Advances in Science and Technology Research Journal
Volume 11, Iss. 1, March 2017, pages 75–81
DOI: 10.12913/22998624/66500

Research Article

THE EFFECT OF DEGREASING ON ADHESIVE JOINT STRENGTH

Anna Rudawska[1], Justyna Nalepa[2], Miroslav Müller[3]

[1] Lublin University of Technology, Faculty of Mechanical Engineering, ul. Nadbystrzycka 36, 20-618 Lublin, Poland, e-mail: a.rudawska@pollub.pl

[2] Lublin University of Technology, Faculty of Mechanical Engineering, ul. Nadbystrzycka 36, 20-618 Lublin, Poland, e-mail: justynalepa@o2.pl

[3] Czech University of Life Science Prague, Faculty of Engineering, 165 21, Prague, Czech Republic, e-mail: muller@tf.czu.cz

Received: 2016.12.15
Accepted: 2017.02.01
Published: 2017.03.01

ABSTRACT
The paper investigates the effects of degreasing, surface preparation methods in adhesive bonding and con adhesive joint strength. Five types of degreasing agents were used in the study: acetone, extraction naphtha, Ultramyt, Wiko and Loctite 7061. The degreasing operation was performed by three methods: rubbing, spraying and immersion. Strength tests were performed on single-lap adhesive joints of hot-dip galvanized metal sheets made with Loctite 9466 adhesive according to the above variants of surface preparation. The experimental results demonstrate that adhesive joint strength is significantly affected by the applied degreasing agent. Moreover, the method of application of the degreasing agent is crucial, too. The results of strength testing reveal that the most effective degreasing method for hot-dip galvanized metal sheet adhesive joints is spraying using extraction naphtha. Thereby, degreased samples have the highest immediate strength and shear strength. The use of extraction naphtha is also effective in combination with degreasing by rubbing; however, it is not effective when used in combination with immersion, as reflected in the lowest strength results.

Keywords: adhesive joints, strength, surafce treatment, degreasing.

INTRODUCTION

Adhesive bonding is one of the methods for joining galvanized metal sheets [1, 2]. Other methods like welding or pressure welding often cause defects such as damage to the zinc coating in the joining spot. For this reason, adhesive bonding is an important and competitive technique when compared to other methods [1].

Adhesive joint strength is affected by technological, operational, design- and material-related factors; considering these, it is possible to produce adhesive joints with the required strength and resistance to various operational factors [3–10]. The first and foremost stage of adhesive bonding involves surface preparation of adherents. The significance of this stage is stressed in numerous works [11–17], and there are many researchers who investigate surface preparation of elements for adhesive bonding [3, 4, 18–20]. This stage should be highly effective in order to prevent insufficient bonding that makes the joint weak. To this end, it is necessary that the surfaces of adherents be pretreated [6, 11]. Because of suitable surface preparation, the cohesion of an adhesive is lower than its adhesion, which meets a vital condition pertaining to adhesive joint strength and resistance to ambient conditions. This operation should ensure the strongest possible adhesive joints characterized by high strength [3, 5, 10]. The highest effectiveness is ensured by cleaning the adhered surface from all kinds of impurities such as lubricants, gas bubbles, fats, oils, water, dusts or micro-organisms, as they considerably hamper the effect of adhesive forces [6, 14]. The crucial operation of surface preparation in adhesive bonding includes degreasing, surface purification by acid or alkaline bath, rinsing, drying, special surface treatment, and primer application [11, 19–24].

The aim of this study was twofold: one, to determine the strength of adhesive joints made of galvanized metal sheets in compliance with the EN 1465 standard [25], the surfaces of which were degreased by five different agents prior to adhesive bonding; and, two, to make a comparative analysis of experimental results.

METHODS

Adhesive joint characteristics

The experiments involved producing single-lap adhesive joints of hot-dip galvanized metal sheets with a thickness ranging from 0.64mm ± 0.06 mm. Fig. 1 shows the schematic design of the tested adhesive joint.

The real dimensions of the produced adhesive joints measured with an electronic slide caliper were as follows: L = 99.60 ± 0.12 mm, l_z = 14.00 ± 4 mm, b = 20 ± 0.06 mm, g = 0.64 ± 0.06 mm, g_k = 0.20 ± 0.05 mm.

Adhesive properties

Galvanized metal sheets were bonded with a construction adhesive, Loctite 9466 A&B. This bi-component epoxy adhesive exhibits high strength and adhesion, low density, average viscosity, and it does not conduct any electric current (Table 1 and Table 2). Prior to adhesive bonding, the adhesive components (resin and curing agent) were mixed in 2:1 volumetric ratio until a uniform mixture. After that, the mixture was spread on one adhered surface. The working life of the produced adhesive mixture is about 60 minutes [26].

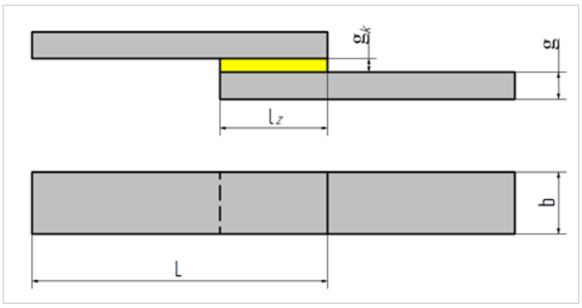


Fig. 1. Single-lap adhesive joint of hot-dip galvanized metal sheets

Table 1. Selected properties of Loctite 9466 before curing

Property	Resin	Curing agent
Chemical type	epoxy	amino
Specific gravity	1.00 N/m³	1.00 N/m³
Brookfield viscosity in 25°C spindle 7 at 20 rev/min	42.400 Pa·s	5.500 Pa·s

Table 2. Selected properties of Loctite 9466 after curing

Property	Value
Volumetric ratio (epoxy/curing agent)	2:1
Weight ratio (epoxy/curing agent)	100 : 50
Brookfield viscosity in 25°C	30 Pa·s
Lifetime of mixed adhesive	60 minutes
Working temperature range	- 55°C do 120°C

Epoxy adhesives are among the most effective and widely used adhesive polymers due to high intermolecular forces, i.e. adhesive forces ensuring accurate contact between the joined surfaces. In addition, they are resistant to chemical action [6, 23].

Experimental details

The surfaces of galvanized metal sheets were prepared using five types of degreasing agents: acetone, extraction naphtha, Loctite 7063, Ultramyt and Wiko, the properties of which are given in Table 3. The degreasing was ran by employing three methods: rubbing, spraying and immersion. The data is given in Table 4, and the schematic designs of these methods are illustrated in Table 5.

Table 3. Degreasing agents (prepared based on [27-31])

Degreasing agent	Composition
Acetone	acetone (100%)
Extraction naphtha	hydrogen-treated light petrol (petroleum); benzene < 0.05 %, toluene ≥ 3 % or n-hexane ≥ 3 %, < 5 %.
Ultramyt	toluene, polyoxyethylene ether of synthetic fatty alcohols
Loctite 7063	hydrogen-treated light petrol (petroleum), <0.1% benzane, ethanol, methylal, carbon dioxide
Wiko	hydrocarbons, acetone, isobutane , propane, carbon dioxide

Table 4. Degreasing methods used in the experiments

Degreasing method	Degreasing agent				
	Acetone	Extraction naphtha	Ultramyt	Loctite 7063	Wiko
Rubbing	+	+	+	-	-
Spraying	+	+	+	+	+
Immersion	+	+	+	-	-

Degreasing by rubbing consisted of rubbing the specimens three times with a paper towel soaked with a degreasing agent. After the third rub, the degreasing agent was left to evaporate (ca. 2 minutes). This method was not used when degreasing with Loctite 7063 and Wiko due to the fact that these agents come in containers with spray applicators. In contrast, other degreasing agents come in glass containers, which enable degreasing by different methods.

Degreasing by spraying was ran in several stages:

- application of the degreasing agent by spraying over the specimen surface,
- wiping off the wet surfaces with a clean paper towel to remove impurities,
- the above operations were repeated twice; following its final application, the degreasing agent was left to evaporate (ca. 2 min).

This degreasing method was performed using degreasing agents that come in special containers with sprinklers, i.e. acetone, extraction naphtha and Ultramyt. Loctite 7063 and Wiko came in original packaging provided with spray applicators.

Immersion consisted in immersing the specimen for 2 minutes in a glass container with a degreasing agent; after that, the specimen was taken out from the container and dried for ca. 3 minutes. This method was applied for acetone, extraction naphtha and Ultramyt, but it was not applied for other degreasing agents due to the above-mentioned limitations.

The degreasing process was performed by the above methods in a temperature ranging from 24°C to 26°C and humidity between 28% and 38%.

Following the degreasing, adhesive joints were made using the Loctite 9466 adhesive. The conditions of the adhesive bonding process for galvanized metal sheets were the same as in the degreasing of metal sheet surface.

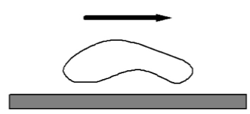
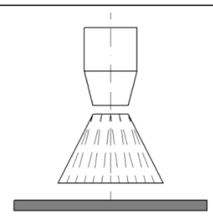
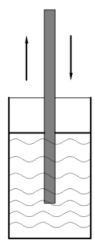
First, the adhesive was applied to the adherent surface; after that, the adherents were fixed and subjected to a load of 0.07 MPa. The adhesive was exposed to single-stage cold curing at a temperature ranging from 24°C to 26°C and humidity between 34% and 38%, and the curing time was set to 72 hours. After curing, the specimens of adhesive joints were subjected to strength testing on the Zwick/Roell Z150 testing machine. The shear strength testing was performed in compliance with the EN DIN 1465 standard [25].

RESULTS

Adhesive joint strength versus degreasing agent

The diagrams in Figures 2–4 show the results of strength tests of adhesive joints after surface degreasing with acetone, Ultramyt, extraction naphtha by rubbing, spraying and immersion.

Table 5. Schematic design of the applied degreasing methods

Method	Schematic design
Rubbing	
Spraying	
Immersion	

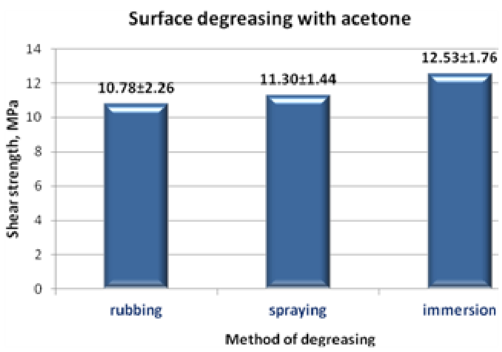


Fig. 2. Shear strength results of adhesive joints of galvanized metal sheet after surface degreasing with acetone by rubbing, spraying and immersion

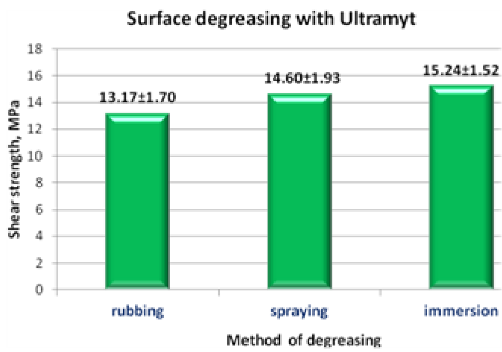


Fig. 4. Shear strength results of adhesive joints of galvanized metal sheet after surface degreasing with Ultramyt by rubbing, spraying and immersion

The diagram in Figure 2 reveals that the shear strength of adhesive joints where the adhered surface was degreased with acetone ranges from 10.78 MPa to 12.53 MPa. The lowest adhesive joint shear strength was observed when the adhered surface was degreased by rubbing, and it amounts to 86% of the highest value of shear strength observed for the specimens degreased by immersion in acetone.

Nonetheless, it seems that the similar values of shear strength produced with the tested degreasing methods point to a fact that the degreasing methods does not have a significant effect on adhesive joint strength; they rather serve for purification to ensure higher adhesion of the adhesive to the adhered surface.

The results reveal that degreasing with extraction naphtha affects the adhesive joint strength of galvanized metal sheets. The highest shear strength of the tested metal sheet adhesive joints was produced for the specimens degreased by spraying with extraction naphtha, while the lowest – by immersion in extraction naphtha. The strength of the tested adhesive joints of galvanized metal sheet specimens subjected to degreasing by immersion in extraction naphtha before adhesive bonding is 53% of the adhesive joint strength of galvanized metal sheets subjected to surface degreasing by spraying according to the employed experimental method.

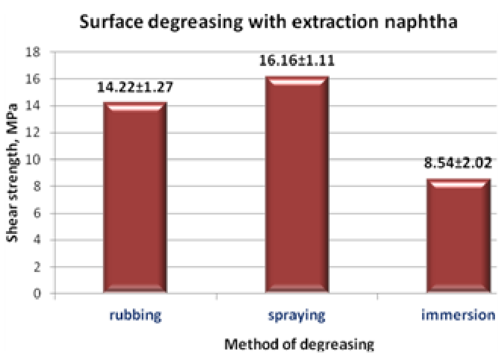


Fig. 3. Shear strength results of adhesive joints of galvanized metal sheet after surface degreasing with extraction naphtha by rubbing, spraying and immersion

The above diagram (Fig. 4) demonstrates that the shear strength of the specimens subjected to surface degreasing with Ultramyt ranges between 13.17 MPa and 15.24 MPa for the different degreasing methods. Moreover, it is observed that this degreasing agent is effective when applied by immersion. When comparing the results given in Figure 2 and in Figure 4 it can be observed that the lowest shear strength of the tested adhesive joints produced by rubbing with Ultramyt is equal to the highest adhesive joint strength obtained by degreasing by immersion in acetone. In addition, degreasing by immersion using both Ultramyt and acetone has a positive effect on the adhesive joint strength of galvanized metal sheets.

Adhesive joint strength versus degreasing method

The shear strength results following surface degreasing by rubbing, spraying and immersion using five degreasing agents are given in Figures 5-7. The results demonstrate that the applied degreasing method using five different degreasing agents has impact on the adhesive joint strength of galvanized metal sheets. It can be observed that there are differences in the values of shear strength depending the applied degreasing agent and degreasing method.

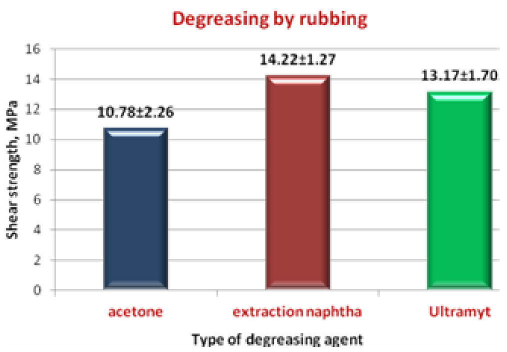


Fig. 5. Shear strength results of adhesive joints of galvanized metal sheet after surface degreasing by rubbing

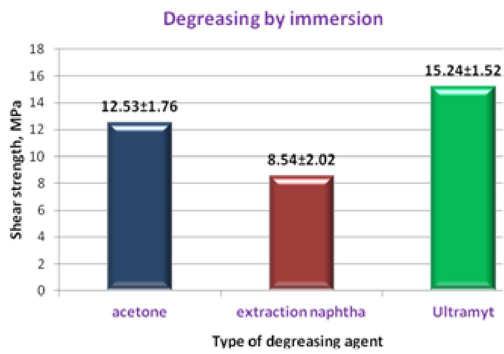


Fig. 7. Shear strength results of adhesive joints of galvanized metal sheet after surface degreasing by immersion

As for surface degreasing by rubbing (Fig. 5), the highest adhesive joint strength was observed for the specimens degreased with extraction naphtha (14.22 MPa). With degreasing by rubbing with acetone, the adhesive joint strength is 76% of the highest value of the adhesive joint strength of galvanized metal sheets subjected to degreasing with extraction naphtha.

As regards degreasing by spraying, it is also important to emphasize that the choice of a degreasing agent has an impact on adhesive joint strength. Following the application of the tested degreasing agent by spraying, the results demonstrate that the adhesive joint strength differs by 35%. In addition, it is observed that the degreasing of surfaces of galvanized metal sheets using acetone or acetone-containing degreasing agents is the least effective (Fig. 6), as the tested adhesive joints have the lowest shear strength following the application of these degreasing agents. These results also confirm that the degreasing agent must be selected depending on the material. Interestingly, surface degreasing by spraying is a widely used method, as many degreasing agents come in containers provided with applicators which facilitate this operation. From an economic point of view, it is also important that this method ensures the lowest consumption of adhesive agents.

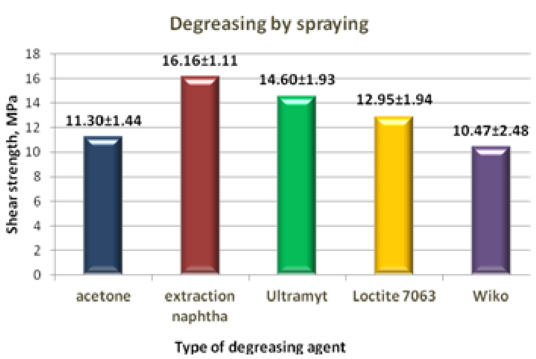


Fig. 6. Shear strength results of adhesive joints of galvanized metal sheet after surface degreasing by spraying

The highest differences in adhesive joint strength are observed for the specimens degreased by immersion (Fig. 7). The adhesive joint strength of galvanized metal sheets after surface degreasing by immersion in extraction naphtha is 56% of the adhesive joint strength of the specimens subjected to surface degreasing by immersion in Ultramyt. What is more, when using the immersion method one should take into consideration not only the consumption of the degreasing agent (higher consumption than with other methods) but also the degree of its impurity resulting from subsequently degreased specimens.

In terms of choice of a degreasing method, the results demonstrate that the largest differences in the adhesive joint strength of galvanized metal sheets occur in degreasing by immersion, whereas the smallest differences are observed for degreasing by rubbing.

It can be claimed that the adopted surface treatment method of materials for adhesive bonding should take into account not only the type of degreasing agent but also the degreasing method, as the results reveal that these two factors have a significant effect on the strength of the tested adhesive joints. Importantly, it is necessary to examine the type of adherent because the use of both an unsuitable degreasing agent and a degreasing method can lead to lower strength. Moreover, it is necessary to take account of the available workshop conditions (the possibility of application the immersion method, production type, available devices), operational conditions (the size and accessibility of degreased surfaces), economic conditions (the amount of degreasing agent used directly for surface degreasing), and many others.

CONCLUSIONS

The experimental results demonstrate that adhesive joint strength greatly depends on the applied degreasing agent. In addition, adhesive joint strength can be affected by the method of application of the degreasing agent, too.

The strength results reveal that the most effective degreasing method for the adhesive bonding of galvanized metal sheets is degreasing with extraction naphtha by spraying. Thereby degreased specimens of adhesive joints have the highest shear strength. Extraction naphtha is also effective when applied by rubbing; however, it is the least effective when applied by immersion (the lowest shear strength). The results of degreasing by spraying demonstrate that Wiko and acetone are the least effective degreasing agents, hence, it can be claimed that the degreasing agents containing acetone are not recommended for the degreasing of galvanized metal sheets. The highest shear strength is exhibited by the adhesive joints degreased with toluene and extraction naphtha. The highest and most uniform strength results were observed for the specimens degreased by immersion in acetone and Ultramyt, even though the results tend to differ to a significant degree. In such a case, the choice of a degreasing agent often depends on two aspects: the economic aspect (a less expensive degreasing agent is applied) and the technological aspect (e.g. a way of application, as the use of agents which come with applicators enables a faster, easier and more economic application of the degreasing agent to the adhered surface).

Notwithstanding the above, a crucial aspect of joining elements for aircraft, automotive or other machinery designs is to ensure the highest possible tightness and strength of the adhesive joint, which means that a given material should be degreased using the best degreasing agent available.

REFERENCES

1. Rudawska, A., Kuczmaszewski, J.: Klejenie blach ocynkowanych. Monografia. Wydawnictwa Uczelnianie PL, Lublin 2005.
2. Rudawska, A., Kuczmaszewski, J.: Surface free energy of zinc coating after finishing treatment. In. Materials Science- Poland, Vol. 24/2006, Issue 4, 975-981.
3. da Silva, L.F.M., Carbas, R.J.C., Critchlow, G.W., Figueiredo, M.A.V., Brown, K.: Effect of material, geometry, surface treatment and environment on the shear strength of single lap joints. International Journal of Adhesion and Adhesives 29, 2009, 621-632.
4. Brack, N., Rider, A.N.: The influence of mechanical and chemical treatments on the environmental resistance of epoxy adhesive bonds to titanium. International Journal of Adhesion and Adhesives 44, 2014, 20-27.
5. Prolongo, S.G., Ureña, A.: Effect of surface pretreatment on the adhesive strength of epoxy-aluminium joints. International Journal of Adhesion and Adhesives 29, 2009, 23-31.
6. Adams, R.D., Comyn, J., Wake, W.C.: Structural Adhesive Joints in Engineering Book, 2nd edition. UK: Springer; 1997.
7. Valášek, P., Müller, M., Hloch, S.: Recycling of corundum particles – two-body abrasive wear of polymeric composites based on waste. In. Tehnicki Vjesnik-Technical Gazette, vol. 22, 3/2015, 567-572.
8. Müller, M.: Polymer composites based on AL_2O_3 reinforcing particles. Engineering for Rural Development 26.05.2011, Jelgava. Jelgava: Latvia University of Agriculture, 2011, 423-427.
9. Müller, M., Cidlina, J., Dědičová, K., Krofová. K.: Mechanical properties of polymer composite based on aluminium microparticles. In. Manufacturing Technology, vol. 15, 4/2015, 624–628.
10. Müller, M.: Research of liquid contaminants influence on adhesive bond strength applied in agricultural machine construction. In. Agronomy Research, vol. 11, 1/2013, 147–154.
11. Rudawska, A.: Surface Free Energy and 7075 Aluminium Bonded Joint Strength Following Degreasing Only and Without Any Prior Treatment. Journal Adhesion Science and Technology, 26, 2012, 1233-1247.
12. Rudawska, A.: Selected aspects of the effect of mechanical treatment on surface roughness and adhesive joint strength of steel sheets. International Journal of Adhesion and Adhesives, 50, 2014, 235-243.
13. Rudawska, A., Reszka, M., Warda, T., Miturska, I.,

Szabelski, J., Stančekova, D., Skoczylas, A.: Milling as a method of surface pre-treatment of steel for adhesive bonding, Journal of Adhesion Science and Technology, 2016, http://dx.doi.org/10.1080/01694243.2016.1191585.

14. Ebnesajjad, S., Ebnesajjad, C.: Surface Treatment of Materials for Adhesive Bonding, 2 nd Edition. Norwich: William Andrew; 2013.
15. Podhajny, R.M.: Comparing surface treatments. Converting, 8, 1990, 46-52.
16. Bouquet, F., Cuntz, J.M., Coddet, C.: Influence of surface treatment on the durability of stainless steel sheets bonded with epoxy. Journal of Adhesion Science and Technology, 6, 1992, 233-242.
17. Hass, P., Kläusler, O., Schlegel, S., et al.: Effects of mechanical and chemical surface preparation on adhesively bonded wooden joints. International Journal of Adhesion and Adhesives, 51, 2014, 95-102.
18. Rotella, G., Alfano, M., Schiefer, T., Jansen, I.: Evaluation of mechanical and laser surface pretreatments on the strength of adhesive bonded steel joints for automotive industry. Journal of Adhesion Science and Technology, 30, 2016, 747-758.
19. Czarnota, M., Rudawska, A.: Wpływ operacji odtłuszczania na właściwości adhezyjne warstwy wierzchniej oraz wytrzymałość połączeń klejowych blach miedzianych. Postępy nauki i techniki, 8, 2011, 137-147.
20. Rudawska, A.: Wpływ sposobu przygotowania powierzchni na wytrzymałość połączeń klejowych blach ze stali odpornej na korozję. Technologia i Automatyzacja Montażu, 3, 2010, 36-39.
21. Borsellino, C., Bell, G., Di, Ruisi, V.F.: Adhesive joining of aluminium AA6082: The effects of resin and surface treatment. International Journal of Adhesion and Adhesives, 9, 2009, 36-44.
22. Guedes Pinto, A.M., Magalhães, A.G., Gomes da Silva, F., Monteiro Baptista, A.P.: Shear strength of adhesively bonded polyolefins with minimal surface preparation. International Journal of Adhesion and Adhesives, 28, 2008, 452-456.
23. Ebnesajjad, S.: Adhesives Technology Handbook. 2nd edition: William Andrew Publishing; 2008.
24. Uehara, K., Mitsuru, S.: Bonding strength of adhesives and surfaces roughness of joined parts. Journal of Materials and Processing Technology, 127 (2) 2002, 178-181.
25. DIN EN 1465. Adhesives. Determination of tensile lap-shear strength of bonded joints.
26. http://www.loctite-kleje.pl (accessed on 26.01.2016).
27. http://pl.gluetec-industrieklebstoffe.de/ (accessed on 01.03.2016).
28. http://www.chem-rozlew.com (accessed on 01.03.2016).
29. http://www.loctite-dip.pl (accessed on 01.03.2016).
30. http://www.polmorplus.pl (accessed on 01.03.2016).
31. http://ekos.gda.pl (accessed on 01.03.2016).