aliphatic CO groups in the 1028s are observed in organic structures (Figure 2).

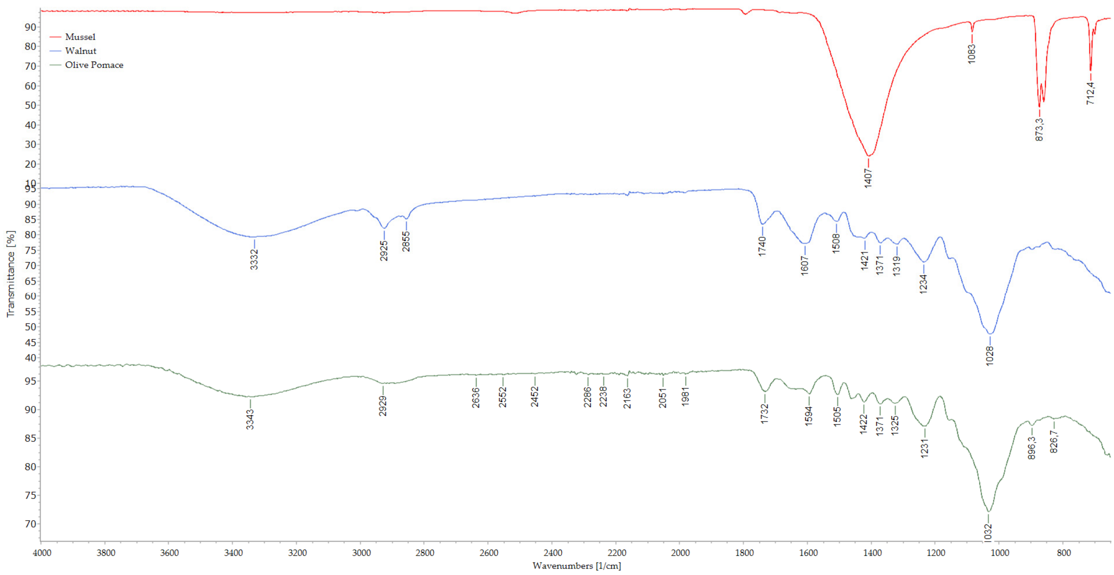


Figure 2. FTIR spectroscopy of organic powders (mussel, walnut, and olive pomace).

For mussels, the carbonate $CO_3{}^{-2}$ groups are observed around 1407 cm^{-1} and 873 cm^{-1} band. These findings support the calcium carbonate structure for mussel powder.

2.2. Joint Geometry

The type of joint used in the experiments was the single-lap joint model given in Figure 3. The usual test for this type of joint is the ASTM D 1002 [39].

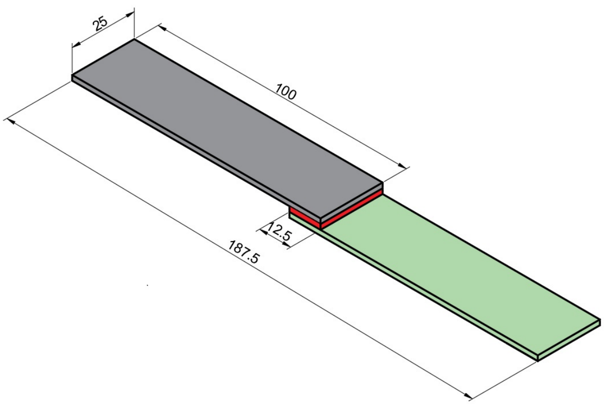


Figure 3. Single-lap joint type (mm).

2.3. Surface Preparation

Mechanical cleaning was conducted to prepare the surface of the adhesive joints. The surfaces of the samples were sanded with 120 SiC sandpaper. To roughen the entire bonding area, the sanding process was carried out in the bonding area in the horizontal and vertical directions, respectively. The sanded surfaces were wiped with acetone, washed with distilled water, and dried. The adhesive material and the filler powder were mixed manually in a plastic plate and then applied on the joint surfaces with the help of a spatula [40–42]. In the bonding area, metal paper clips were placed opposite each other, and the required pressure was provided. The bonding thickness of the joints was measured as 0.1 mm using mechanical caliper.

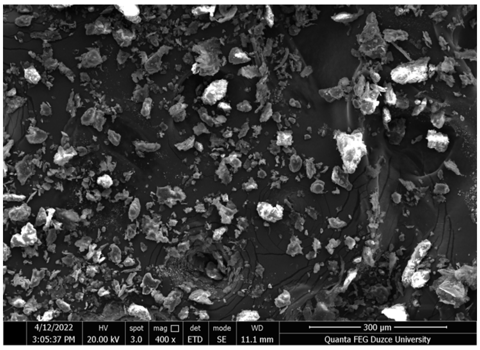
2.4. Surface Roughness and Tensile Testing

A Mitutoyo brand SJ-301 type desktop profilometer device with a digital display was used for surface roughness measurements. Average surface roughness values Ra were obtained by taking the arithmetic average of the five measurement values taken from the surfaces, according to the EN ISO 21920-2 standard [43].

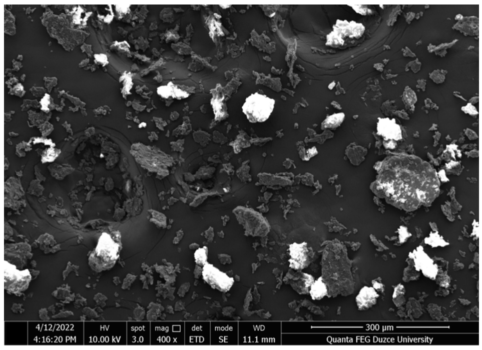
Tensile tests were carried out at room temperature at a constant crosshead rate of 1 mm/min using the ALSA tensile test machine. All experiments were performed in triplicate.

2.5. Surface Morphology Analysis and Characterization (SEM, Joint Interfaces)

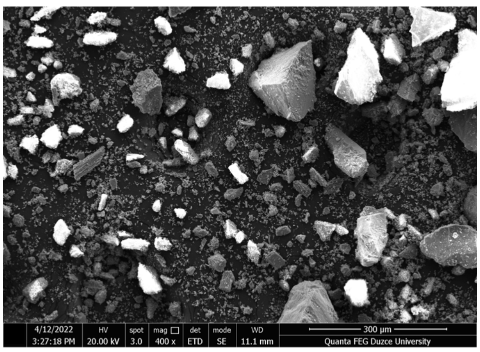
In order to better understand the strength results of the joint formed as a result of the bonding, images were taken of the powder materials and joint regions, and they were examined. All the samples were coated with gold palladium. Images were taken with a scanning electron microscope (SEM). SEM images of the powders are given in Figure 4, and joint interface images are given in Figure 5.



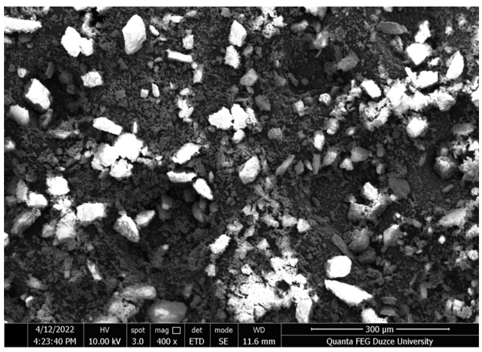
Walnut 38 µm



Walnut 45 µm

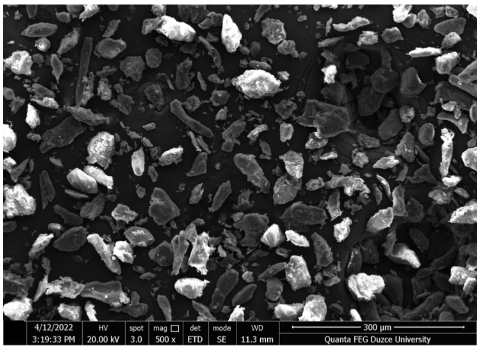


Mussel 38 µm

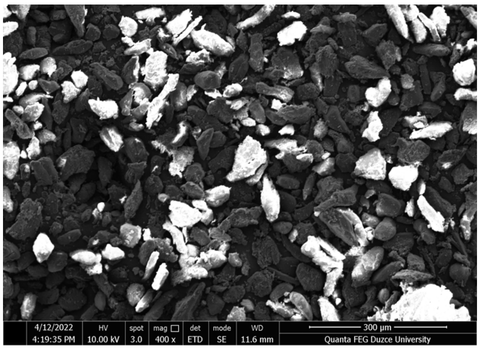


Mussel 45 µm

Figure 4. *Cont.*

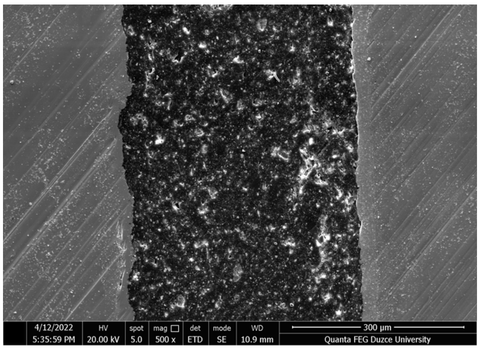


Olive pomace 38 µm

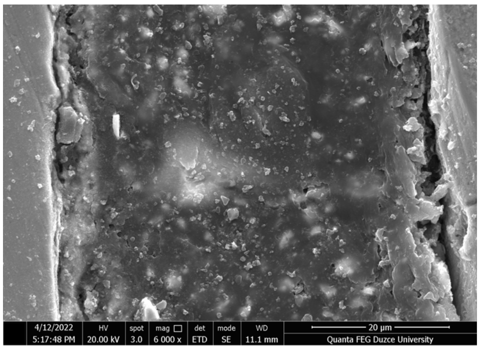


Olive pomace 45 µm

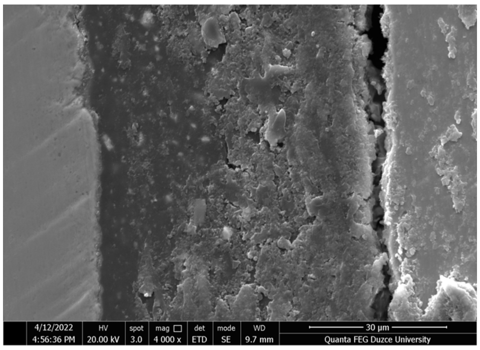
Figure 4. SEM images of powders.



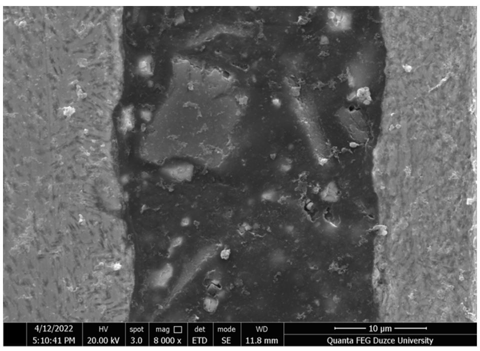
Pure adhesive



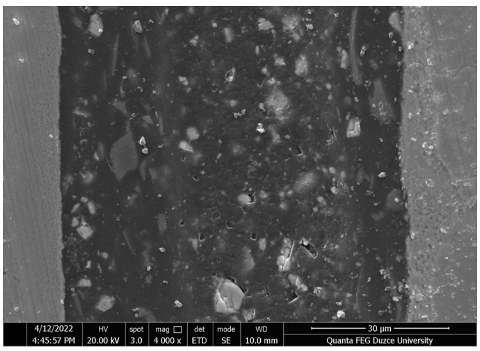
38 µm Olive pomace 30%



45 µm Olive pomace 30%

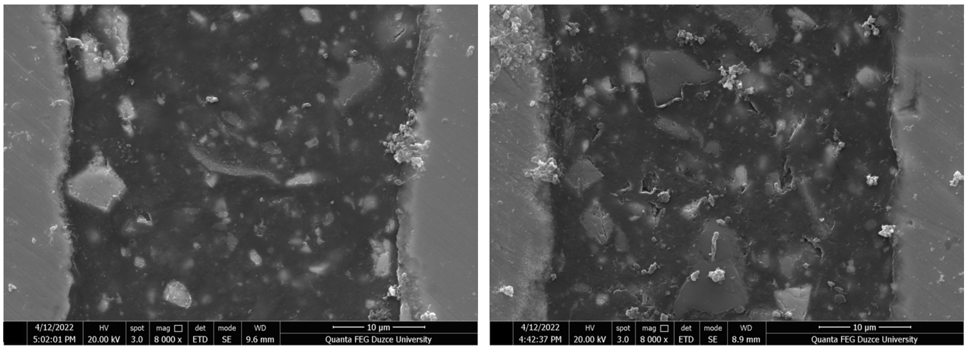


38 µm Walnut 5%



45 µm Walnut 5%

Figure 5. *Cont.*



38 μm Mussel 5% 45 μm Mussel 30%

Figure 5. Interface images of bonded joints.

The distributions of the powders at the interfaces of the bonded joints are shown in Figure 5. The SEM images given in Figure 5 were selected from the experiments to explain the joint strength values.

3. Results and Discussion

3.1. Single-Lap Shear Tests

The tensile tests were carried out for the bonded test samples. The tensile test graphs are given in Figures 6 and 7. The average shear strength of the bonded joint obtained using pure adhesive was found as 12.24 N/mm^2. It is known that the bond strength decreases when the amount of powder added to the pure adhesive is higher than a certain amount (threshold value). In the studies verifying this statement, metal powders are generally used as the additives [44]. The results of the experiments performed using olive pomace, walnuts, and mussels with a size of 38 μm are consistent with the literature. On the other hand, when the powder size increases to 45 μm, there is an effect of increasing-decreasing-increasing strength as the amount of additive increases. Accordingly, it is understood that there is a threshold value for the amount of powder added to the adhesive.

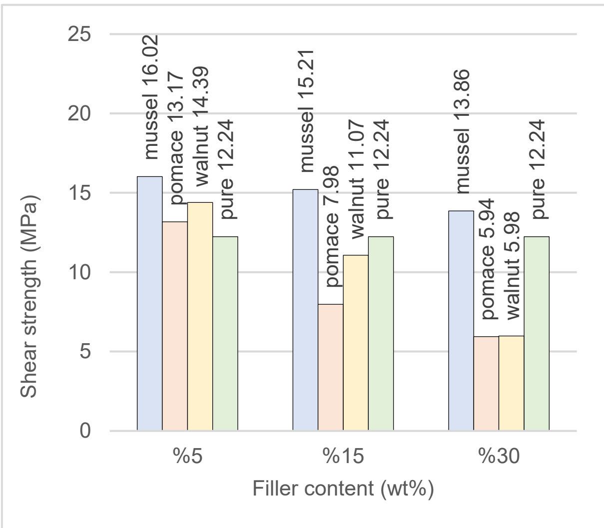


Figure 6. Average shear strengths of the 38 μm adhesive joints.

When the size of the powder added to the adhesive was 45 μm, bond strengths increased for all additive ratios compared to the pure adhesive. The powder-added bonded joints (in 45 μm size) provided better bond strength overall.

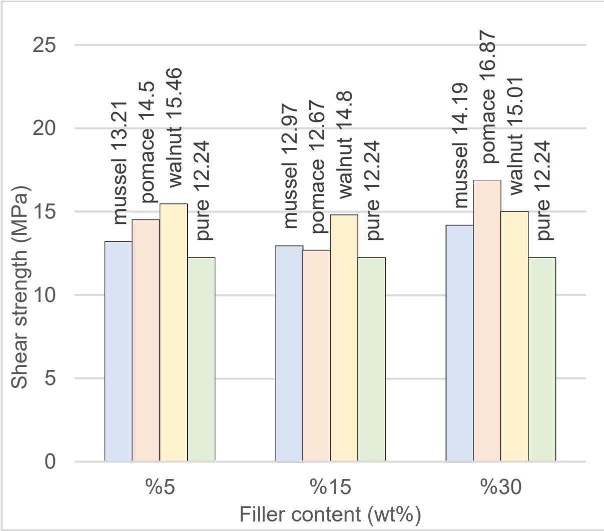


Figure 7. Average shear strengths of the 45 µm adhesive joints.

In the experiments, the lowest strength value was measured as 5.94 MPa in joints where 38 µm olive pomace was added in the ratio of 30% by weight, and the highest strength value was measured as 16.87 MPa in joints where 45 µm olive pomace was added in the ratio of 30% by weight. In addition, when the effect of olive pomace and walnut additives added by 30% on the joint strengths was examined, it was seen that the increases in the joint strength were close to each other. It can be assumed that the organic structural similarity of olive pomace and walnut materials resulted in similar increase amounts.

The mussel additive produced a higher strength value compared to the pure adhesive in all ratios without any size difference. This indicates that a strong bond was formed between the mussel shell and the adhesive. It can be said that the layered natural structure of the mussel shell strengthens this bond [26].

As can be seen from the graphics in Figures 6 and 7, the bond strength varies considerably depending on the type of additive (especially for 38 µm powder). Although the same grinding processes were carried out, there were changes in shape due to the type (structural properties) of the powders. The shape changes can be seen from the SEM images in Figure 4. This may be a parameter that changes the ability of the additive powders to adhere to the adhesive. This situation explains or is affected by the change in the strength values of the joints.

3.2. Surface Roughness Measurement

A surface roughness measurement was taken from the surfaces of the galvanized steel material that was mechanically abrasive using 120 SiC sandpaper, and the average Ra value was found to be 1 µm.

3.3. Surface Morphology Analysis and Characterization

In the visual examination of the powders, there was no difference other than color separation. However, it was seen that there were significant differences when SEM images were taken. Mussel powders are brittle, olive pomace powders are round, and walnut powders have a fringed fractured surface. When the SEM images in Figure 5, taken from the joints where 38 µm olive pomace was added in the ratio of 30% by weight, are examined, it is seen that there is a large void where the olive pomace added adhesive contacts the lower and upper surfaces of the base metal. This void is also seen along the joint interface. Therefore, it is understood that adhesion is not fully realized. Moreover, it is understood that the void spreads over the base metal surface into the bonding zone. Mixing the adhesive and the 38 µm olive pomace with each other at this rate reduced the adhesion effect of the adhesive.

When the SEM images taken from the joints where 45 μm olive pomace was added in the ratio of 30% by weight are examined, it is seen that there is a void where the adhesive contacts the metal surface. However, the width of this void is small.

4. Conclusions

By modifying the adhesives with a natural filler, a strong joint can be obtained, which can improve the parameters of the adhesive joint in terms of mechanical properties. The use of waste materials as additives after recycling makes it possible to reduce the production cost. The research follows the future trend in the field of ecological composites with fillers (or powder) based on waste material. Olive pomace, mussels, and walnut shells are recyclable organic waste materials. In this study, recycled organic materials in 38 μm and 45 μm sizes were added to the adhesive in the ratios of 5%, 15%, and 30% by weight. Single-overlap joints were formed by combining the steel materials with the modified adhesives which the ground powders were added to.

The results of tests to determine the mechanical properties of the adhesive filled with organic powders are presented. All results were compared with those obtained in the adhesive without powder additions.

The findings obtained as a result of the experimental studies are summarized as follows:

- The bond strength decreased when the amount of the powder (in 38 μm size) added to adhesive was more than 5%.
- The strength of all joints obtained by 5% powder additives (for 38 μm) increased compared to that with pure adhesive.
- The bond strength at all additive ratios (for 45 μm powder) increased compared to that with pure adhesive.
- In the experiments, the lowest strength value was obtained in joints where 38 μm olive pomace was added in the ratio of 30% by weight, and the highest strength value was obtained in joints where 45 μm olive pomace was added in the ratio of 30% by weight.
- When the additives in 38 μm powder size were used, it was seen that the change in bond strength varied depending on the powder type, while the effect of powder type on the bond strength did not make a difference in 45 μm powder sizes.

Author Contributions: The study was carried out as a two-person team. Conceptualization and methodology was introduced by N.A.; data curation, N.A. and Z.D.; writing—original draft preparation, N.A. and Z.D.; writing—review and editing, N.A. and Z.D.; visualization, Z.D.; supervision, N.A.; project administration, N.A. and Z.D.; funding acquisition, N.A. and Z.D. All authors have read and agreed to the published version of the manuscript.

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