Research article

COMPARATIVE ANALYSIS OF THE ENDURANCE OF UV-CURED ADHESIVE JOINTS

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ABSTRACT

The aim of the study was to compare the strength of adhesive joints bonded with two different UV-cured adhesives in different exposure times. The adhesives used in the research differed in viscosity. The first adhesive Multibond 5550 was characterised by low viscosity, the second adhesive Multibond 5544 was characterised by high viscosity. The adhesive joints were made from a AISI 316L stainless steel sample and a sample made from polymethyl methacrylate. The adhesive joints were cured at three time intervals: 5 minutes, 10 minutes and 20 minutes. The strength of the adhesive bonds was compared using the MANOVA test. Statistical analysis showed that there was a statistical difference between the adhesives cured for 10 minutes. It was also found that joints where a high viscosity adhesive was used showed similar strength results for 10 and 20 minutes of adhesive exposure to UV rays. Statistical analysis was performed using RStudio software and the R programming language.

KEYWORDS: adhesive joints, UV-curing, adhesion, statistical analysis

1. Introduction

The assembly technology incorporates a group of methods that allow components to be joined together. One such method is adhesive joints, which are widely used in the automotive and aerospace industry. The reason for using adhesive joints in these industries is the possibility to achieve a joint without excessive weight, which is the case with screws or welding joints. Another reason is the ability to join parts with different material structure. One of the adhesive technology methods is UV-cured joints.

Ultraviolet irradiation, called in short UV irradiation, is an electromagnetic wave that is part of the light invisible to the human eye. It occurs in the form of sunlight. UV irradiation is divided into 4 types according to the length of the light used [1]:

- VUV: 100-200 nm,
- UV-C: 200-280 nm,
- UV-B: 280-315 nm,
- UV-A: 315-400 nm.

The process of curing the bond by UV irradiation takes place through crosslinking of the liquid oligomers contained in the adhesive and the formation of a solid polymer under the UV light. The advantages of this method include fast curing of the bond, low organic compound emissions and thermal and dimensional stability [2]. In UV-cured adhesive joints, it is important that one of the components being joined allows UV light to penetrate deep into the material up to the adhesive bond. The type of UV irradiation mainly used in adhesive technology to join the components is UV-A irradiation. An example of the use of this adhesive bonding method is the bonding of glass [3].

In this paper, shear strength tests were carried out on adhesive joints with a UV-A cured bond. Research contains two adhesives with different adhesive viscosity coefficient. The adhesive joints were formed by bonding a steel sample to a plastic sample. Three different exposure times were adopted: 5 minutes, 10 minutes and 20 minutes.

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Using statistical analysis, adhesive joint preparation variants were compared in terms of the adhesive used and the exposure time of the adhesive joint. This was to show statistical similarities between the variants of the created adhesive joints. The R programming language and the RStudio programme were used to perform the statistical analysis.

2. Methodology

2.1. Adhesive selection

Two UV-curing adhesives based on methacrylate resin were used in the study. The factor that differed the selected adhesives was the viscosity coefficient. The first adhesive was Multibond 5544 with a high viscosity coefficient. The second adhesive used in the study was Multibond 5500, an adhesive with a low viscosity coefficient. The adhesive properties of both adhesives are shown in Table 1.

	Multibond 5544	Multibond 5500
Form	Liquid/gel	Liquid
Colour	Transparent	Transparent
Viscosity	7000 mPa*s	80 mPa*s
Density	1.1 g/ml	1.1 g/ml
Adhesive joint thickness	<1.5 mm	<0.15 mm
Thermal resistance	-60 + 150 C	-60 + 125 C

Table 1. Properties of Multibond 5544 and	d Multibond 5500 [4,5]
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The initial curing time for the adhesive bonds using the above mentioned adhesives was dependent on the material through which the UV irradiation passed. In the case of glass, this time is approximately 30 seconds. The curing time for plastics is much longer, due to their structure. The full curing of the adhesive joint occurs after 24 hours have elapsed.

2.2. Preparation of joints

In the study overlapping adhesive joints were prepared. They consisted of 2 dissimilar materials. The first type of the specimen was made of AISI 316L stainless steel. This steel has anti-corrosion and anti-allergenic properties [6].

Polymethylmethacrylate was used as the material for the second type of samples. The polymethylmethacrylate used for the test was transparent, which allowed UV-A rays to pass through the material to cure the adhesive. The specimens of both materials had a locating hole, which allowed the specimens to be axially fixed in relation to each other in the adhesive holder. Fig. 1 shows the dimensions of the samples and the adhesive bond.



Figure 1. Dimension of adhesive joint, A - AISI 316L stainless steel sample, B - polymethylmethacrylate sample

The first step in constructing the adhesive bond was preparation of the adhesive surface of the stainless steel specimens. In this operation, sandpaper with grit P240 was used. In order to obtain an undirected surface texture, the adhesive surface of the steel specimens was manually rubbed with sandpaper by circular motions for 1 minute. The surface preparation allowed to increase the mechanical adhesion [7].

In the next step, the samples were washed with Loctite 7063 surface cleaner. The surface cleaning operation was carried out by applying the cleaner to the surface and then washing with a dust-free swab in a single motion from the edge of the sample. This procedure was repeated twice. After mechanical cleaning of the adhesive surface, the cleaner was applied to the surface and left until it evaporated. The cleaning of the surface of the samples was carried out for both materials used in the test.

The adhesive joints were made in 6 variants, which differed in exposure time and the adhesive used. Each variant consisted of 6 adhesive joints. In total 36 adhesive joints were prepared. The exposure times for curing the adhesive bond were 5, 10 and 20 minutes. The test used a UV chamber equipped with an adhesive holder and a Bohl UVA-STAR lamp, whose light length corresponds to UVA irradiation [8].

The adhesives were applied to a steel sample which was placed in the adhesive holder. Then a polymethylmethacrylate sample was joined with the steel sample. The pressure was provided by transparent polycarbonate rods with a diameter of 16 mm loaded with 1 kg blocks. The exposure time of the samples depended on the selected variant. The temperature and air humidity during the preparation of the specimens and the making of the adhesive joints were 23 °C ± 1 °C and 20 % ± 1 % respectively. After curing the adhesive joints, the completed joints were seasoned for a period of 1 week under conditions that prevented exposure of the samples to sunlight.

A Zwick/Roell Z150 testing machine was used to carry out the strength tests. The adhesive joints were subjected to shear strength tests using PN EN 1465 [9], with a test speed of 2 mm/min. The following conditions prevailed during the strength test and the seasoning of the adhesive joints: temperature 23 °C \pm 1 °C, humidity 20 % \pm 1 %.

3. Results

On the basis of the results of the strengths of the different adhesive joint variants, descriptive statistics were prepared as shown in Table 2. This table includes the mean shear strength, median and standard deviation and variance for each adhesive joint variant.

Adhesive	Period of curing	Number of joints	Mean [MPa]	Median [MPa]	Standard deviation [MPa]	Variance [MPa]
Multiband	5 minutes	6	0.541	0.562	0.157	0.024
5500	10 minutes	6	1.480	1.480	0.159	0.025
	20 minutes	6	1.432	1.480	0.165	0.027
	5 minutes	6	0.562	0.584	0.063	0.004
5544	10 minutes	6	1.102	1.060	0.119	0.014
	20 minutes	6	1.542	1.510	0.111	0.012

Table 2. Adhesive joints strength results

Adhesive joints bonded with Multibond 5500 showed that the highest mean value was obtained for joints whose adhesive bond was exposed for a period of 10 minutes. However, the median for this variant is equal to the median of the adhesive joints with the adhesive bond cured for 20 minutes. The lowest mean shear strength value was obtained for the joint with the bond cured for 5 minutes. For this type of the joint, the median is close to the mean value.

For adhesive joints bonded with Multibond 5544, an increase in average strength is noticeable depending on the exposure time. The lowest average value was obtained for the variant with the joint cured for 5 minutes, while the highest value for the joint cured for 20 minutes.

Analysing the strength of all the variants, the highest average strength was obtained by the adhesive joints bonded with Multibond 5544 cured for 20 minutes.

4. Statistical analysis

The aim of the statistical analysis was to demonstrate the statistical significance of the strength of the adhesive joints between the length of the exposure time and the adhesive used for the created adhesive joints. To perform the analysis, a MANOVA statistical test was used, allowing multiple cases to be compared against more than 2 variable factors. In the statistical test, the variable factor was the length of the exposure time of the adhesive joint. The MANOVA test requires checking for the presence of a normal distribution of the cases analysed and homogeneous variance between the cases [10].

The Shapiro-Wilk test was used to demonstrate the normal distribution of the adhesive bond strengths in the tested variants. Features of this test include the ability to test the normal distribution on small groups and the high power of the test [11]. The homogeneity of variance between the shear strength values of the tested adhesive joint variants was checked using the Bartlett test. The level of statistical significance for all statistical tests was $\alpha = 0.05$.

	H ₀ : No basis for rejecting the hypothesis of a normal distribution				
assumptions	s H ₁ : There is a basis for rejecting the hypothesis				
	normal distribution				
	Statistical significance $\alpha = 0.05$				
adhesive	period of curing	p-value			
Multibond 5500	5 minutes	0.360			
	10 minutes	0.614			
	20 minutes	0.109			
Multibond 5544	5 minutes	0.447			
	10 minutes	0.281			
	20 minutes	0.429			

Table	3.	Normal	distribution	test	results
	•••	1.0111101	alstindation		1000100

Statistical calculations of the normal distribution performed with the Shapiro-Wilk test are shown in Table 3. The p-value for each variant showed that p-value > α , which leads to a conclusion that there are no grounds for rejecting the hypothesis that a normal distribution exists.

Table 4. Results of the test for homogeneity of variance

	H_0 : No basis for rejecting the hypothesis of a normal distribution				
assumptions	H ₁ : There is a basis for rejecting the hypothesis of a norm				
	distribution				
	Statistical significance α = 0.05				
bartlett		df	p-value		
3.8055		5	0.5777		

On the basis of the homogeneity of variance test performed (Table 4), it can be concluded that there are no grounds for rejecting the null hypothesis claiming that there is homogeneity of variance. The test probability p-value is greater than the level of statistical significance α .

The performed statistical tests of normal distribution and homogeneity of variance allow for the test of multivariate analysis of variance.

The values of the results of the performed multivariate analysis of variance are shown in Fig. 2. The statistical analysis performed compares the strength values of the adhesive joints for the three factors:

- Between the adhesive used,
- Between curing time,
- Between adhesive used and curing time.

	Df	Sum Sq	Mean Sq	F value	Pr(>F)	
factor(adhesive)	1	0.051	0.0511	2.849	0.104410	
factor(time)	2	4.867	2.4335	135.687	8.28e-14	***
<pre>factor(adhesive):factor(time)</pre>	2	0.337	0.1687	9.407	0.000963	***
Residuals	24	0.430	0.0179			
Signif. codes: 0 '***' 0.001	* **	*' 0.01	'*' 0.0	5 '.' 0. 1	1''1	

Figure 2. MANOVA test results

By analysing the MANOVA results, we can conclude that there is no statistical significance only for the comparison of groups against the adhesive used. The P-value for the other factors is less than the assumed level of statistical significance. For further verification, we used Tukey's POST-HOC test (Fig. 3), which allows pairwise comparisons of individual factors.

multibond 5550:5-multibond 5544:5 -0.0204 -0.28228248 0.2414825 0.9998	734 184
	184
multibond 5544:10-multibond 5544:5 0.5402 0.27831752 0.8020825 0.0000	
multibond 5550:10-multibond 5544:5 0.9182 0.65631752 1.1800825 0.0000	000
multibond 5544:20-multibond 5544:5 0.9802 0.71831752 1.2420825 0.0000	000
multibond 5550:20-multibond 5544:5 0.8702 0.60831752 1.1320825 0.0000	000
multibond 5544:10-multibond 5550:5 0.5606 0.29871752 0.8224825 0.0000	104
multibond 5550:10-multibond 5550:5 0.9386 0.67671752 1.2004825 0.0000	000
multibond 5544:20-multibond 5550:5 1.0006 0.73871752 1.2624825 0.0000	000
multibond 5550:20-multibond 5550:5 0.8906 0.62871752 1.1524825 0.0000	000
multibond 5550:10-multibond 5544:10 0.3780 0.11611752 0.6398825 0.0020	072
multibond 5544:20-multibond 5544:10 0.4400 0.17811752 0.7018825 0.0003	291
multibond 5550:20-multibond 5544:10 0.3300 0.06811752 0.5918825 0.0079	475
multibond 5544:20-multibond 5550:10 0.0620 -0.19988248 0.3238825 0.9758	788
multibond 5550:20-multibond 5550:10 -0.0480 -0.30988248 0.2138825 0.9923	132
multibond 5550:20-multibond 5544:20 -0.1100 -0.37188248 0.1518825 0.7828	209

Figure 3. Tukey's POST-HOC test results

The results of the POST-HOC test showed that there was a lack of statistical significance between the strength results of the following pairs of variants:

- Multibond 5550/5 min Multibond 5544/5 min,
- Multibond 5544/20 min Multibond 5550/10 min,
- Multibond 5550/20 min Multibond 5550/10 min,
- Multibond 5550/20 min Multibond 5544/20 min.

For the remaining variants, statistical differences between pairs of variants can be identified based on the POST-HOC test.

5. Conclusion

The test carried out on the strength of UV-cured adhesive joints leads to the following conclusions:

- The highest average strength of the adhesive joints was obtained by a joint created with Multibond 5544 cured for 20 minutes, while the lowest average value was obtained by a joint made with 5500 adhesive and cured for 5 minutes,
- There was statistical significance between both adhesives when cured for 10 minutes,
- The curing of the 5500 adhesive for more than 10 minutes does not statistically increase the strength of the adhesive joint,

• Adhesive joints bonded with both Multibond 5500 and Multibond 5544 whose adhesive bond was cured for 20 minutes are statistically equal.

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