Adherability and weldability of poly(lactic acid) and basalt fibre-reinforced poly(lactic acid)

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Abstract

The adherability and weldability of pure poly(lactic acid) (PLA) and basalt fibre-reinforced PLA were investigated in this research. The joining efficiency rate is introduced as a comparative parameter among different joining processes. In the case of adhesive bonding, 16 different adhesives were used to join specimens together. The highest bond strength and joining efficiency rate for both the pure (16 MPa, 78%) and basalt fiber-reinforced (18 MPa, 44%) adhesive-bonded specimens was achieved with acrylate-based two-component adhesives. The bond strength and joining efficiency rates of bonded specimens manufactured with four welding technologies (hot gas welding, friction stir welding, ultrasonic welding, laser welding) were also investigated. The highest bond strength for both pure PLA and basalt fibre-reinforced PLA specimens (51 MPa and 125 MPa, respectively) was attained by laser welding. The highest joining efficiency rate for pure PLA specimens (85%) was attained by ultrasonic welding, while it was achieved by laser welding for basalt-fibre reinforced PLA specimens (70%).

1. Introduction

Poly(lactic acid) (PLA), both in its solid and foamed form is mainly utilised by the packaging industry because of its gas and aroma-sealing properties. The final product can be transparent and also grease- and oil-resistant. PLA can be used to produce bottles, disposable cutlery, biodegradable bags and even clothes that can be used at room temperature because of its acceptable mechanical properties [1-3]. PLA is often used as the base material of absorbable stitches and retainers (mainly screws and grafts that facilitate tissue growth), as well as controlled drug delivery systems [4, 5]. Nowadays PLA is also increasingly used to manufacture engineering products, although its rigidity and high shrinkage limit its use for products which are exposed to high stresses [1-3]. Much research is being done to find additives and reinforcements that can be used with PLA to broaden its applicability.

The automotive industry has significantly transformed in recent decades, in fact manufacturers use far more polymer-based parts in their vehicles mainly to reduce weight. A general demand is that these parts must be strong and have high load-bearing capacity at the same time [6], while being easily recyclable, or even biodegradable. One material that fulfils these requirements is PLA, although it must be properly stabilised with flame retardant additives as its flame resistance is poor. Wang *et al.* [7] investigated the possibilities of increasing flame retardancy in PLA with traditional flame retardant additives, and found that these impaired mechanical properties. However, they also found that flame retardancy can be greatly improved with a mixture of traditional flame retardant additives and nano-sized fillers, for example montmorillonite or carbon nanotubes.

Besides flame retardancy, it is necessary to ensure that the mechanical properties of PLA meet the requirements imposed by the conditions of use. This can usually be achieved by mixing fibrous reinforcement, for example glass or carbon fibres into the base material. Attention has shifted towards the reinforcement of PLA with natural fibres in recent years. This way, the resulting composite is made entirely of renewable sources [8-15]. One possible natural reinforcement to use is basalt fibre. Added to polymers, it not only reinforces the material mechanically, but also increases flame retardancy [16, 17]. Tábi *et al.* [18] manufactured injection moulded, partially-crystalline PLA specimens with various basalt fibre filler content. During the investigation of these specimens, they found that basalt fibres acted as nucleating agents, which positively influenced the thermal dimension stability of PLA.

Niaounakis [19] thoroughly investigated the applicability of PLA-based products and composites in multiple industries, including the automotive industry. Some of the parts that can be made from PLA inside a vehicle are the dashboard, door panels and floor mats. Covers and energy absorbing parts on the inside and bumpers and lightbulb casings on the outside of the vehicle can be manufactured from natural or glass fibre-reinforced PLA. The fastening of these parts to each other or to the chassis of the vehicle can be best done by welding or with adhesives, since with these technologies a large number of complex structural elements can be manufactured in a short period of time. These technologies include bonded joints and welded joints but while composites with a cured matrix can only be bonded with an adhesive, composites with a thermoplastic matrix can be welded, too [20, 21].

The aim of this research paper was to investigate the strength of bonded and welded joints of basalt fibre-reinforced PLA compared to the strength of pure PLA. During our investigations, we examined the adhesive bonding technology and some of the most widely used reactive and non-reactive adhesives in the industry [22-24]. We investigated some welding technologies, too.

Vogel *et al.* [25] welded thin sheets of PLA together with ultrasonic and impulse welding and found that with both technologies, joints with great strength can be produced. They also found that during impulse welding, increasing weld time and thus the amount of heat put into the weld increased the strength of the joint to a certain point, but after that point, joint strength started to decrease. During ultrasonic welding they found that the strength of the joint can be improved by increasing the weld force or the amplitude of vibrations. Heidenreich *et al.* [26] investigated the possibilities of using parts made from PLA in medical applications. In their research, they joined screws made from PLA to animal bones using an ultrasonic welding machine. They found that when the screws were put into place and fastened, heat – generated by friction between the screw and the bone during fastening – melted the material of the screws to some extent, thus a greater bond strength between screw and bone was achieved. Zhang *et al.* [27] investigated the effect of the parameters of ultrasonic welding on the bond strength of PLA and PMMA sheets joined together with ultrasonic welding. Before welding, a sheet of PLA/PMMA (manufactured in an injection moulding machine in 1:1 ratio) was fastened to the sonotrode of the ultrasonic welding machine, between the pure PLA and the pure PMMA sheets. The bond was created by melting both the base materials and the PLA/PMMA sheet together in the ultrasonic welding machine. They found that the tensile strength of the specimens created with this technique reached 90% of the tensile strength of pure, unwelded PLA sheets. Pagano *et al.* [28] joined aluminium films to thin sheets of PLA with laser transmission welding and examined the strength of the joints. They found that optimal strength of the bonds was achieved when the power output of the laser was high, because in this case more heat was generated on the PLA-aluminium boundary, which increased bond strength.

The goal of this research was to examine various widely used joining technologies to join pure PLA sheets together and basalt fibre-reinforced PLA sheets together. These technologies include bonding with reactive and non-reactive adhesives, friction stir welding (a popular technology with composites), hot gas welding (this is often applied in corrective welding), and ultrasonic and laser welding (these are extensively used in the automotive industry).

2. Materials and Methods

2.1. Materials

The base material was Ingeo™ Biopolymer 4032D PLA made by NatureWorks LLC (Blair, NE, USA). 30 wt% of Basfibre® BCS TDS VK12 (Kamenny Vek, Dubna, Russian Federation) type basalt fibre was also added on a Labtech LTE 26-44 type dual-screw extruder (Samutprakarn, Thailand) to one half of the PLA batch. From now on this composite is marked as PLA+30BF. The pure, unreinforced half of the PLA batch was also processed on the same extruder with the same process parameters in order to assure that both batches had the same thermal history. After pelleting, sheets with a base area of 80x80 mm and a thickness of 2 mm were manufactured on an ARBURG Allrounder Advance 370S 700-290 (Loßburg, Germany) injection moulding machine.

Some of the reactive and non-reactive adhesives that are frequently used in industrial applications [21-23] were used to join PLA and PLA+30BF sheets together, these are listed in Table 1.

Single-component adhesives	Loctite SI 5910, Teroson MS 939, Teroson PU92
Two-component adhesives	Loctite EA 3430, Loctite 330+7386, Loctite Chemical Metal, 3M DP410, 3M DP8010, 3M DP8405
Cyanoacrylate adhesives	Loctite 401, Loctite 406, Loctitce 454
Dispersion adhesives	Pattex PL50, Pattex Palmatex
Solvent-based adhesive	Terokal 2444
Melt adhesive	Technomelt 5355

Table 1. Classification of the investigated adhesives

2.2. Adhesive Joining Technology

16 different adhesives (as shown in Table 1) were used to join specimens. 25 mm-wide specimens were cut from the original, injection moulded specimens and were cleaned and degreased with Loctite 7063 liquid cleaner. Overlapped joints were formed, with an overlap distance of 12,5 mm (~0,5 inch), as seen in Figure 1. In each case, the amount of adhesive used to form the joint was chosen based on the recommendation of the manufacturer of the adhesive. We waited 24 hours before testing the adhesive-bonded joints, in order to let the adhesives fully cure.

2.3. Welding Technologies

The following welding technologies were investigated:

- Hot gas welding: mainly used in the automotive industry as it is fast, easy and cheap to operate [29].
- Friction stir welding: a novel, easy-to-automate technology to join polymer parts, or even polymer and other materials together [30-33].
- Ultrasonic welding: a widely used, productive joining technology that can be easily automated and controlled. The seams created with ultrasonic welding are generally of good quality [25-27, 34-36].
- Laser welding: the equipment is very expensive but it is easy to use and automate, and very accurate and precise [25, 28].

Specimens joined with ultrasonic welding were welded together with an overlapped joint. In the case of hot gas welding, friction stir welding and laser welding, butt joints were formed (see Figure 1).

For hot gas welding, a hot air welder made by Leister Technologies (Itasca, IL, USA) was used. The specimens were joined together with an "X" seam with PLA rods as filler material, which were melted by the hot air welder. The temperature of the hot air welder was set to the melting point of the PLA rods, 220°C.

The friction stir welding machine was custom-built, it was basically a milling machine mounted on an XY table. However, the direction of the turning of the milling tool was reversed, so that it did not cut the materials, but joined them by mixing the material of two sheets together. The speed of the milling tool was set to 2700 RPM, its diameter was 6 mm and the feed rate (the speed of the XY table) was set to 84 mm/min.

For ultrasonic welding a Hermann HiQ Evolution 20 kHz ultrasonic welding machine (Karlsbad, Germany) was used. The ultrasonic welding process was energy controlled, the frequency of the vibrations was 20 kHz, the energy value was set to 300 Joules, while the amplitude of the vibrations was set to 100% and the welding force enacted by the sonotrode on the welded parts was set to 200 N.

The laser welding machine was a Universal Laser Systems UL 25 OEM type machine (Scottsdale, AZ, USA) equipped with a CO_2 laser source and with a 150 mm focal length lens. During laser welding two thin sheets of specimens were pressed together at their 2 mm-wide edge (as shown in Figure 1) by a spring-loaded locking system (force enacted by the springs on the sheets: 10 N). During welding, the power of the laser beam was set to 25 W, while the speed of the optics of the welding machine was set to 10 m/s. In a welding cycle, the optics of the welding machine ran back and forth above the adherends 100 times, creating the seam.

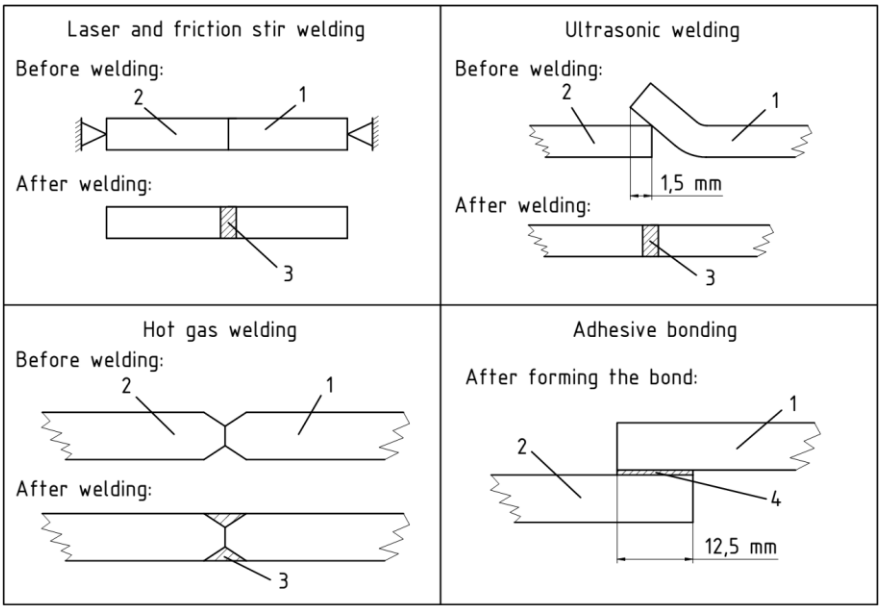

Figure 1. The illustration of bonds (Legend: 1-2 Adherends, 3 Bond, 4 Joint)

2.4. Measurement Methods

A Zwick Z020 (Ulm, Germany) universal testing machine was used to investigate the mechanical properties of the welded and adhesive bonded joints at room temperature. The most important test parameters are shown in Table 2. Test speeds were chosen according to the recommendations of relevant standards.

Bonding technology	**Type of bond**	**Test type**	**Test speed [mm/min]**
Hot gas welding	Butt joint	Three-point bending	10
Friction stir welding			
Laser welding			
Ultrasonic welding	Overlapped joint	Tensile test	5
Adhesive bonding			2.5

Table 2. Test speeds of PLA and PLA+30BF specimens

Joints were characterized and ranked by their joining efficiency rate. It was determined as a proportion of two strength values: in the case of pure PLA specimens, joining efficiency rates were specified by dividing the strength value of a bonded specimen by the strength value of a single, injection moulded thin sheet of pure PLA specimen. In the case of basalt fibre-reinforced PLA specimens, joining efficiency rates were specified by dividing the strength value of a bonded, basalt fibre-reinforced specimen by the strength value of a single, injection moulded thin sheet of basalt fibre-reinforced PLA specimen. Joining efficiency rates described in Chapter 3 were calculated as the average of three measurements, and their standard deviation was also calculated from the same three measurements. Joining efficiency rates make it possible to compare different joining techniques. In this paper all joining efficiency rates are published as a percentage (1).

$$Joining\ efficiency\ rate = \frac{strength\ of\ bonded\ specimen}{strength\ of\ specimen\ without\ bonds} * 100\ [\%] \quad (1)$$

3. Results and Discussion

3.1. The Results of Adhesive Bonding Methods

Uniform overlapped bonds with a joint area of 25x12,5 mm (1x0,5 inch) were formed to investigate the strength of adhesive bonded pure and basalt fibre-reinforced PLA sheets. For both the pure and the basalt fibre-reinforced PLA, the best joining efficiency rates were attained when acrylate-based (two-component and cyanoacrylate) adhesives were used to form the joint, as can be seen in Figure 2. A dividing line was drawn in Figure 2, which indicates the border between cohesional (see Figure 3) and adhesional (see Figure 4) failure forms of the tested joints.

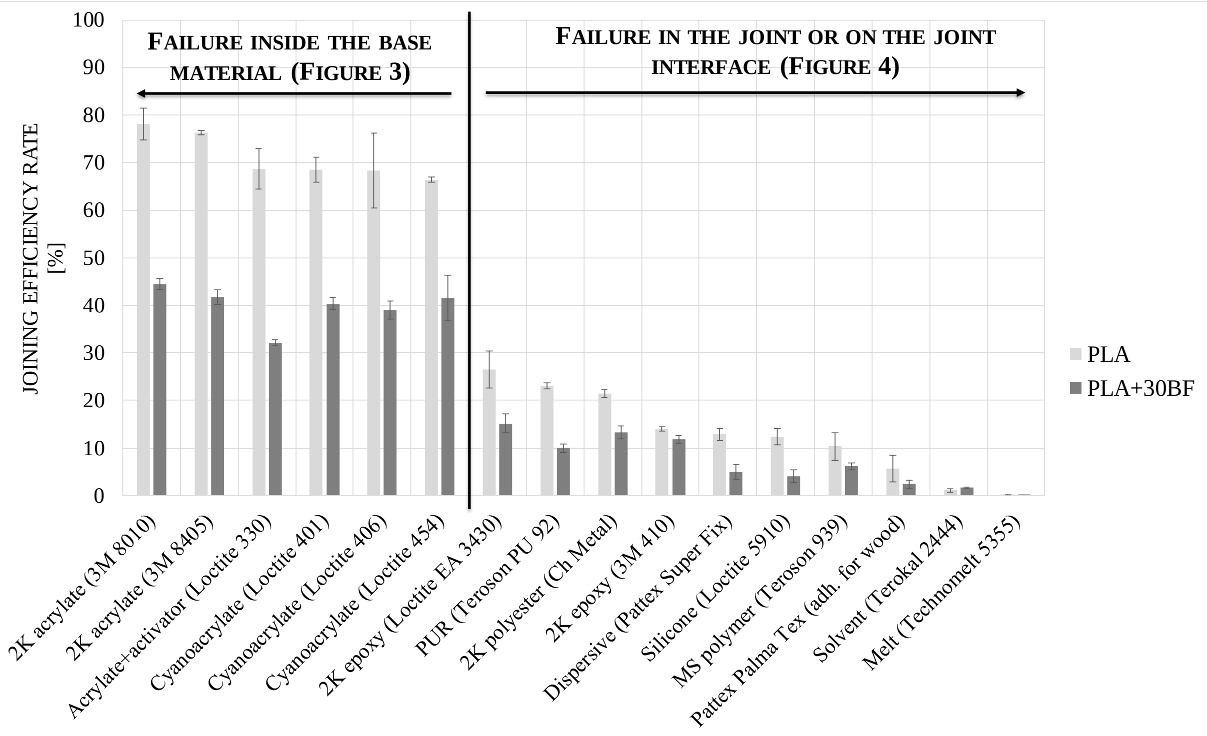

Figure 2. Joining efficiency rates of adhesive bonded specimens

As can be seen in Figure 3, joints made with two-component acrylate-based and cyanoacrylate adhesives had great cohesion: failure was observed almost always in the base material and only rarely in the adhesive. On the other hand, joints manufactured with dispersion, melt, solvent-based and single-component adhesives were found to be weak: the characteristic failure of these specimens was adhesional failure of the joint, which meant that the adhesive peeled off the PLA sheets, without the sheets being broken because of the tensile stress (as shown in Figure 4). It can also be observed that in most cases of adhesive joining, the standard deviation of the measured values was found to be lower than in the case of welding technologies (see Figure 5). This meant that joints made with two-component adhesives (for example with 3M 8405 or Loctite 401) were easily reproducible. The great difference between the joining efficiency rates of pure and basalt fibre-reinforced PLA can be explained with the fact that the adhesives did not contain basalt fibres, therefore the strength of the joint was the same for all the specimens (both pure and reinforced). However, the strength of the base material – which was also used in the calculation of joining efficiency rates – was largely increased in the case of basalt fibre-reinforced PLA specimens.

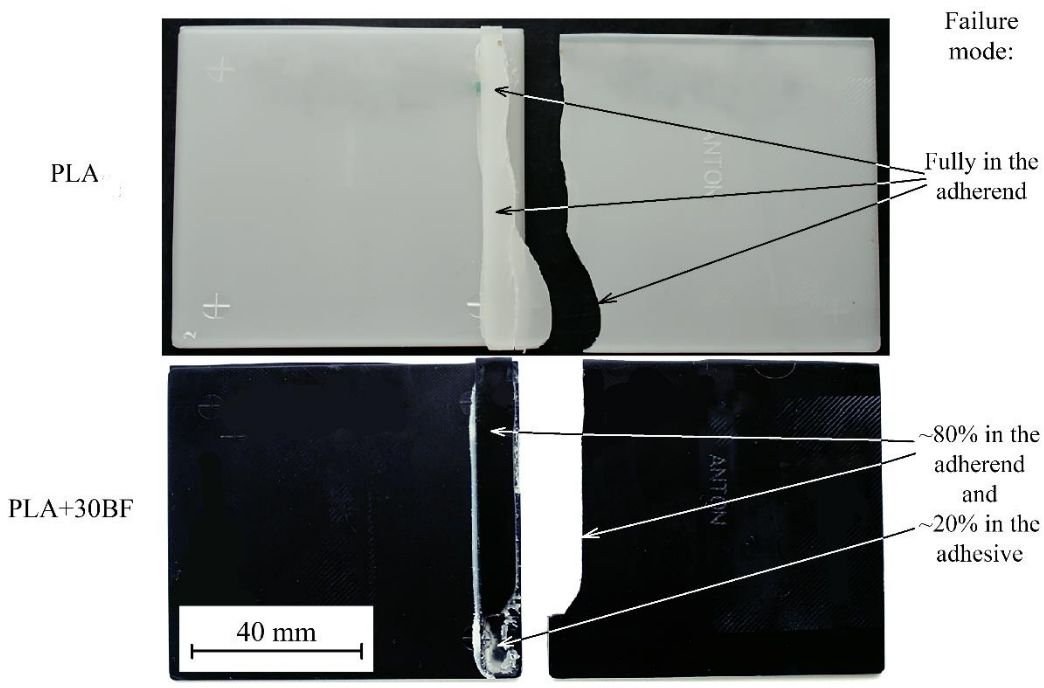

Figure 3. Tensile tested thin sheets of pure PLA and PLA+30BF joined together with acrylate-based adhesives

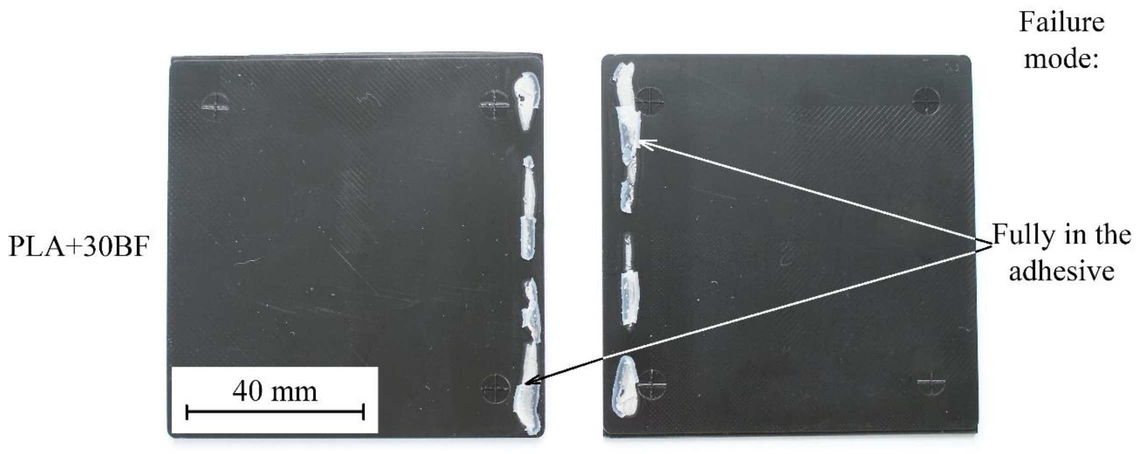

Figure 4. Tensile tested thin sheets of PLA+30BF joined together with a dispersion adhesive

It is also important to note that in selecting the best adhesive for a product to be bonded, it is not always a priority to attain the highest possible joint strength. Adhesive joints can be flexibly deformed and have some vibration dampening effect, which can be important parameters during an adhesive vetting procedure.

3.2. Joining Efficiency Rates of Welded Joints

Specimens joined with hot gas welding were made with an „X” type seam. As can be seen in Figure 5, these joints failed at about half of the stress that was needed for the failure of the pure, unbonded specimens. Pure and basalt fibre-reinforced specimens did not behave differently in this respect. This was because during welding there was no material mixing between the two specimens. The joint was made in both cases with the added PLA material, which did not contain basalt fibres, therefore it could not make an impact on the strength of the seam.

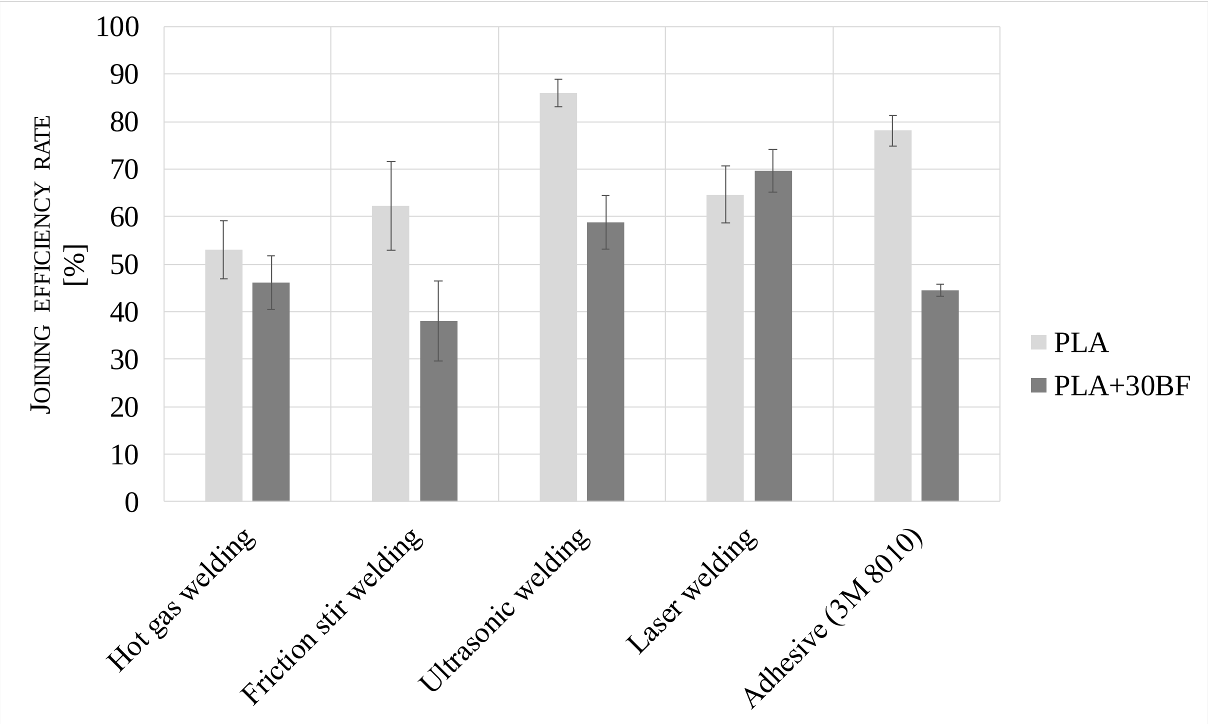

Figure 5. Average joining efficiency rates of the best-performing welded bonds and adhesive-bonded joints

In the case of friction stir welding, higher joining efficiency rates were measured for the pure PLA specimens, which contradicted expectations. This was because the tool of the friction stir welding machine broken the long basalt fibres into smaller pieces, which could not go over the barrier between the base material and the seam. This meant that the fibres could not make an impact on the strength of the seam. The effect of this phenomenon can be seen in Figure 6, which depicts a basalt fibre-reinforced friction stir welded specimen with long basalt fibres in the base material, short, broken basalt fibres in the seam and no fibrous connection through the border between the base material and the seam.

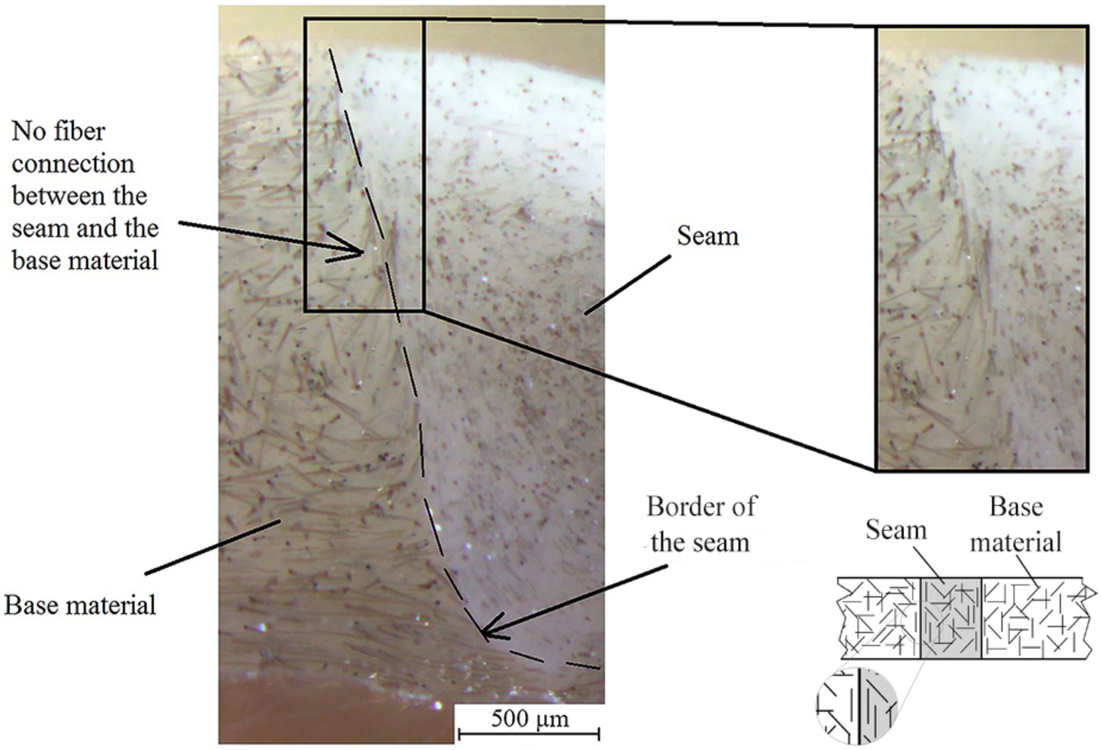

Figure 6. Friction stir welded thin sheet made of PLA+30BF

In ultrasonic welding the sheets were overlapped (as seen in Figure 1). It was found that the best joining efficiency rate (about 80%) could be achieved for pure PLA sheets with ultrasonic welding (Figure 5). However, a significant decrease in joining efficiency rate was observed for the sheets reinforced with basalt fibres. This was due to the vibration dampening property of basalt fibres that were distributed in the PLA base material: this diminished the plasticizing effect of ultrasonic vibrations. This led to less heat generated inside the base material, thus cohesion and mechanical properties of the seam worsened and the strengthening effect of basalt fibres could not compensate for this. The standard deviation of measurement data was found to be quite high both for pure and basalt fibre-reinforced, ultrasonic welded PLA specimens, which was caused by the fact that the arrangement of basalt fibres inside the seam, and on the border of the seam and the base material was diverse. As a result, welded specimens often had different joint strength and quality, even with the same process parameters.

During laser welding, welded sheets were pressed together by their 2 mm wide edge. The strength of the seams was tested with three-point bending tests. For pure, welded PLA sheets, the joining efficiency rate of laser welding was found to be ~ 66%. The joining efficiency rate of welded, basalt fibre-reinforced PLA sheets exceeded that of the pure PLA sheets, as can be seen in Figure 5.

Joining efficiency rate is a perfect parameter to compare achievable joint strength between bonding technologies. However, joining efficiency rate is a ratio (1), thus it did not provide information about the increase in strength of the base material made possible by basalt fibre reinforcement. In Table 3, the measured strength values are presented: basalt fibres did have reinforcing properties, as the strength of PLA+30BF specimens was consistently higher than the strength of pure PLA specimens. Based on measurement data, it can be stated that basalt fibre-reinforced PLA can be best welded with laser welding: the strength of specimens containing 30% basalt fibres were found to be about two and a half times higher than the strength of pure PLA specimens.

Average strength of specimens [MPa]		PLA Bonded specimen	PLA Base material	PLA+30BF Bonded specimen	PLA+30BF Base material
Tensile strength (Tensile test)	Adhesive bonding (3M 8010)	16±1	20±1	18±1	40±2
	Ultrasonic welding	17±1		24±2	
Flexural strength (Three-point bending test)	Hot gas welding	42±7	80±4	82±15	180±9
	Friction stir welding	50±3		67±10	
	Laser welding	51±5		125±10	

Table 3. Average strength of PLA and PLA+30BF specimens

4. Conclusions

In this research paper, the adherability and weldability of PLA and its basalt fibre-reinforced composite was examined. In order to form the joint between two injection moulded specimens, 16 different adhesives and also the following welding technologies were used: hot gas welding, friction stir welding, ultrasonic welding and laser welding. Specimens with butt joints were welded using hot gas welding, friction stir welding and laser welding, these were examined with three-point bending tests. Specimens with overlapped joints were welded with ultrasonic welding, and other joints were also formed with the help of adhesives. The strength of the bonds of ultrasonic welded and adhesive bonded specimens was examined with tensile tests. The measured strength of bonded specimens was compared with the strength of their respective unwelded and unbonded specimens. This yielded a ratio that is called joining efficiency rate, and which can be used to compare bonding technologies.

Ultrasonic welding and adhesive bonding resulted in the best joining efficiency rates and joint strength in the bonding of pure PLA specimens. In the case of basalt fibre-reinforced PLA specimens, laser welding yielded the best joining efficiency rates and also the best joint strength. Joining efficiency rates were generally lower for basalt fibre-reinforced PLA composites, but these "all-natural" composites were manufactured solely from natural sources. Based on their measured joint strength, they could certainly be used in engineering applications.

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