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To prevent the preload loss, threadlockers are frequently used in threaded connections (e.g. to avoid self-loosening). Threadlockers are generally anaerobic adhesives and are commercially available either as semi-solid (pre-applied) or as liquid (to be applied just before tightening). Previous research deals with the frictional behavior of threaded joints with applied threadlocker. However, fasteners with applied threadlocker may sometimes operate at variable levels of temperature and humidity. A literature survey indicates there is a lack of data about the strength of joints with added threadlocker when they operate at different temperature/humidity levels with respect to conventional environmental conditions. In this research, the aforementioned issue has been tackled by means of an experimental campaign performed on liquid medium/high strength anaerobic adhesives (respectively Loctite 243 and Loctite 270) applied to M10, 8.8 grade, zinc-coated screws. Following complete adhesive polymerization, the joints were aged in an environmental chamber, accounting for four combinations of temperature (room temperature – 90°C) and humidity (20% – 90%) for one week. The joints were then untightened, sampling the breakaway torque. Statistical analyses of the data were carried out, to assess the influence of each parameter on the breakaway torque of the aged joint, thus deriving useful data for design.

Keywords: bolted joints, threadlocker, temperature, humidity, ageing

List of symbols:

ANOVA	Analysis of Variance
A_t	Stress area [mm^2]
d	Nominal diameter of the screw [mm]
d_2	Pitch diameter [mm]
d_3	Minimum diameter [mm]
d_t	Stress diameter [mm]
d_b	Mean diameter at the underhead [mm]
DoF	Degrees of freedom
Fcalc.	Fisher's ratio [-]
F_V	Axial preload force [N]
MSQ	Mean Squares (general term)
p	pitch [mm]
p-v.	p-value
RT	Room temperature of 20°C [°C]
R_{p02}	Stress at 0,2 % nonproportional elongation [MPa]
SSBC	Sum of Squares Between Columns
SSBR	Sum of Square Between Rows
SSQ	Sum of Squares (general term)

SSW	Sum of Squares Within Columns
SSI	Interaction sum of squares
TSS	Total Sum of Squares
T	Tightening torque [Nm]
T_{th}	Thread torque [Nm]
Tr	Release torque [Nm]
Temp	Temperature [°C]
W_t	Torsional modulus [mm^3]
μ_b	Friction coefficient in the underhead [-]
μ_{th}	Friction coefficient in the thread [-]
ν	Screw utilization factor [-]
σ_{VM}	Equivalent von Mises stress [MPa]
%rh	Relative humidity [-]

1. Introduction

Adhesives are nowadays widely used in the field of mechanical joints since they provide a reliable design solution. They generally provide an acceptable strength with respect to specification, along with lightweight properties thanks to the high strength/weight ratio. Moreover, they can provide other features, such as the improvement of corrosion or fretting resistance [1]. Several studies have been carried out with regard to anaerobic adhesives, to investigate the effect of the pressure acting on the bonding on its static strength [2–6]. The same studies propose some design tips regarding the joint most suitable proportioning. In particular in [6], an experimental campaign was carried out to assess the influence of the aspect ratio (coupling length over diameter) on the shear strength of press-fitted and adhesively bonded joints. Focus was placed on the influence on strength of the clearance/interference levels between the shaft-hub coupling. It was pointed out that the joint overall strength takes advantage of the capability of the adhesive of filling the voids among the roughness crests, thus incrementing the actual mating surface. Therefore, in hybrid joints, when adhesive is added, a remarkably high coupling strength can be achieved, also preventing the mating surfaces from fretting wear. As a consequence, anaerobic adhesives can be used not only to replace a conventional coupling but also to integrate, thus improving the mechanical strength. Anaerobic adhesives are indeed widely used in the field of threaded connections, in order to avoid self-loosening. The adhesive, commonly regarded as threadlocker, has the capability of safety locking the threaded fastener against self-loosening that may be induced by vibration or service loads. In addition, threadlockers are able to provide an additional protection against moisture and corrosion by sealing the voids between the screw and the nut threads [7, 8]. These adhesives

are generally anaerobic acrylic and are able to cure at room temperature, when in contact with a metal in the absence of oxygen. The cure is generally affected by the physical properties of the involved metal and by the oxygen concentration. The most frequently used metals for threaded fasteners (e.g. steel and aluminum alloys) are active enough to cure the adhesive rapidly. Threadlockers are commonly classified as weak, medium, and strong, based on their achievable strength upon polymerization, and regarding their tribological effect upon tightening. Regarding this point, the capability of medium and strong threadlockers of lubricating the mating surfaces upon tightening is investigated in [8]. Some studies dealing with the beneficial properties of threadlockers are available in the technical literature, e.g. in [9, 10], but must be more properly regarded as internal studies by suppliers. A low number of studies on threadlockers are available in the scientific literature, and most of them deal with breakaway untightening torque increment as an effect of threadlocker application [11–13].

Motivations of this study arise from a lack of knowledge regarding the influence of the combination involving temperature and humidity on the static shear strength of threadlockers. On the other hand, it is possible to find many studies regarding the same topic, when conventional structural adhesives are involved. Several authors investigated the effect of hygroscopic ageing of structural adhesives [14–23]. These kinds of adhesive tend to become more ductile and weaker, if exposed to environments. Other authors investigated the effect of surface treatment, aimed at improving durability and they found out it is indeed an important parameter with specific regard to zinc-plated surfaces [24, 25]. Some authors investigated the ageing behavior of high temperature anaerobic adhesives,

highlighting a correlation between the observed adhesive degradation and the variation of some fracture mechanics parameters of the joint. [26].

Other authors in [27] investigated the effect of several environmental conditions (environmental condition, cycles from 20 to 80°C at 20% relative humidity and cycles from 20 to 80°C at 85% relative humidity, 12 hours each condition, two cycles per hour). This study revealed the improvement of the adhesive static strength after a suitable thermal cycling but also the worsening of the adhesive ductility properties. ASTM D5363 – 16 Standard [28] deals with the test methods for single component anaerobic adhesives, including as well the thermal ageing test. Nonetheless, the same standard does not provide recommendations about hygroscopic ageing test methods. Therefore, this study aims at filling the gap in the field of the hygroscopic and thermal behavior of two anaerobic threadlockers with medium and high strength.

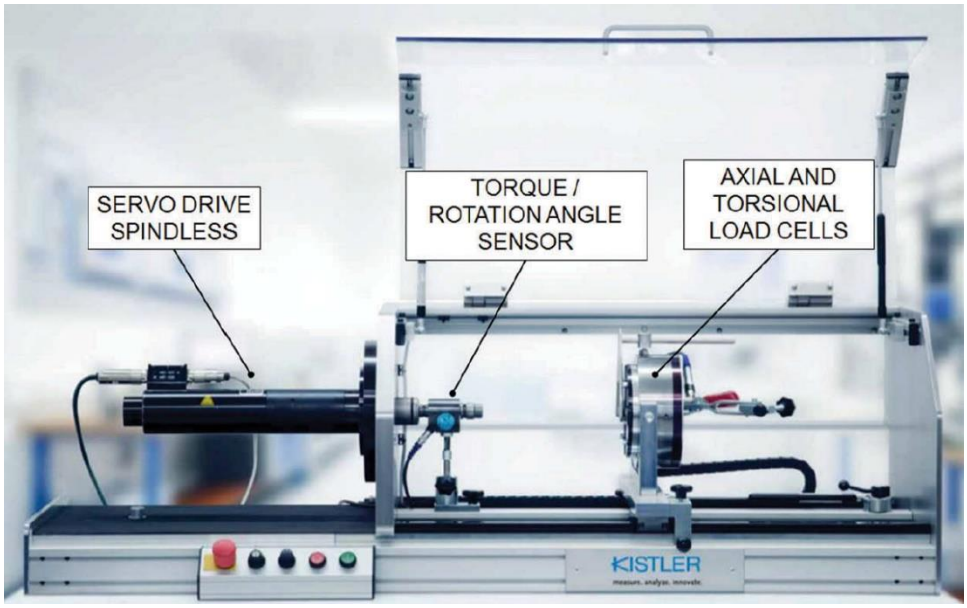


Figure 1 - Kistler ANALYSE test bench.

2. Materials and methods

Standard M10 hexagonal head screws [29] class 8.8 [30] were used. Zinc plated screws were coupled with Zinc plated hexagon nuts [31]. The bolts were assembled on cylindrical sleeves made of grey cast iron EN-GJL-250 [32]: suitable plain washers [33] were installed both at the screw underhead and under the nut. The assembly phase was carried out with the aid of a Kistler ANALYSE test bench (Kistler, Winterthur, Switzerland), whose picture is provided in Fig. 1. This test bench is capable of a maximum tightening torque of 500 Nm and a maximum tightening speed of 300 rpm. A preliminary test was carried out, in order to evaluate the friction coefficients of the joint at first tightening: μ_b and μ_{th}. This test included 5 replicated trials, each of them were performed with unused washers, screws, and nuts. A drawing of the specimen is shown in Fig. 2a whereas the key dimensions of the joint are reported in Tab. 1 and Fig. 3. Two threadlockers were involved in the experiment: Loctite 243 (Henkel Italia S.r.l, Milano, Italia), classified as medium strength, and Loctite 270 (Henkel Italia S.r.l, Milano, Italia). The latter is a high strength acrylic dimethacrylate ester. Each threadlocker was applied on the threaded portion of the screw, complying with the manufacturer's instruction, before every test (Fig. 2b). The friction coefficients upon first tightening were used to properly estimate the most suitable tightening torque for the main experimental campaign. The tightening torque was chosen, in order to achieve a screw utilization of $\nu \approx 0.9$. This value is a widely used value in many practical applications [34]. The utilization factor of the screw ν was evaluated by means of

Motosh's Equation [35], comparing the von Mises stress in the shank σ_{VM} with the yield strength of the material (R_{p02}=640MPa for a 8.8 grade screw [30]) as follows (Eq.s 1-3):

$$T_{th} = F_V \cdot (0.159 \cdot p + 0.577 \cdot \mu_{th} \cdot d_2) \tag{1}$$

$$\sigma_{VM} = \sqrt{\left(\frac{F_V}{A_t}\right)^2 + 3 \cdot \left(\frac{T_{th}}{W_t}\right)^2} \tag{2}$$

$$\upsilon = \frac{\sigma_{VM}}{R_{p02}} \tag{3}$$

Both tightening and release tasks were performed by means of the aforementioned Kistler Analyse System. As above, a medium and a high strength threadlocker were tested: 20 specimens for each of them were set up. After fastening, which was performed at 10rpm, while applying torque to the screw head, and constraining the nut, the specimens were cured for 72 hours at room temperature (RT). After cure, the specimens were thermal and hygroscopic aged by means of a humidity chamber Memmert HCP105 (Memmert GmbH + Co. KG, Schwabach, DE), whose temperature-humidity working range is Temp = [RT+7°C - +90°C] ±0.1°C, %rh = [20%-95%] ±0.5%.

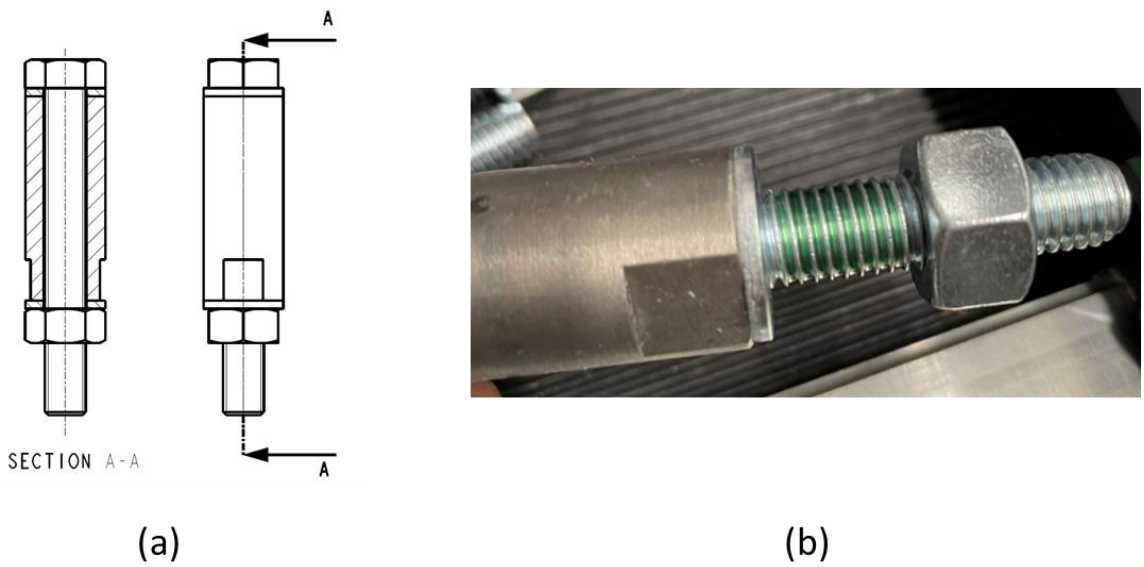


Figure 2 – Specimens assembly: (a) drawing of the test specimen (not in scale); (b) application of the adhesive on the screw.

Table 1 – Key features of the investigated joint with reference to Fig. 3

d	10	mm
p	1.5	mm
d_2	9.026	mm
d_3	8.160	mm
d_b	13.10	mm
d_t	8.593	mm
A_t	58	mm^2
W_t	125	mm^3

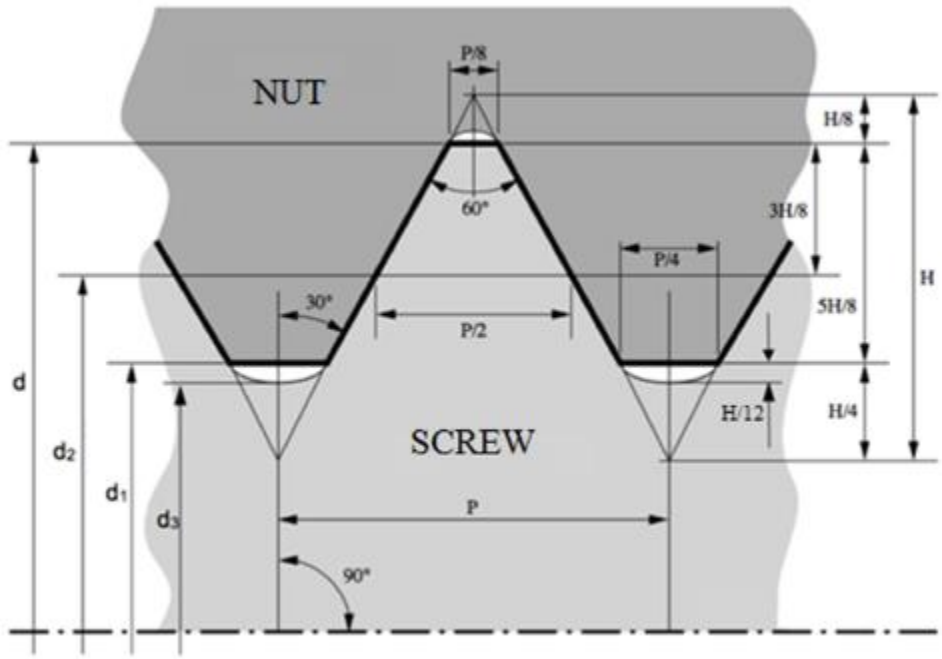


Figure 3 – Fundamental dimensions of the investigated joint

For both threadlockers, a two-parameter/two-level experiment was designed, whose resume is reported in Tab 2. It is worth noticing, the combination Low temperature – High humidity is characterized by a temperature of 35°C. This occurrence is related to the operating range of the humidity chamber, which cannot achieve %rh = 90% at 20°C. However, no significant effect is expected in the light of the small temperature gap. All the specimens were aged for 1 week [36], involving five samples per combination. After ageing, each specimen underwent untightening at 10 rpm, carried out by means of the aforementioned Kistler device. The untightening operations for the four combinations were carried out at room temperature, rotating the head, while the nut was fully constrained by a suitable fixture. During the untightening, upon each trial, the entire torque-angle diagram was sampled. A typical torque angle-curve is displayed in Fig. 4. The key parameter that was recorded during the experimentation and subsequently processed is the breakaway torque, namely the peak of the release torque curve (T_r in Fig. 4). The torque-angle plot

provides at a glance the typical response of a structural adhesive and its trend may be split into three zones. Initially, a linear behavior is present (indicating a linear-elastic response); this is then followed by a sudden breakage of the adhesive layer, corresponding to the decoupling torque peak. In the third phase, a smooth decrease of torque takes place, indicating a gradual joint release. For data processing purposes, the breakaway torque was divided by the tightening torque, in order to determine the normalized breakaway torque of the threadlocker-treated fastener as shown below (Eq. 4):

$$\alpha = \frac{T_r}{T} \tag{4}$$

Table 2 - Design of experiment

Specimen type	Curing method	Hygroscopic and thermal ageing			
		Temperature [°C]	Humidity [%rh]	Ageing time [weeks]	Replicas
Low temperature Low humidity	20°C for 72h	20	20	1 week	5
Low temperature High humidity		35	90		
High temperature Low humidity		90	20		
High temperature High humidity		90	90		

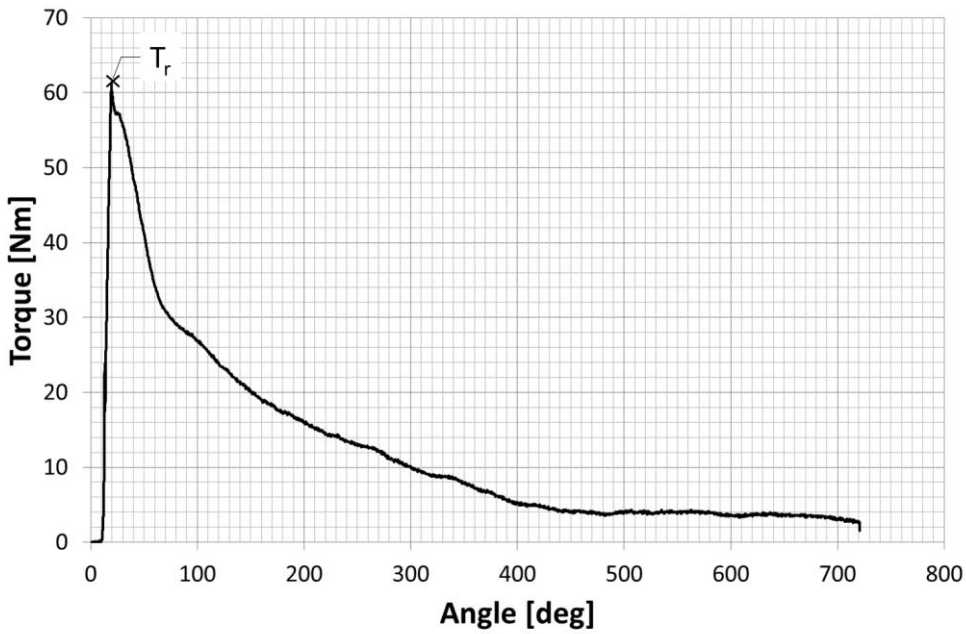


Figure 4 – Torque-angle trend of a typical release test.

It may be argued that breakaway torque values may have been normalized with respect to the corresponding breakaway torques in dry conditions, (i.e: without threadlocker). Proceeding this way would have led to ratios highlighting the beneficial contribution of the applied threadlockers at enhancing the joint strength against loosening. However, these ratios would have involved untightening torques in completely different tribological conditions: a torque affected by metal-to-metal friction (at the denominator) and a torque mainly affected by adhesion (at the numerator). Conversely, rationalizing the retrieved breakaway torques for threadlocker-reinforced fasteners with respect to the corresponding tightening torques (according to Eq. 4) makes it possible to properly compare results obtained in different conditions. Regarding this point, it must be remarked that, even using a torque wrench, it is not feasible to tighten all the samples by the same torque of T=50 Nm: a slight scatter is inevitably present, as in Tab.s 3-4. It is clear that the higher the

tightening torque, the higher the achievable release torque. From this point of view, normalization is able to cancel the possible bias arising from this scatter. In addition, the so computed ratios may be a rough but efficient reference for joint strength prediction, based on its tightening condition.

3. Results

The tightening torque was estimated by the aforementioned equations (Eq.s 1-3), based on the outcome of the frictional pre-tests, in order to achieve a utilization factor of $\nu \approx 0.9$. The following friction coefficients were obtained, regardless of the utilized threadlocker: μ_b=0.14 and μ_{th}=0.21. A constant tightening torque T=50 Nm was therefore adopted for the subsequent experiment involving both specimen types. This torque provides a mean axial force on the shank F_v=22.75 kN and an actual utilization factor ν=0.897. The experimental results in terms of tightening torque, breakaway torque and normalized breakaway torque for the trials utilizing LOCTITE 243 are collected in Tab. 3, with the temperature parameter being associated to the rows and the humidity parameter to the columns. The results of the experimental test involving LOCTITE 270 samples are collected in Tab. 4.

The results in terms of normalized breakaway torque are also collected for the sake of clarity in the bar graphs in Fig. 5 and Fig. 6. In particular, Fig. 5 refers to the LOCTITE 243 specimen set, whereas Fig. 6 deals with the experiments with applied LOCTITE 270. The white bars refer to the low humidity condition, whereas the grey ones are related to the high humidity condition. Temperature level is reported in the horizontal axis; moreover, each chart is completed by maximum-minimum scatter bands.

Table 3 - Experimental LOCTITE 243 data: tightening torque (T), breakaway torque (Tr) and normalized breakaway torque (α) of the threadlocker

		Humidity					
		Low			High		
		Tr [Nm]	T [Nm]	α [–]	Tr [Nm]	T [Nm]	α [–]
Temperature	Low	54.01	49.90	1.08	56.59	51.00	1.11
		56.30	50.40	1.12	56.82	50.50	1.03
		53.29	49.60	1.07	50.91	49.20	1.03
		48.17	49.60	0.97	55.32	49.70	1.11
		50.27	50.70	0.99	52.57	49.80	1.06
	High	61.78	51.20	1.21	65.70	50.70	1.30
		62.64	50.90	1.23	60.80	49.40	1.23
		60.27	49.70	1.21	61.40	49.90	1.23
		67.71	49.90	1.36	60.60	49.10	1.23
		61.27	50.70	1.21	55.90	50.20	1.11

Table 4 - Experimental LOCTITE 270 data: tightening torque (T), breakaway torque (Tr) and normalized breakaway torque (α) of the threadlocker

		Humidity					
		Low			High		
		Tr [Nm]	T [Nm]	α [–]	Tr [Nm]	T [Nm]	α [–]
Temperature	Low	62.76	51.62	1.22	66.01	51.75	1.28
		65.50	51.62	1.27	67.65	51.32	1.32
		58.83	51.55	1.14	57.18	51.50	1.11
		70.91	51.67	1.37	57.45	51.82	1.11
		66.25	51.75	1.28	63.80	51.80	1.23
	High	70.94	51.97	1.36	76.46	51.85	1.47
		61.34	51.82	1.18	70.53	52.74	1.34
		65.78	52.34	1.26	69.31	51.35	1.35
		80.47	51.82	1.55	72.83	52.09	1.40
		58.21	52.07	1.12	67.25	51.80	1.30

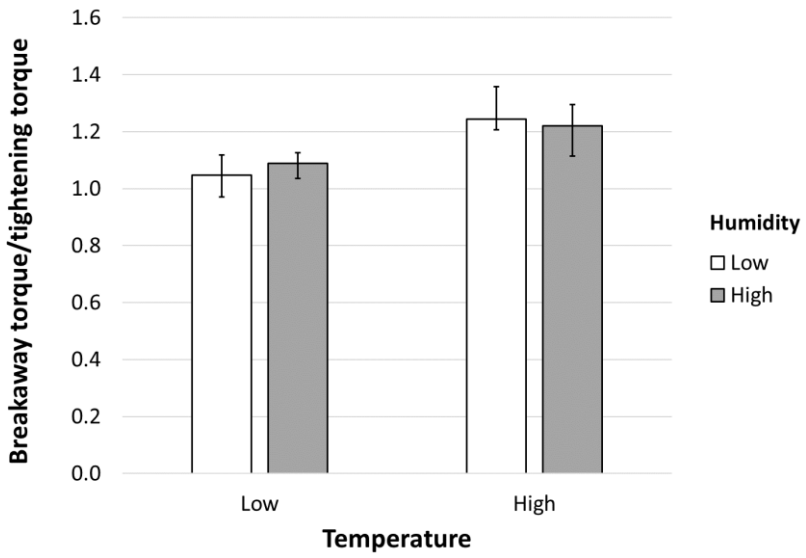


Figure 5 – Chart of the experimental results for LOCTITE 243 specimens in terms of normalized breakaway torque, with max-min scatter bands

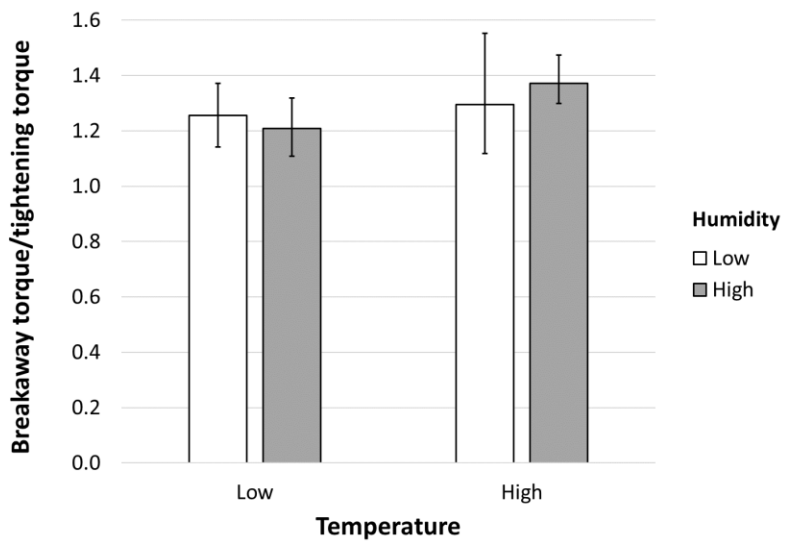


Figure 6 – Chart of the experimental results for LOCTITE 270 specimens in terms of normalized breakaway torque, with max-min scatter bands

4. Discussion

The results shown in in Fig. 5 indicate that temperature seems to have a beneficial effect on the normalized breakaway torque whereas the humidity shows a lower apparent influence on the output variable. In order to better highlight the actual effectiveness of the

two parameters (temperature and humidity) on the output variable, the data were processed by means of the statistical tools of Analysis of Variance (ANOVA) and Fisher's test [37]. In particular, a two-way ANOVA was run to assess the effects of the investigated factors on the normalized breakaway torque. A subsequent Fisher's test was applied to establish the significance of these effects at the 5% significance level. The outcome of this analysis, reported in Tab. 5, confirms that the temperature has a significant effect (p-value in the order of 10^{-5}) on the output parameter of the analysis. On the other hand, humidity as well as the interaction between the two parameters do not significantly affect the breakaway torque. Therefore, the retrieved results indicate that exposure to temperature levels as high as 90°C for one week increases the performance of a medium strength threadlocker.

Table 5 - Results analyzed by two-way ANOVA and f-test for LOCTITE 243 threadlocker

	SSQ	DoF	MSQ	Fcalc.	p-value	Significance
SSBR	0.13538	1	0.13538	38.56	$1.25 \cdot 10^{-5}$	**YES**
SSBC	0.00042	1	0.00042	0.118	$7.352 \cdot 10^{-1}$	**NO**
SSI	0.00487	1	0.00487	1.388	$2.559 \cdot 10^{-1}$	**NO**
SSW	0.05617	16	0.00351			
TSS	0.19684	19				

In order to justify this outcome, the fracture surfaces of the threadlocker were observed (Fig. 6). It can be pointed out the specimens aged at low temperature exhibit a lack of polymerization at some threads, despite initial cure according to the adhesive supplier

recommendations. Conversely, after aging at high temperature, the threadlocker looks completely polymerized on the whole engaged length. As a consequence, 90°C aging has a beneficial effect on the threadlocker strength: this outcome suggests that the adhesive polymerization continues under high temperature exposure, until it covers the entire engaged length. A previous study [27], has shown that the after post-polymerization heating, may produce some degree of embrittlement of the adhesive. In order to verify this occurrence, two torque-angle curves are compared in Fig. 8. This plot shows two curves, both relevant to the case of low humidity: the solid curve belongs to a specimen aged at low temperature, whereas the dashed line refers to a specimen aged at high temperature.

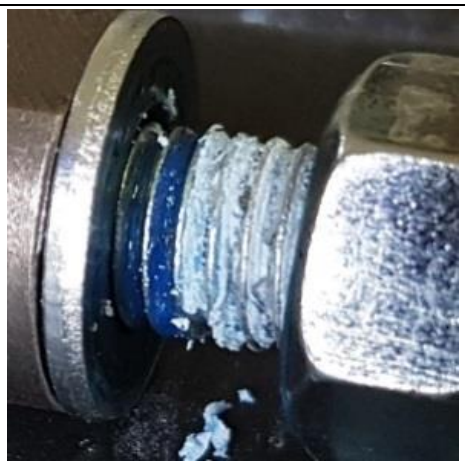
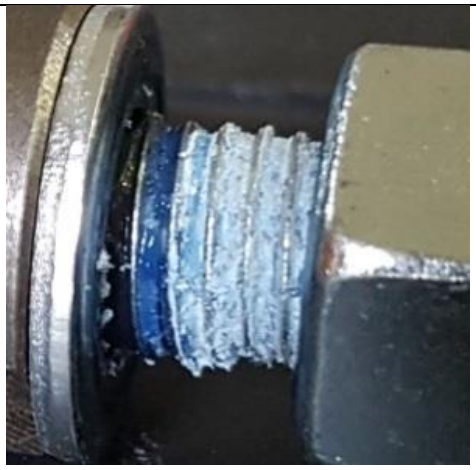
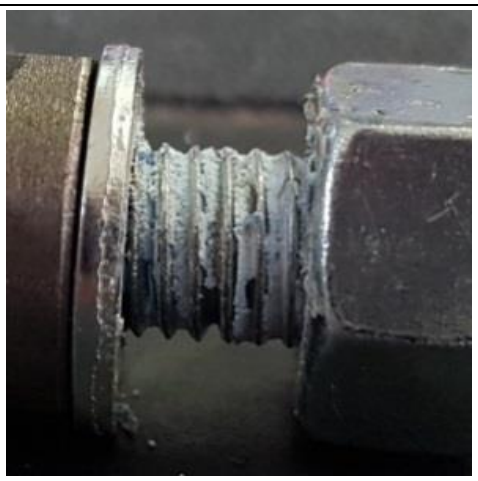
T=20°C, rh%=20%	T=35°C, rh%=90%
	
T=90°C, rh%=20%	T=90°C, rh%=90%
	

Figure 7 - Photographs of LOCTITE 243 specimens after ageing and release

The low temperature specimen exhibits an adhesive failure around 50Nm, whereas the high temperature leads to an incremented strength that leads to adhesive breakage at 62Nm. Moreover, in the first part of the curve (linear-elastic phase) the two plots have similar trends, with approximately the same slope under the same torque. Therefore, there is no experimental evidence that embrittlement occurs for the high temperature aged specimen, as it would be highlighted by a much steeper rise.

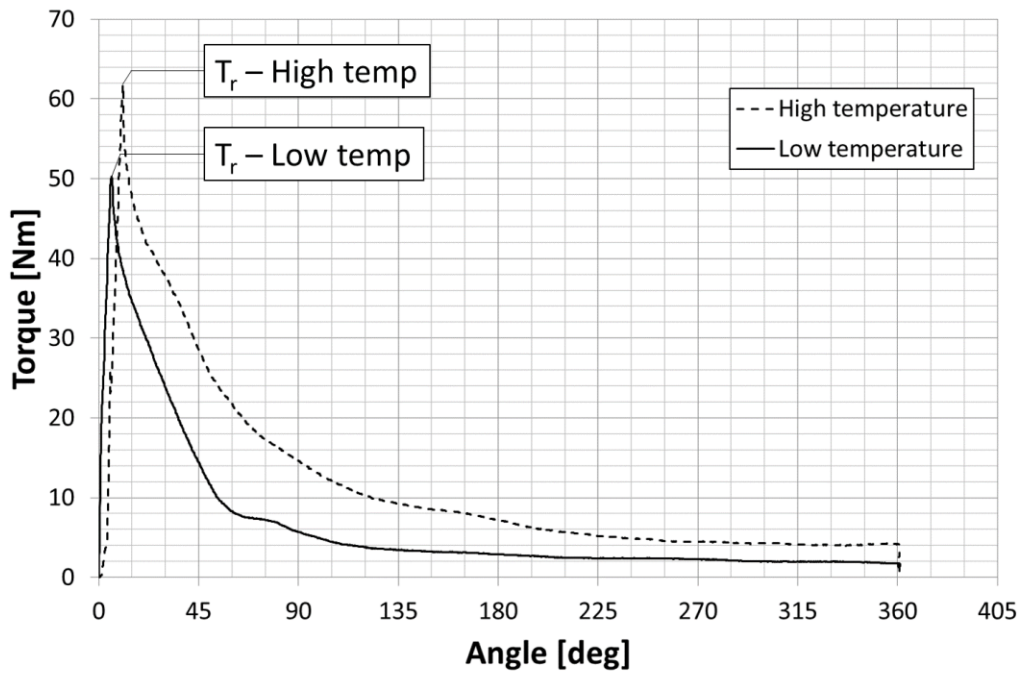


Figure 8 - Torque-angle plots of two LOCTITE 243 specimens, aged at different temperature levels and same humidity level (high – 90%rh)

The same analyses presented for the LOCTITE 243 was then carried out with regard to LOCTITE 270. Looking at the results shown in Fig. 6 it is possible to infer that neither temperature nor humidity seem to affect the statical strength of the studied threadlocker (again, in terms of normalized breakaway torque). Anyway, the data were processed by the aforementioned tools. The outcome of this analysis (reported in Tab. 6) is that none of the studied parameters has a significant impact on the output parameter. The interaction between the temperature/humidity parameters is also negligible. However, it must be pointed out that the temperature, has a p-value ($6.1 \cdot 10^{-2}$) that is very close to the significance threshold of 5%, meaning the effect of temperature would turn to be significant at the 6.1% significance level.

Table 6 - Results analyzed by two-way ANOVA and f-test for LOCTITE 270 threadlocker

	SSQ	DoF	MSQ	Fcalc.	p-value	Significance
SSBR	0.05114	1	0.05114	4.077	$6.056E{\cdot}10^{-2}$	**NO**
SSBC	0.00109	1	0.00109	0.087	$7.717{\cdot}10^{-1}$	**NO**
SSI	0.01900	1	0.01900	1.514	$2.363{\cdot}10^{-1}$	**NO**
SSW	0.20073	16	0.01255			
TSS	0.27197	19				

A particular focus must be placed on this outcome, because it indicates that temperature effect is not completely negligible. Therefore, adhesive fracture surfaces were carefully analyzed after untightening (Fig. 9). Similar remarks as above may be made, highlighting some small spots of not polymerized adhesive following low temperature aging and a complete polymerization after high temperature aging, regardless of humidity levels. However, the differences between the two highlighted conditions appear to be smaller than what highlighted for the medium strength threadlocker. Two torque-angle curves (Fig. 10) were then analyzed to assess the occurrence of threadlocker embrittlement as an effect of high aging temperature.

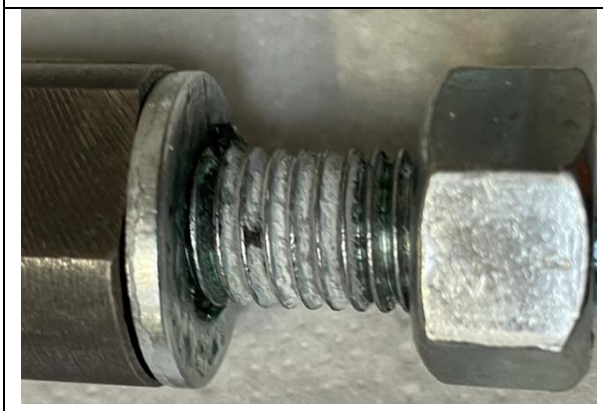
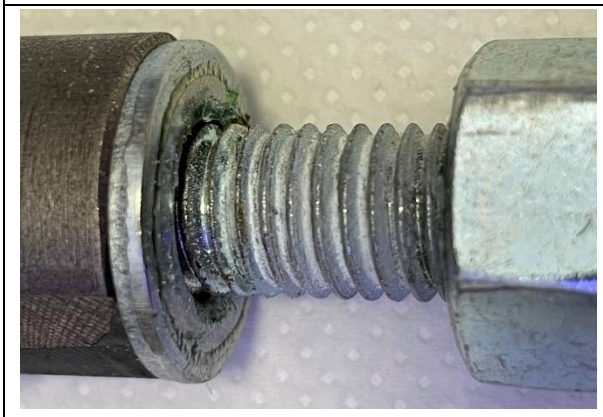
T=20°C, rh%=20%	T=35°C, rh%=90%
	
T=90°C, rh%=20%	T=90°C, rh%=90%
	

Figure 9 - Photographs of LOCTITE 270 specimens after ageing and release

In particular, the plot shows the torque-angle curves for two different specimens aged at the same humidity level (rh%=90%) and at different temperature (low and high temperature). The solid curve belongs to a specimen aged at low temperature, whereas the dashed one refers to a sample aged under high temperature. The low temperature specimen exhibits a failure torque around 57 Nm, whereas, under the high temperature, the adhesive failure occurs around 73 Nm. The same trends in the linear-elastic parts of the curves indicates that no embrittlement phenomenon may be observed, based on the retrieved data.

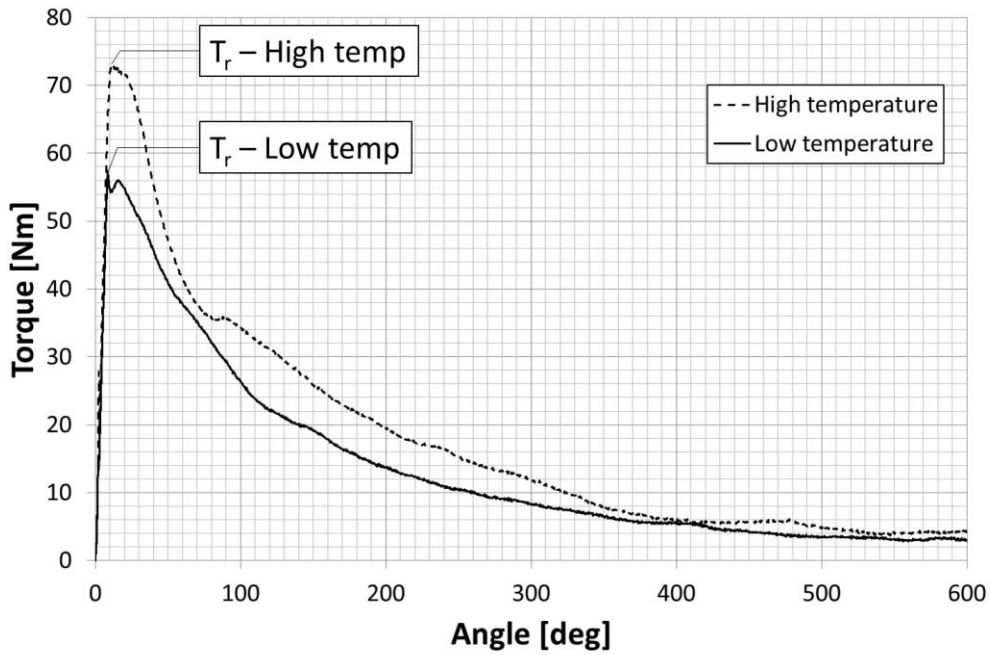


Figure 10 - Torque-angle plots of two LOCTITE 270 specimens, aged at different temperature levels and same humidity level (high – 90%rh)

Finally, a comparison of the results achieved for both medium/high strength threadlocker was made (Fig. 11). In order to simultaneously assess the influence of each parameter (temperature, relative humidity and threadlocker type) on the normalized breakaway strength, a three-factor ANOVA was used to process the data. For the sake of synthesis, the detail of such analysis is not here reported. As expected, the threadlocker choice has a highly significant effect on the output parameter (p-value=$6.8 \cdot 10^{-4}$). The temperature also exhibits an overall significant influence on the normalized breakaway torque (p-value=$2.1 \cdot 10^{-4}$). Humidity, as well as all the interactions between the factors, confirm to be not significant.

As a final remark, it may be observed that, considering averaged values over the humidity levels, high temperature ageing leads to a breakaway torque ratio incremented by 16.6%

for the medium strength threadlocker. The high strength threadlocker leads to an improved strength of the joint, as its average torque ratio with aging at low temperature corresponds to that for the medium strength threadlocker after beneficial high temperature aging. In this case, temperature aging leads to a lower increment, by 8.1%, which is in a borderline condition regarding its significance. This behavior may also be interpreted in the light of the data in the datasheets for the two adhesives. As for the medium strength LOCTITE 243, long time aging around 100°C yields a generally positive strength enhancement. Consequently, it is reasonable that the breakaway torque is remarkably incremented, following a 90°C aging. Conversely, for the high strength threadlocker, LOCTITE 270, a 100°C aging leads to an unchanged or even a slightly reduced strength. Therefore, it is again consistent the effect of temperature is much lower in this case.

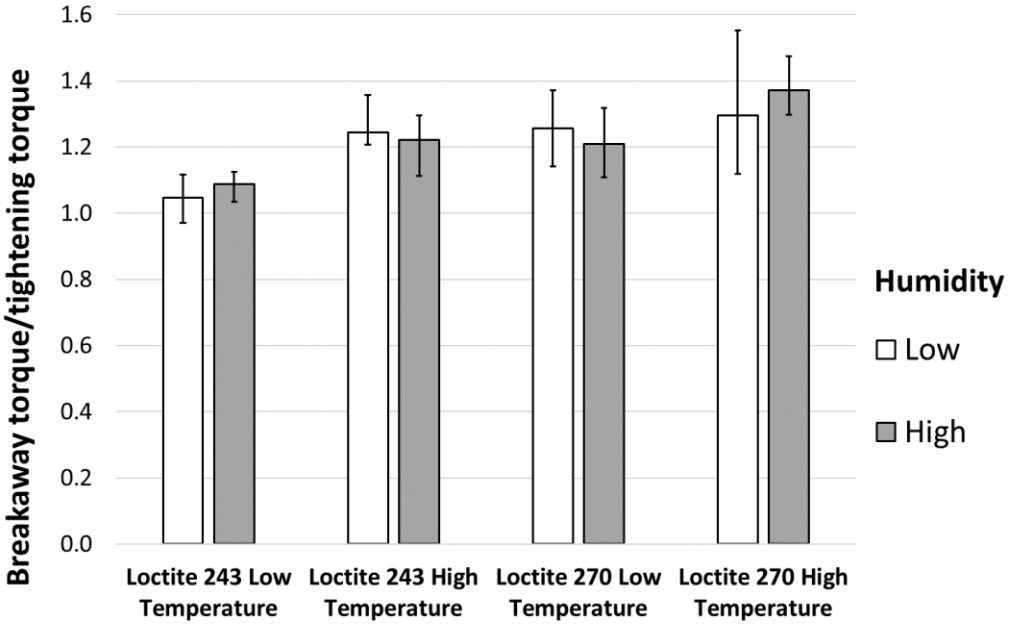


Figure 11 – Comparison of all the results achieved for both threadlocker

5. Conclusions

The purpose of this study was to investigate the ageing behavior of a medium/high strength threadlockers, under combined thermal and hygroscopic condition. The experiments were carried out on M10, class 8.8, zinc-plated screws coupled with suitable washers and nuts. The assembly phase included also grey cast iron sleeves. Specimens were tightened using both Loctite 243 and Loctite 270 threadlockers, respectively exhibiting medium and high strength. Subsequently, the adhesive was cured at room temperature for 72 hours to allow a complete polymerization. The design of the experiment included two parameters for each threadlocker (temperature and relative humidity) that were switched on two levels each generating four specimen groups for each threadlocker. Five replicas for each condition were tested. The experimental results in terms of normalized breakaway torque (breakaway torque over tightening torque) were processed by the statistical tools of two-way ANOVA and Fisher's test, in order to assess the influence of the parameters on the threadlockers strength. Regarding the medium strength threadlocker the temperature proved to be highly effective on the static response of the adhesive, whereas humidity as well as interaction proved to be ineffective. The results indicate the 90°C aging has the capability of incrementing the joint strength by 16.6%.

The campaign on the high strength threadlocker and subsequent data processing indicated the parameters are not effective at the 5% significant level. Anyway, the effect of temperature parameter is very close to the significance threshold. Considering averaged values, a small increment, in the order of 8.1%, may be observed. Further analyses were carried out. The adhesive fracture surfaces were observed, and it was observed that polymerization seems to continue under high temperature aging, until it completely covers

the engaged length. Conversely, without aging, some spots of unpolymerized adhesive are still present. This behavior is surely responsible for the beneficial effect of high temperature aging. Finally, no embrittlement phenomena of the adhesive were observed. Further development of the present research may involve the evaluation of longer ageing periods or the influence of temperature cycles.

References

[1] Croccolo, D.; De Agostinis, M.; Fini, S.; Morri, A.; Olmi, G. Analysis of the Influence of Fretting on the Fatigue Life of Interference Fitted Joints. In *ASME International Mechanical Engineering Congress and Exposition, Proceedings (IMECE)*; 2014; Vol. 2B. https://doi.org/10.1115/IMECE2014-38128.

[2] Ragni, M.; Castagnetti, D.; Dragoni, E. Experimental Validation of a Simple Shear Strength Model for Hybrid Friction-Bonded Interfaces. *Int. J. Adhes. Adhes.*, **2018**, *83*, 130–136. https://doi.org/10.1016/j.ijadhadh.2018.02.026.

[3] Castagnetti, D.; Dragoni, E. Experimental Investigation and Model Validation of the Shear Strength of Hybrid Interfaces up to Complete Failure. *J. Adhes.*, **2016**, *92* (7–9), 679–697. https://doi.org/10.1080/00218464.2015.1115740.

[4] Dragoni, E.; Mauri, P. Intrinsic Static Strength of Friction Interfaces Augmented with Anaerobic Adhesives. *Int. J. Adhes. Adhes.*, **2000**, *20* (4), 315–321. https://doi.org/10.1016/S0143-7496(99)00062-7.

[5] Croccolo, D.; De Agostinis, M.; Mauri, P.; Olmi, G. Influence of the Engagement Ratio on the Joint Strength of Press Fitted and Adhesively Bonded Specimens. *Int. J. Adhes. Adhes.*, **2014**, *53*, 80–88. https://doi.org/10.1016/j.ijadhadh.2014.01.017.

[6] Croccolo, D.; De Agostinis, M.; Fini, S.; Olmi, G.; Paiardini, L.; Robusto, F. Influence of the Interference Level and of the Assembly Process on the Shear Strength of Loctite 648 Anaerobic Adhesive. *J. Adhes.*, **2019**. https://doi.org/10.1080/00218464.2019.1681268.

[7] Haviland, G. S. *Machinery Adhesives for Locking, Retaining and Sealing.*; 1986.

[8] Croccolo, D.; De Agostinis, M.; Fini, S.; Olmi, G.; Paiardini, L.; Robusto, F. Threaded Fasteners with Applied Medium or High Strength Threadlockers: Effect of Different Tightening Procedures on the Tribological Response. *J. Adhes.*, **2019**. https://doi.org/10.1080/00218464.2019.1679630.

[9] LOCTITE® Threadlockers - Henkel adhesives https://www.henkel-adhesives.com/us/en/products/industrial-adhesives/threadlockers.html (accessed Jun 28, 2021).

[10] J., M.; Dube; Gamwell, W. R. Performance Characterization of Loctite 242 and 271 Liquid Locking Compounds (LLCs) as a Secondary Locking Feature for International Space Station (ISS) Fasteners https://ntrs.nasa.gov/citations/20110011064 (accessed Jun 28, 2021).

[11] Castagnetti, D.; Dragoni, E. Adhesively-Bonded Friction Interfaces: Macroscopic Shear Strength Prediction by Microscale Finite Element Simulations. *Int. J. Adhes. Adhes.*, **2014**, *53*, 57–64. https://doi.org/10.1016/j.ijadhadh.2014.01.016.

[12] Sekercioglu, T.; Kovan, V. Torque Strength of Bolted Connections with Locked Anaerobic Adhesive. *Proc. Inst. Mech. Eng. Part L J. Mater. Des. Appl.*, **2008**, *222* (1), 83–89. https://doi.org/10.1243/14644207JMDA157.

[13] Croccolo, D.; De Agostinis, M.; Fini, S.; Olmi, G. An Experimental Study on the Response of a Threadlocker, Involving Different Materials, Screw Dimensions and Thread Proportioning. *Int. J. Adhes. Adhes.*, **2018**, *83*, 116–122. https://doi.org/10.1016/j.ijadhadh.2018.02.024.

[14] Sugiman, S.; Crocombe, A. D.; Aschroft, I. A. Experimental and Numerical Investigation of the Static Response of Environmentally Aged Adhesively Bonded

Joints. *Int. J. Adhes. Adhes.*, **2013**, *40*, 224–237. https://doi.org/10.1016/j.ijadhadh.2012.08.007.

[15] Sugiman, S.; Crocombe, A. D.; Aschroft, I. A. Modelling the Static Response of Unaged Adhesively Bonded Structures. *Eng. Fract. Mech.*, **2013**, *98* (1), 296–314. https://doi.org/10.1016/j.engfracmech.2012.10.014.

[16] Hua, Y.; Crocombe, A. D.; Wahab, M. A.; Ashcroft, I. A. Modelling Environmental Degradation in EA9321-Bonded Joints Using a Progressive Damage Failure Model. *J. Adhes.*, **2006**, *82* (2), 135–160. https://doi.org/10.1080/00218460600559557.

[17] Liljedahl, C. D. M.; Crocombe, A. D.; Wahab, M. A.; Ashcroft, I. A. The Effect of Residual Strains on the Progressive Damage Modelling of Environmentally Degraded Adhesive Joints. *J. Adhes. Sci. Technol.*, **2005**, *19* (7), 525–547. https://doi.org/10.1163/1568561054352513.

[18] Liljedahl, C. D. M.; Crocombe, A. D.; Wahab, M. A.; Ashcroft, I. A. Modelling the Environmental Degradation of the Interface in Adhesively Bonded Joints Using a Cohesive Zone Approach. *J. Adhes.*, **2006**, *82* (11), 1061–1089. https://doi.org/10.1080/00218460600948495.

[19] Crocombe, A. D.; Hua, Y. X.; Loh, W. K.; Wahab, M. A.; Ashcroft, I. A. Predicting the Residual Strength for Environmentally Degraded Adhesive Lap Joints. *Int. J. Adhes. Adhes.*, **2006**, *26* (5), 325–336. https://doi.org/10.1016/j.ijadhadh.2005.04.003.

[20] Lee, S. Y.; Bruce, K. C. THERMAL STABILITY AND AGING OF SOME NEW ANAEROBIC ADHESIVES FOR STRUCTURAL BINDING. *S.A.M.P.E. Q.*,

1980, *11* (4), 22–29.

[21] Corigliano, P.; Ragni, M.; Castagnetti, D.; Crupi, V.; Dragoni, E.; Guglielmino, E. Measuring the Static Shear Strength of Anaerobic Adhesives in Finite Thickness under High Pressure. *J. Adhes.*, **2021**, *97* (8), 783–800. https://doi.org/10.1080/00218464.2019.1704271.

[22] Castagnetti, D.; Dragoni, E. Experimental Assessment of a Micro-Mechanical Model for the Static Strength of Hybrid Friction-Bonded Interfaces. *J. Adhes.*, **2013**, *89* (8), 642–659. https://doi.org/10.1080/00218464.2012.747179.

[23] Karnolt, C. L. Anaerobic Adhesives for Sheet Metal Assembly. *SAE Tech. Pap.*, **1975**. https://doi.org/10.4271/750140.

[24] Pantoja, M.; Velasco, F.; Broekema, D.; Abenojar, J.; Del Real, J. C. The Influence of PH on the Hydrolysis Process of γ-Methacryloxypropyltrimethoxysilane, Analyzed by FT-IR, and the Silanization of Electrogalvanized Steel. *J. Adhes. Sci. Technol.*, **2010**, *24* (6), 1131–1143. https://doi.org/10.1163/016942409X12586283821559.

[25] Pantoja, M.; Martínez, M. A.; Abenojar, J.; Velasco, F.; Del Real, J. C. Structural and Mechanical Characterization of γ- Methacryloxypropyltrimethoxysilane (MPS) on Zn-Electrocoated Steel. *J. Adhes. Sci. Technol.*, **2010**, *24* (11–12), 1885–1901. https://doi.org/10.1163/016942410X507632.

[26] Cherry, B. W.; Ye, Y. Q. The Behaviour of High-Temperature Anaerobic Adhesives. *Int. J. Adhes. Adhes.*, **1992**, *12* (3), 206–210. https://doi.org/10.1016/0143-7496(92)90055-Z.

[27] Sakai, K.; Nassar, S. A. Failure Analysis of Composite-Based Lightweight

Multimaterial Joints in Tensile-Shear Tests after Cyclic Heat at High-Relative Humidity. *J. Manuf. Sci. Eng. Trans. ASME*, **2017**, *139* (4). https://doi.org/10.1115/1.4034888.

[28] ASTM D5363 - 16. Standard Specification for Anaerobic Single-Component Adhesives (AN). *Reproduction*. https://doi.org/10.1520/D5363-16.2.

[29] ISO 4017 : 2014 Fasteners — Hexagon Head Screws — Product Grades A and B. **2014**.

[30] ISO 898 - 1: 2013 Mechanical Properties of Fasteners Made of Carbon Steel and Alloy Steel Part 1. **2004**, *2004*.

[31] EN ISO 4032:2012 Hexagon Regular Nuts (Style 1) — Product Grades A and B. **2012**.

[32] ISO 1561:2011 Founding — Grey Cast Irons. **2011**.

[33] EN ISO 7089:2000 Plain Washers - Normal Series - Product Grade A. **2000**.

[34] Part, B. Vdi-Richtlinien Ingenieure Vdi 2230. *October*, **2003**.

[35] Motosh, N. Development of Design Charts for Bolts Preloaded up to the Plastic Range. *J. Manuf. Sci. Eng. Trans. ASME*, **1976**, *98* (3), 849–851. https://doi.org/10.1115/1.3439041.

[36] ISO 9142:2003 Adhesives — Guide to the Selection of Standard Laboratory Ageing Conditions for Testing Bonded Joints. **2003**.

[37] Montgomery, D. C. *Design and Analysis of Experiments*; Wiley: New York, 2001.