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EVALUATION OF THE MECHANICAL STRENGTH OF AN ELASTOMERIC ADHESIVE DEPENDING ON TEMPERATURE AND CURE TIME

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Abstract. *The bonding is presented as an option for the union of materials that has advantages such as non-concentration of stress in the joints, increase in fatigue and galvanic corrosion resistance and is usually a lighter union option compared to traditional methods such as welding and screwing. The main objective of this work was to evaluate the mechanical properties of the 3M EC-847 adhesive according to temperature variation and cure time. For this was done the comparison of the results with data of the manufacturer regarding the methodology of bonding. In addition, a statistical analysis of the results was performed through variance analysis and Tukey test to obtain the most positive curing processes in the shear strength. The complete factoring planning was used for the execution of the experiments. The experiments were divided into three groups; each group being defined by the cure temperature. Each experiment, i.e. for each temperature and cure time, five replicates were performed. The median time used is recommended by the manufacturer and the other two had cure times 20% above and below the reference value provided by the manufacturer. The best responses were given for cures done at 150 °C in times of 8, 10 and 12 minutes and at 100 °C in the 95 minutes. In addition, it has been proven that the curing process at room temperature does not present good results.*

Keywords: *adhesive, cure temperature, cure time, elastomer, shear strength*

1. INTRODUCTION

Joining of joints is a necessity in many industrial sectors since the fabrication and transport of equipment and structures would be difficult if the elements were formed by a single block. In view of the need to join materials, a number of solutions have been created throughout the history for the connection, permanent or otherwise, between two or more solid elements.

Currently, there are several options of joints such as welding, riveting, screwing and gluing. The bonding has the advantages of having greater resistance to fatigue, presents a better load distribution which results in a lower concentration of tension in the parts. The best performance of adhesives in relation to welded and riveted joints in relation to problems related to corrosion can also be mentioned. And is still, in general, the most suitable technology for the connection between composite materials. On composites is observed by Xavier et al. (2016) that then use of metallic reinforcements in adhesives (transforming the adhesive into a composite) can increase equipment life and minimize maintenance costs. Lopes (2014), comments on the advantage of the adhesive bonds simplify the connection between irregularly shaped surfaces resulting in lighter joints. The advantages of adhesives compared to other modes of bonding between materials initially led to certain industries, such as aeronautics and naval, to increase the use of adhesives and, consequently, to encourage their development. It is also possible to observe the presence of adhesives in the packaging, automotive, civil construction and electronics industries (Pizzi & Mittal, 2003).

In particular, during the 20th century, great advances were made in the creation of new adhesives and in the characterization of their properties, such as shear strength, tear strength and temperature. The knowledge of the characteristics of an adhesive and its behavior under conditions of use are fundamental for any project as demonstrated by Faneco (2014). In view of this, assays which simulate the conditions of service of the part are essential. With respect to the mechanical stresses on bonded joints, one of the most used tests, due to its speed and relative simplicity, is the single lap joints shear test. This test consists of tensile stress applied to the cast joint.

Monteiro (1995) observed that the healing process can also be ruled, in large part, by the temperature. The cure can be accelerated, and even the resistance of an adhesive can be modified by applying controlled temperature under specified minimum times.

The main objective is to evaluate the shear strength properties of an elastomer adhesive according to temperature variation and cure time. The specific objectives are: compare the methodology of the experiments with the results with the data of the manufacturer, evaluate the adhesive properties in relation to the shear strength, according to the variation of time and temperature of cure, perform statistical analysis of the results through Analysis of Variance (ANOVA) and Test of Tukey to obtain the curing processes that most positively influence the shear strength.

2. METODOLOGY

The adhesive used is a copolymer composed of a nitrile rubber base formed by the mixture of acrylonitrile-butadiene and to a lesser extent phenolic resin. It presents predominantly characteristics of an elastomer. The trade name of the adhesive is EC-847 produced by 3M Co. The manufacturer gives suggestions by the of times and temperatures to be used for curing the adhesive. These data were used for comparison and they are shown in Tab. 1.

Table 1. Curing process indicated by the manufacturer.

Thread temperature (°C)	Minimum time to cure (min)
93	120
115	40
138	12
160	8
182	5
204	2

The process of bonding the steel plates, manufactured according to the standard ASTM D1002, consists in the application of the adhesive to the properly cleaned plates. And to achieve the correct thickness of adhesive the plate shown in Fig. 1(a) is used.



(a)



(b)

Figure 1. Experimental apparatus (a) Placement of the plates on the glue base (Barbosa, 2017) (b) Test plates positioned on the traction machine.

After the correct thickness dimensioning the specimens can be taken to the kiln for the curing process. With the adhesive properly cured the planks can be taken to be tested in the traction machines. There was a need for a specific apparatus for fitting the specimens to the machine. The test piece and the fixture are shown in Fig. 1(b). The machine used to carry out the tests is the manufacturer MTS Systems Corporation, model Bionix Servohydraulic Test System. The loading used was as recommended by ASTM D1002 for metal substrate adhesive testing, equivalent to the test speed of approximately 1.3 mm / min. The area that is effectively bonded through the adhesive corresponds to $3.2258 \times 10^{-4} \text{ m}^2$.

For the planning of the experiments, the complete factorial planning was adopted. This scheduling board has been chosen because it is possible through it to evaluate two or more independent variables. In addition, Calado and Montgomery (2003) point out that factorial planning is extremely useful for measuring the effects or influences of one or more variables on the response of a process. In this paper, a response based on two factors is evaluated. The response is the mechanical shear strength and the independent factors are time and cure temperature. For the measurements performed in this study, 95% confidence intervals were considered.

Table 2 presents the experiment planning. The central curing time values are the values indicated by the manufacturer. On the other hand, the other values were assumed to be 20% below and 20% above the reference time. This methodology was used to evaluate the influence of the different temperatures on the adhesive properties and for the analysis of how different curing times affect the strength of the joint. This variation in curing times allows the comparison of the information provided by the manufacturer and the indication of which curing time brings the best mechanical resistance to the studied temperature.

Table 2. Experimental planning based in complete factorial planning.

Cure temperature (°C)	Cure time (min)
100	114
	95
	76
150	12
	10
	8
200	3
	2.5
	2

For the analysis of the obtained data were used the analysis of variance and the Tukey's test. The analysis of variance (ANOVA) aims to compare the means to determine if there is a difference between the distribution of one group and another. It should be noted that for the use of ANOVA the following assumptions are used: (i) normal data distribution (ii) randomness of errors (iii) statistical independence. On the other hand, the Tukey's test is a comparative test that evaluates groups two to two and shows if there is a significant difference between these groups. Through this test it is possible to determine between which groups there are differences. The Tukey's test complements the analysis of variance because the ANOVA only indicates if exist or not a significant difference, but does not indicate which groups there are differences.

3. RESULTS

Table 3 presents all the results from the factorial planning done. Taking into account the curing temperature of 100°C it can be seen that there is an increase in mechanical strength as the curing time is increased from 76 to 95 minutes. However, when the cure time is increased again by a further 20% to 114 minutes, there is a decrease in strength indicating that in this case, the increase in curing time beyond the value indicated by the fabric is not beneficial. The overexposure of the adhesive at a temperature of 100°C may have led to degradation of the adhesive, decreasing its adhesion capacity.

Table 3. Results of shear resistance taking into account time and temperature of cure.

Parameters		Variable (Average ± Standart Deviation)
Cure temperature (°C)	Cure time (min)	Shear resistance (MPa)
100	114	0.59 ± 0.142
	95	0.83 ± 0.307
	76	0.52 ± 0.145
150	12	0.87 ± 0.204
	10	0.99 ± 0.329
	8	0.74 ± 0.173
200	3	0.34 ± 0.079
	2.5	0.23 ± 0.049
	2	0.25 ± 0.035

For the treatments made with curing times of 8 and 12 minutes at 150 °C the mechanical resistance did not present significant changes. But for the time of 10 minutes an increase in resistance of approximately 20% was observed in relation to the other two times studied. However, it can be stated that the shear strength of this treatment was similar to the treatment done at 100 °C for 95 minutes, but the required cure time was considerably lower. Considering the best results obtained in the two experiments there is a decrease of 85 minutes in the cure time. Since for obtaining a resistance of 0.83 MPa it took 95 minutes at 100 °C while for obtaining 0.99 MPa of resistance it took only 10 minutes at 150 °C. Thus, a similar resistance is obtained in an 88% less time.

For the temperature of 200 °C, it is possible to observe that there is a slight decrease of average mechanical resistance in the heat treatment of 2.5 minutes in relation to one of 2 minutes, not constituting a substantial difference. Analyzing this group of experiments as a whole, as the curing time increased, there was a tendency to increase the resistance, with the maximum value found in the time of 3 minutes. In this case, the manufacturer's indication was not the best option for increasing the adhesive properties. It should be noted that even the highest tension value obtained with this treatment is much lower than previously found. Therefore, this curing temperature should still be analyzed for the optimal exposure time which would result in higher tensions.

3.1. Type of rupture

It should be noted that about the type of rupture observed in the samples throughout the tensile tests, all presented cohesive type rupture. That is, the adhesive was broken, but the adhesive-substrate bond remained. This type of breakage indicates that the adhesive has tightly adhered to the substrate so that the breakage occurs when the internal mechanical strength of the adhesive itself no longer supports the stresses and fails. However, adhesion of adhesive / substrate adhesion is maintained.

3.2. Analysis of variance and Tukey test

In spite of this preliminary analysis, it is necessary to use statistical analysis for the more consistent evaluation of the interference of the variation of the curing parameters on the mechanical properties of the adhesive. Table 4 presents the ANOVA.

Table 4. Analysis of Variance (ANOVA).

	Degrees of freedom	Sum of squares	Average squares	F _{calculated}	F _{critical}
Cure time	8	1.24206	0.15526	4.31826	3.17
Cure temperature	2	0.25303	0.12652	3.51887	4.10
Model	10				

The F_{calculated} is calculated from the ratio of the sum of the squares to the mean of the squares. By observing the ratios F for the curing temperature, it is noted that the F_{calculated} is less than the F_{critical} value for the curing temperature. This indicates that there is no significant difference in the response variable for the different cure temperatures. Then, the ANOVA indicates that the temperature is not a statistically significant factor to obtain the best adhesive properties. On the other hand, at least one significant difference exists between the means of the resistances taking into account the variation of the times because F_{calculated} is bigger than F_{critical} considering cure time. However, it does not indicate between which times of cure there is a significant difference. The analysis of variance only indicates that curing time is the predominant factor influencing the response variable, ie, the mechanical strength of the adhesive.

The Tukey test is a complement to the analysis of variance since it compares the groups of experiments two to two and evaluates if there are significant differences between the groups. Thus, Tukey's test compares the influence of different cure times on the response by making all possible combinations between two different cure times. The result of this test shows between which healing processes there is a statistical difference and among which there is no difference. It is possible to observe that the best results of shear stress were given for the curing conditions presented in the Tab. 5.

Table 5. Better results obtained taking into account the healing process.

150°C	8 min	0.84 MPa
150°C	10 min	0.99 MPa
150°C	12 min	0.87 MPa
100°C	95 min	0.94 MPa

Since, by Tukey's test, there are no significant differences between these three healing processes. This means that for any of these cases the curing methodology that will be applied is statistically irrelevant. therefore, it is more advantageous to choose the one that is faster and that spends less energy.

3.3. Analysis of the control group at room temperature

Analyzing now a group of extra experiments that were carried out with the purpose of comparing the treatments made at high temperatures and the treatment at room temperature, the results are presented in Tab. 6. This control group had the curing process done at a temperature of approximately 25 °C in the 3 weeks' time.

Table 6. Results for room temperature.

Cure temperature	Cure time (weeks)	Shear resistance (MPa)					Average ± Standart Deviation (MPa)
		PB1	PB2	PB3	PB4	PB5	
25 °C	3	0.16	0.16	0.15	0.14	0.17	0.16 ± 0.01

PB: proof body

The manufacturer states that the adhesive, under a tear test, done after three weeks cure at about 25 °C, has 1.07 MPa strength. It is observed that the shear strength of this adhesive evaluated in this work under the same conditions is substantially lower. This result indicates that the evaluated adhesive has mechanical resistance under pullout stress greater than under tensile stress. Or even that the time of three weeks is not a cure time sufficient for a complete cure at room temperature. More days would be needed for the adhesive to achieve maximum strength.

3.4. Response surface

For analysis of the response surface, in relation to temperatures, -1, 0 and 1 correspond to 100 °C, 150 °C and 200 °C, respectively as presented in Fig. 2. In relation to the curing times, 0 indicates the intermediate time (indicated by the manufacturer) and -1 and 1 indicates times with variation of 20% for less and more in relation to the time suggested by the manufacturer. The shear resistance is vertically presented in MPa.

The response surface indicates the dark red area as the region where the highest mechanical strengths are obtained. This region corresponds to the treatment of 150 °C with the shortest cure times (8, 10 and 12 minutes). In turn, the bright red and orange region indicates medium mechanical strengths. To this region corresponds the treatment of 100 °C (76, 95 and 114 minutes), and the yellow and green regions correspond to the smallest resistances whose treatment was 200 °C with the smallest times of cure (2, 5 and 3 minutes).

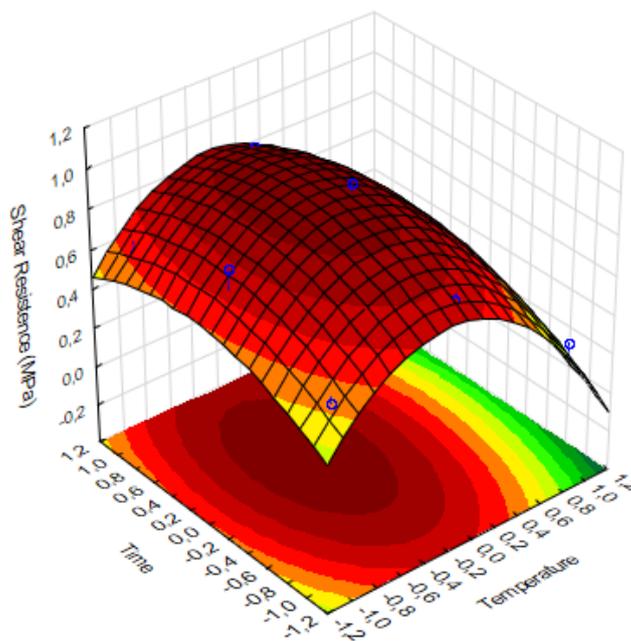


Figure 2. Response surface for data obtained experimentally.

The response surface is a way of observing optimization points and for this case, the treatments made at a temperature of 150 °C seem to be the best treatments in view of the increased mechanical resistance.

4. CONCLUSIONS

The influence of curing time and temperature variations on shear strength was analyzed. Statistical analysis of variance (ANOVA) and Tukey's test were used to compare the means of the results, using reliability in the 95% measurements. The main results and conclusions were: 1. cure done at room temperature does not show good results. Its resistance was approximately 75% lower than the average obtained at 100 °C and 150 °C and 40.7% lower than the average obtained at 200 °C; 2. curing the adhesive using high temperatures is beneficial for shear strength. Even the lowest resistances obtained with processing at high temperatures were higher than the average resistance obtained in the curing process at room temperature; 3. the manufacturer's recommendations have not always been presented as good alternatives for increasing adhesion resistance. For the temperature of 200 °C the indicated time was inappropriate; presenting results 57.8% below those presented for cures done at 100°C and 150 °C. However, at 100 ° C and 150 °C, the time indicated by the manufacturer showed the optimal time for achieving the best adhesive properties; 4. The highest strengths occurred for the curing processes using 150 °C for 12, 10 and 8 minutes and using 100 °C for 95 minutes, obtaining results of, respectively, 0.87, 0.99, 0.74 and 0.83 MPa under tensile test; 5. from the results of the analysis of variance (ANOVA) it is possible to notice that the great factor influencing the resistance is the cure time. 6. The analysis of the response surface shows that the optimization region is around the temperature of 150 °C.

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6. REFERENCES

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7. RESPONSIBILITY NOTICE

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