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LCA of laser surface activation and traditional pre-treatments for adhesive bonding of engineering polymers

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Abstract

The use of engineering polymers for mechanical applications has seen increasing uptake due to properties such as low density, flexibility, ease of manufacturing and cost effectiveness. Despite these advantages, joining and assembly methods for these types of materials is still an open issue. Traditional assembly processes such as screw fastening and riveting are increasingly being replaced by new processes such as adhesive bonding. Engineering polymers, however, are difficult to bond using adhesives due to their low surface energy and low wettability. For this reason, surface chemical activation techniques with primers are often used. The utilization of various chemicals associated with such pre-treatments has a significant environmental impact. Within this context, the present paper aims to compare the environmental performance of four adhesive bonding pre-treatments: (i) mechanical (i.e., abrasion), (ii) chemical (i.e., primer), (iii) plasma and (iv) laser activation. The work was performed in three phases: (i) setup of the surface activation processes, (ii) mechanical characterization of bonded joints (static tests) and (iii) LCA analysis to evaluate and compare the different pre-treatments. The outcome of this study provides important insight into the development of laser and plasma technologies as sustainable surface activation methods for polymers through the creation of models correlating process parameters to the type of surface and joint strength.

Keywords: laser ablation; surface activation; polymers; adhesive bonds; sustainability; environmental impact; LCA.

1. Introduction

The use of engineering polymers for mechanical applications has seen increasing uptake due to properties such as low density, flexibility, ease of manufacturing and cost effectiveness. Despite these advantages, joining and assembly methods for these types of materials is still an open issue and traditional assembly processes such as screw fastening and riveting are increasingly being replaced by new processes such as adhesive bonding. Adhesion science and technology are of relevance in modern industrial joining processes as the use of adhesive bonding has become a key aspect in multiple sectors such as the automotive, aerospace and wood industries [1]. Joining and assembly methods based on adhesives provide a solution to several engineering issues by introducing: (i) the possibility to couple dissimilar materials, (ii) the ability to join thin materials with complex shapes, (iii) weight reductions, (iv) ease of manufacturing by avoiding machining operations for fasteners, (v) better stress distribution over the joint area, (vi) good fatigue resistance due to the reduction of stress concentration and (vii) improved aesthetic appearance [2]. There are also several drawbacks associated with the adoption of adhesives for assembly of engineering polymers, most of which are associated with environmental concerns. First of all, structural adhesives (known as petroleum-based adhesives) are derived from co-products of petroleum processing, which are non-renewable resources [3]. Adhesives are the main industrial source of volatile organic compounds (VOC), releasing

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formaldehyde vapors that contribute to the formation of photochemical air emissions and can harm human health [4]. End-of-life (EoL) management (i.e., recycling) is another important issue associated with the use of adhesives in mechanical assembly. A serious problem is related to the mixture of different polymers, leading to poor properties due to incompatibility between different types of polymers. The recycling of adhesives is even more challenging due to the fact that they represent a small proportion of a larger assembly, with the separation of components requiring chemical or thermal treatments [5]. These issues have been addressed from several points of view in the literature: (i) use of bio-based adhesives [6], (ii) adoption of sustainable bonding processes [7], (iii) application of non-toxic chemicals and non-carcinogenic substances [8] and (iv) development of reversible adhesives [9][10]. Life cycle analysis has been used to estimate the environmental burden and benefit associated with these new solutions in different contexts such as the wood, automotive and aerospace industries [11][12][13][14], coupling technical solutions provided through adoption of structural adhesives (i.e., weight reduction) with a reduction in fuel/energy consumption and consequent greenhouse gas emissions during the product use phase [15][16]. Consideration of energy savings during the use phase of energy-related products is relevant for products that use adhesives as a technical solution for assembly [17][18]. Thus, life cycle analysis is able to assess the environmental benefit introduced by the adoption of structural adhesives in mechanical applications, considering the benefits introduced by weight reductions and energy savings during use, which compensate for problems relating to recycling and disposal. On the other hand, assembly performance (i.e., mechanical strength) achieved with the adoption of adhesives requires validation from a mechanical point of view, with the design of polymeric joining surfaces necessary to apply adhesive solutions to mechanical products. Surface chemical activation techniques involving acids or primers are often employed to enhance adhesion performance (i.e., increase the surface energy of low wettability polymeric substrates). The utilization of various chemicals associated with such pre-treatments has a significant environmental impact. An alternative is the adoption of mechanical processes for surface preparation (i.e., sand blasting, pneumatic hammering, plasma treatment or laser irradiation); however, these can be energy-consuming, with associated greenhouse gas emissions [19]. For these reasons, surface engineering is gaining importance from a life cycle perspective, with novel frameworks proposed to couple life cycle engineering and surface engineering [20]. Thus, the environmental impact of surface preparation methods for mechanical joints needs to be investigated in relation to the mechanical behavior and design parameters (i.e., overlap length and quantity of adhesive).

Within this context, the present paper aims to compare the environmental performance of traditional surface activation techniques such as chemical and mechanical activation to alternative processes including laser irradiation and plasma treatment. The work was performed in three phases: (i) setup of the surface activation processes, in particular laser and plasma treatment of polymeric substrates, (ii) mechanical characterization of bonded joints (static tests) and (iii) use of the Life Cycle Assessment (LCA) methodology to evaluate and compare surface activation methods for adhesive bonding. The functional unit for the LCA analysis was defined as follows: "*the process of surface activation for adhesive bonding of engineering polymers that guarantees a static strength of at least 1400 [N] ±10%*". This specific value of static strength was chosen based on a series of preliminary tests with different joint configurations (i.e., overlap length) for each pre-treated adhesive bonded joint. The outcome of this study provides important insight into the development of laser and plasma technology as sustainable surface activation methods for adhesive bonding of polymers.

2. Materials and Methods

The project developed within this work consisted of using the LCA methodology to analyze the impact of different methods for surface activation prior to adhesive bonding. The overall project framework was structured into three phases, reported in Fig. 1, including: (i) setup of the surface activation process, (ii) mechanical characterization of the joints and (iii) LCA analysis.

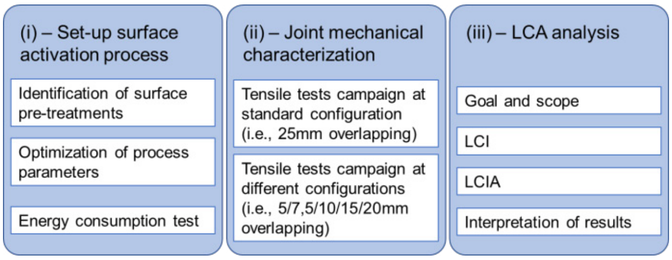

Fig. 1. Project methodology

In the first phase, activation processes for adhesive bonded joints were defined to investigate a set of different pre-treatments that could be adopted for mechanical applications. The considered pre-treatments included: (i) mechanical activation (i.e., abrasion), (ii) chemical activation (i.e., primer), (iii) plasma treatment and (iv) laser irradiation. These pre-treatments were compared with traditional bolted joints comprising a screw, nut and washer (Fig. 2), which were used as a reference to compare the mechanical performance of the two technologies.

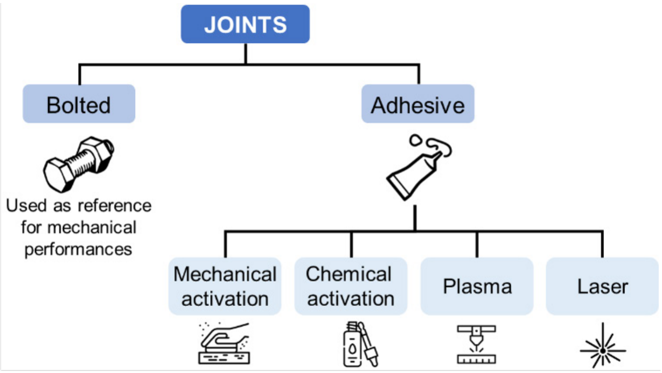

Fig. 2. Joints used in experiments

PA66 supplied by Ensinger Italia was employed as the polymeric material, while TEROSON PU 9225 supplied by Henkel® was used as the structural adhesive for joint formation. For mechanical activation, a belt sanding machine was employed with 320 grit aluminum oxide sandpaper in line with ASTM D 2093-03 [21]. For chemical activation, TEROSON 150, comprising mainly xylene and ethylbenzene, was used as a primer in line with the recommended procedure in the technical data sheet for the adhesive. Laser surface activation was instead carried out with a LaserPoint YFL 20P ytterbium doped fiber laser source capable of emitting 104 ns pulses with a wavelength of 1064 nm, repetition rate of 20 kHz and pulse energy of up to 850 μJ. A galvanometric scanning head was employed for beam movement with an f-theta lens of focal length 160 mm, achieving a focused spot size of 60 μm and a maximum scanning speed of 2500 mm/s. Finally, surface activation with plasma was performed with a Dierer PlasmaBeam atmospheric plasma system operating with air as the processing and cooling gas.

After identification of the surface pre-treatments, optimization of process parameters was carried out. This phase was particularly important for laser activation, where adhesive-bonded joint strength is a function of several parameters including scanning strategy, pulse energy, scanning speed and hatch spacing. Optimum parameters were determined with a Design-of-Experiments (DoE) for systematic variation of parameters over a viable operating window. This activity was performed with the methodology presented in a previous work relating to laser pre-treatment of polyethylene (PE) for adhesive-bonded joints [22]. Preliminary tests were performed to confirm the same viable process parameter window for PA66. The DoE was then performed with parallel line (PL) and crossed line (CL) laser scanning strategies over the same ranges presented in [22]. Best performance was achieved with a PL laser scanning strategy performed normal to the loading direction with a pulse energy of 150 μJ (average power 3 W, fluence 10.6 J/cm^2), scanning speed of 700 mm/s and hatch spacing of 50 μm. The parameters used to perform the plasma treatment were based on a previous work [23] where the effects of treatment speed and nozzle-to-substrate distance on the shear strength of PA66 bonded joints were studied. In particular, a treatment speed of 100 mm/s and nozzle-to-substrate distance of 5 mm were employed to prepare joints for this study.

Once all pre-treatment processes were characterized, energy consumption tests were performed with the aim of retrieving useful information for the inventory phase of the life cycle analysis. In the second phase, bonded joints were characterized by performing tensile shear tests in line with ASTM D 3163 [24] using overlap lengths (OL) of 5, 7.5, 10, 15, 20 and 25 mm to characterize the failure load of each pre-treatment as a function of overlap length. Fig. 3 shows the joint geometry and dimensions. Each one of the two substrates had a length (L) of 100 mm, width (W) of 25 mm and thickness (T) of 6.4 mm, while the adhesive thickness (Ta) was set to 0.3 mm.

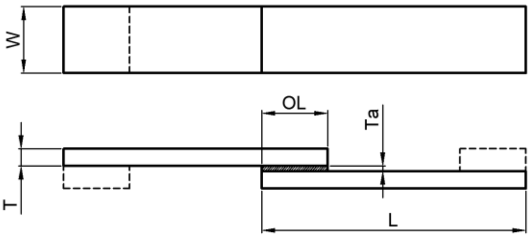

Fig. 3. Joint geometry and dimensions

Fig. 4 presents the failure load as a function of overlap length for adhesive bonded joints with different surface pre-treatments, as well as the failure load of the bolted joints used as a reference. The main objective of this phase was to set a minimum static load requirement (target load) that could be achieved by joints subject to all of the employed pre-treatments. In this specific case, the target load was set to1400 [N] (black dashed line in Fig. 4). Each test was repeated four times to ensure statistical robustness of the results. Static loads were recorded with an INSTRON 4460 tensile testing machine.

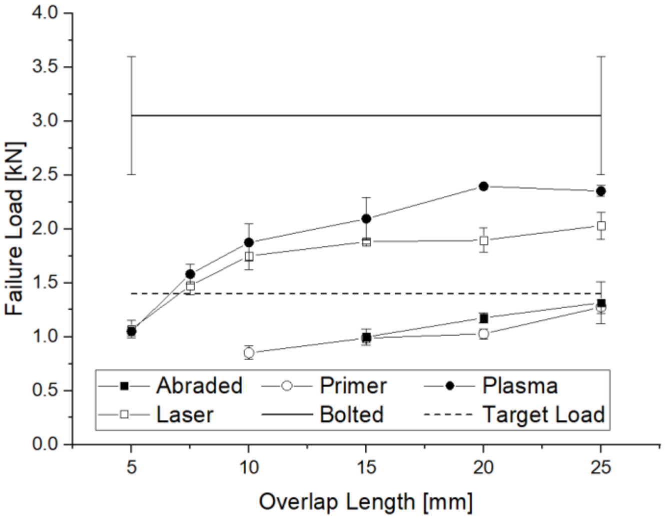

Fig. 4. Failure load vs. overlap length for different adhesive bonded joints

Table 1 presents the minimum overlap length required to achieve a static failure load of at least 1400 [N] ±10% for each pre-treatment. It can be noted that the chemical and mechanical activations required a larger overlap length than the laser and plasma pre-treatments.

Table 1. Minimum overlap length for each pre-treatment (based on target load of 1400 [N]).

Pre-treatment	Overlap Length
Chemical activation (primer)	25 mm
Mechanical activation (abrasion)	25 mm
Plasma	7.5 mm
Laser	7.5 mm

In the third phase, LCA analysis was performed to compare the environmental performance of the different surface activation techniques. The analysis was performed in line with the ISO and ILCD Handbook [25]. According to this methodology, the functional unit was defined as: "*the process of surface activation for adhesive bonding of engineering polymers that guarantees a static strength of at least 1400 [N] ±10%*".

The system boundaries included the following phases: (i) material extraction, (ii) manufacturing and (iii) use. The end-of-life, transportation and maintenance phases were considered as being outside system boundaries. In particular, the maintenance and transport phases were considered to be the same for all configurations and were therefore neglected. For the end-of-life phase, the environmental burden is strictly related to the product under investigation (i.e., WEEE, ELV), for which dedicated considerations are required. In addition, end-of-life analysis would be beneficial in the comparison of different bonding technologies (i.e., adhesive vs. mechanical joint). Indeed, additional considerations are necessary, as a bolted joint is one of the most sustainable joint solution considering the possibility to disassemble components at the end-of-life [28]. For the life cycle inventory (LCI), Table 2 summarizes all items involved in each pre-treatment technique, including the material used for adhesives and related energy consumption.

Table 2. Data used in the LCI.

Pre-treatment technique	Item	Material	Quantity
Chemical activation (primer)	Adhesive PU-based (25mm length)	PU 9225	0.29 [g]
	Primer Teroson 150	Xylene and Ethylbenzene	1.08 [g]
Mechanical activation (abrasion)	Adhesive PU-based (25mm length)	PU 9225	0.29 [g]
	Energy Consumption [26]		0.00015 [Wh]
Plasma treatment	Adhesive PU-based (7.5mm length)	PU 9225	0.09 [g]
	Energy Consumption		0.146 [Wh]
Laser irradiation	Adhesive PU-based (7.5mm length)	PU 9225	0.09 [g]
	Energy Consumption		0.167 [Wh]

In relation to energy consumption, a power meter was used during laser and plasma pre-treatments to determine electrical energy consumption for each process, while data from the literature was used for mechanical activation [26]. For plasma and laser pre-treatments, energy consumption values refer to the source alone, while all peripheral devices (e.g., control and exhaust systems) were not included. In all cases, electrical energy was delivered from the Italian grid mix, corresponding to the location where tests were carried out. The Ecoinvent database was used for background data, while the ReCiPe midpoint (H) was adopted for the Life Cycle Impact Assessment (LCIA) method. All midpoint impact categories (Table 3) were included within the analysis as they affected the three damage categories: (i) human health (HH), (ii) ecosystem (ED) and (iii) resources (RA). In addition, endpoint impact categories were used with the aim of obtaining an overview of the overall environmental impact.

Table 3. Midpoint impact category [27]

Name	Acronym	Unit
Global warming potential	GWP	[Kg CO2 eq]
Ozone depletion potential	ODP	[kg CFC11 eq]
Terrestrial acidification potential	TAP	[kg SO2 eq]
Freshwater eutrophication potential	FEP	[kg P eq]
Human toxicity potential	HTP	[kg 1.4-DCB]
Particulate matter formation potential	PMFP	[kg PM2.5 eq]
Terrestrial ecotoxicity potential	TETP	[kg 1.4-DCB]
Freshwater ecotoxicity potential	FETP	[kg 1.4-DCB]
Marine ecotoxicity potential	METP	[kg 1.4-DCB]
Photochemical oxidant formation potential	OFP	[kg NOx eq]
Fossil fuel potential	FFP	[kg oil eq]
Water consumption potential	WCP	[m^3]

3. Results and discussion

An overview of mechanical test results has already been presented in Fig. 4. From this general overview, it is possible to observe that a traditional mechanical joint using a M6×20 bolt always obtains highest strength (approx. 3 kN), approximately twice the static load obtained with adhesive bonded joints. In the specific case of adhesive bonded joints, an upward trend can be observed for each pre-treatment as the overlap length increases from 5mm to 25mm. Plasma and laser activation exhibit better performance than both chemical and mechanical activation. Based on the results of mechanical tests, a target value of 1400 [N] ±10% was set to identify the overlap length for each pre-treated joint capable of guaranteeing the target failure load. Table 4 shows the mean value of failure load obtained for each pre-treated joint, with a 25mm overlap length for chemical and mechanical activation and a 7.5mm overlap length for plasma and laser activation.

Table 4. Failure load (tensile tests)

Tensile tests		Failure Load
Adhesive bonding	Chemical activation (primer) – 25mm overlap	1.27 [kN]
	Mechanical activation (abraded) – 25mm overlap	1.32 [kN]
	Plasma – 7.5mm overlap	1.55 [kN]
	Laser – 7.5mm overlap	1.47 [kN]

Focusing on the LCA results, Table 5 and Table 6 report the midpoint and endpoint results, respectively, for the four adhesive bonded joints. Amongst these, greatest impact is produced with primer pre-treatment, followed by mechanical abrasion, laser irradiation and plasma treatment. The results for laser and plasma pre-treatments are very similar in terms of environmental burden in all midpoint impact categories. This outcome derives from the fact that the two processes require the same overlap length to reach the failure load target value (1400 N), while both exhibit very similar values of electrical energy consumption during the pre-treatment processes. The largest difference between laser and plasma treatments can be observed in terrestrial acidification, TAP (approx. 2,4%), and global warning potential, GWP (approx. 1,5%).

The midpoint impact categories in which chemical activation (primer) presents the largest differences compared to other techniques include fossil resource scarcity, FFP (an order of magnitude greater than for the other joints), and global warning potential, GWP (almost double that of abraded joints and an order of magnitude greater than plasma and laser activation). For photochemical oxidant formation, OFP, water use, WCP, terrestrial acidification, TAP, and terrestrial ecotoxicity, TETP, values are 43%, 42%, 40% and 32% higher than for abraded joints, respectively. With a focus on chemical activation (primer), it can be observed that the impact caused by the PU-based adhesive (PU 9225) is much higher than the primer (Teroson 150) in all categories, with an average difference of 60%. Exceptions include global warning potential, GWP, and fossil fuel potential, FFP, where the impact of the adhesive is 5% less and 86% more than the primer, respectively.

Table 5. Midpoint impact category results

Midpoint	Primer	Abraded	Plasma	Laser
GWP	4.14×10^{-3}	2.02×10^{-3}	6.63×10^{-4}	6.73×10^{-4}
ODP	4.31×10^{-9}	4.22×10^{-9}	1.30×10^{-9}	1.31×10^{-9}
TAP	1.28×10^{-5}	7.70×10^{-6}	2.75×10^{-6}	2.82×10^{-6}
FEP	1.27×10^{-6}	9.72×10^{-7}	3.07×10^{-7}	3.09×10^{-7}
HTP	2.52×10^{-3}	2.01×10^{-3}	6.19×10^{-4}	6.23×10^{-4}
PMFP	6.01×10^{-6}	3.92×10^{-6}	1.27×10^{-6}	1.28×10^{-6}
TETP	7.98×10^{-3}	5.45×10^{-3}	1.67×10^{-3}	1.68×10^{-3}
FETP	1.13×10^{-4}	9.69×10^{-5}	2.99×10^{-5}	3.01×10^{-5}
METP	1.15×10^{-4}	9.18×10^{-5}	2.89×10^{-5}	2.91×10^{-5}
OFP	1.91×10^{-5}	1.09×10^{-5}	3.47×10^{-6}	3.51×10^{-6}
FFP	2.46×10^{-3}	8.61×10^{-4}	2.73×10^{-4}	2.75×10^{-4}
WCP	7.95×10^{-5}	4.58×10^{-5}	1.47×10^{-5}	1.49×10^{-5}

Table 6. Endpoint impact category results

Endpoint	Primer	Abraded	Plasma	Laser
HH	3.76×10^{-7}	2.24×10^{-7}	7.22×10^{-8}	7.30×10^{-8}
ED	2.53×10^{-8}	1.36×10^{-8}	4.49×10^{-9}	4.55×10^{-9}
RA	3.14×10^{-8}	9.22×10^{-9}	2.90×10^{-9}	2.92×10^{-9}

Focusing instead on mechanical activation (abrasion), it can be observed that the impact related to the pre-treatment itself is negligible compared to the adhesive, with an average difference of more than 99%. Upon analyzing the laser pre-treatment, it can again be observed that the impact of the adhesive is greater than that of the pre-treatment in all cases, with an average difference of 90%. The category with greatest difference (88%) is global warning potential, GWP. Analogous results are obtained for plasma.

With the aim of understand the contribution of each item involved in the various pre-treatments, Fig. 5 displays the contribution of each item for global warning potential, GWP, and human toxicity potential, HTP. The two graphs show approximately the same trend. In particular, for chemical activation, both primer and adhesive have almost the same impact on the two environmental indicators. Conversely, for mechanical activation, plasma treatment and laser irradiation, the most important contribution comes from the amount of adhesive used to produce the joint. Electrical energy used to perform each pre-treatment has a negligible contribution to the final result in both categories.

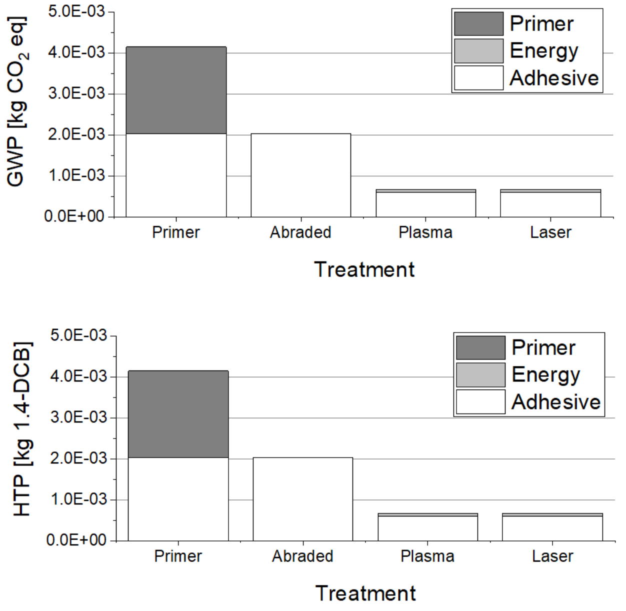

Fig. 5. Contribution of each item to GWP and HTP indicators

An analysis of the endpoint impact categories was carried out to understand the global impact of each of these methods, the results of which are presented in Fig. 6 (normalized values).

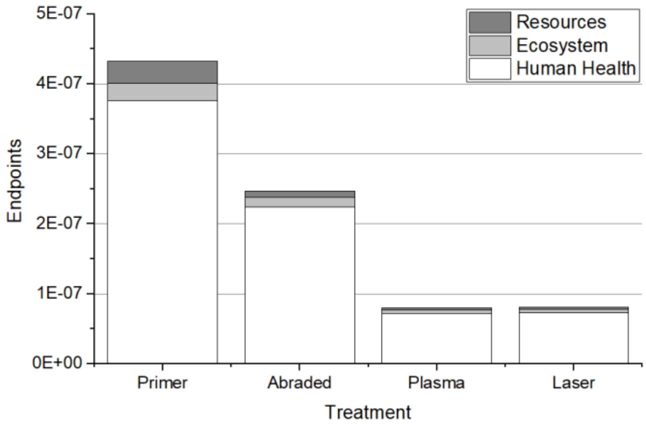

Fig. 6. Impact assessment (endpoints)

These results are in line with the outcomes observed for the midpoint impact categories. Amongst the adhesive bonded joints, plasma and laser activation are the techniques leading to lowest impact, some five times lower than chemical activation with the primer and three times lower than mechanical activation via abrasion, respectively. The graph also highlights an additional outcome, where the human health category represents the highest share for all joints, with contributions from resources and ecosystems almost negligible.

4. Conclusion

An LCA analysis has been performed to compare the impact of various pre-treatment methods including mechanical activation (abrasion), chemical activation (primer), plasma treatment and laser irradiation for preparation of PA66 joints bonded with TEROSON PU 9225 adhesive. Following an initial phase where the activation processes were set up and optimized, mechanical characterization of joints was performed to determine the overlap length necessary to achieve a static load of at least 1400 [N] ±10%. An overlap length of 25 mm was necessary for mechanical and chemical activation, while 7.5 mm was necessary for plasma and laser activation. Greatest impact was observed for chemical activation, followed by mechanical activation then laser and plasma treatments. Of particular importance was the fact that for chemical activation, both the primer and adhesive were found to have almost the same impact on the global warming potential, GWP, and human toxicity potential, HTP. For mechanical activation, plasma treatment and laser irradiation, impact from the primer was absent while electrical energy consumption was negligible, for which the most important contribution instead came from the amount of adhesive used to produce the joint, which was significantly lower for plasma and laser treatments than for mechanical abrasion due to differences in the performance of each pre-treatment. Little difference was observed between laser and plasma treatments, suggesting that the choice of solution can be made based on economic and technological considerations. Though the overall mechanical strength of a well-designed bolted joint is difficult to attain with adhesive bonded joints, advantages of the latter in terms of joining thin materials, weight reductions, ease of manufacturing, better stress distribution, good fatigue resistance and improved aesthetic appearance imply that the outcomes obtained within this study have important implications for further development of laser and plasma technologies as sustainable surface activation methods for polymers.

Acknowledgments

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