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Adhesive joint use and aging in food machinery: a case-study on beverage filling systems

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Abstract

The objective of this work is to evaluate the introduction of bonding technology in the assembly of beverage filling machines. In this specific field, bonding has two potential great advantages with respect to welding, that are the speed of application and the absence of thermal distortions. Furthermore, since it presents fewer risks for safety at work, it allows the simultaneous execution of other assembly operations. On the other hand, the use of bonding requires a design process integrated by tests related to the specific application and operating conditions, that in the case of food machinery are: i) bonding of low surface roughness, austenitic stainless steel without surface preparation but degreasing; ii) contact of the joint with the chemically aggressive substances used for cleaning and sanitization. In the present study, first a technical-economical comparison with welding (business case) was done on a component of the structure of a filling machine. Then the adhesive appropriate for the application was identified experimentally with tests on single-lap joints (SLJ) with different surface finishes and subjected to chemical aging under the cleaning and sanitization agents. Finally, the scale-up to a larger and more complex structure was performed by joining two modules of the outer casing of a filling machine.

Keywords

Beverage filling machines, bonding, stainless steel, chemical aging, demonstrator

1. Introduction

The need to reduce lead time and production costs is increasingly marked in the manufacturing industry, especially in the case of made-to-order food machinery where the minimization of the lead time becomes essential for competitiveness.

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36 The assembly technique currently used in the majority of the food machinery joints is welding,
37 a process with a high lead time due to the advancement times that it entails and the need for
38 subsequent pickling of the welded area in order to restore the passive surface layer that protects
39 the weld seam from corrosion. In addition, welding requires highly specialized labor and, in the
40 case of food machinery metallic carpentry, the operation is carried out on thin sheets (1.5-3
41 mm) resulting in distortions that negatively affect the subsequent assembly phases.

42 Structural bonding is gaining more and more application in the industrial field due to the variety
43 and flexibility of solutions it allows [1], but it should be borne in mind that its use requires a
44 design process integrated by tests related to the specific application and operating conditions
45 [2], [3].

46 The objective of this work is to evaluate the introduction of bonding in place of welding in the
47 assembly of beverage filling machines. In this specific field, bonding has two potential great
48 advantages compared to welding, which are the speed of application and the absence of thermal
49 distortions [1]. Furthermore, since it presents fewer risks for safety at work, bonding allows the
50 simultaneous execution of other assembly operations, which would otherwise have had to be
51 suspended while welding. The application of bonding in the construction of food machinery is
52 foreseen in some technical documents [4], [5], but from the point of view of the scientific
53 literature it is still apparently limited to a previous work by some of the authors [6]. It should
54 also be borne in mind that the geometry and materials of the joint must comply with, or be
55 brought back through appropriate measures, to the standards and guidelines of "hygienic"
56 design [4], [5]. Concerning this point, the case-study will concentrate on the application in parts
57 that are not in contact with the beverage, therefore the only concern is to provide a good
58 cleanability of the joint, but there is no potential risk of migration of substances from the
59 adhesive to the bottled product.

60 In the case of food machinery, it is also necessary to evaluate two peculiar aspects: i) the
61 material of the substrates is in majority austenitic stainless steel with low-roughness surface
62 finish for cleanability reasons; ii) the machines are subjected to a periodic sanitization cycle
63 with chemically aggressive substances. Regarding the first point, austenitic stainless steel is
64 considered a material with a high surface energy, therefore abrasion and subsequent cleaning
65 and degreasing already ensure a good resistance of the joint as shown in [7], even though a
66 chemical treatment with mixtures of inorganic acids (sulfuric, nitric, nitrofluoridric) may be
67 more effective [8]. On the other hand, performing such treatment in the manufacturing line of
68 food machines is troublesome for cleanliness and safety reasons, therefore only degreasing and
69 possibly primerization can be easily accepted by manufacturers. The durability of stainless steel

70 joints has been evaluated instead in several studies [9]-[14], in which aging is accelerated by
71 exposure to cycles of high temperature and humidity, or salt spray, but no elements are found
72 regarding the effect of prolonged exposure to the substances used in the sanitization of food
73 machinery, i.e. strong acid and alkaline inorganic compounds. In the study it is therefore
74 necessary to evaluate the effect of different surface finishes after a simple degreasing and,
75 possibly, primerization, and of the aforementioned chemical agents, that are specific of the
76 beverage filling machine cleaning cycle, on the resistance of the joint over time, in order to
77 select the adhesive appropriate for the application in this field. The feasibility study of using
78 structural adhesives in the assembly of the stainless sheet metal has been done with reference
79 to the inner casing of the protection structure of a filling machine. The specific component was
80 chosen as a case-study, but it is representative of numerous other parts of a filling machine,
81 beside in general joints in stainless steel food machinery carpentry.

82

83 **2. Methods**

84 2.1 Case-study description

85 In high production rate beverage filling machines, empty bottles are continuously transferred
86 by a series of handling modules to a rotating structure (also named “carousel”) with nozzles
87 (filling valves) on its periphery to fill the bottle with product (water, Carbonated Soft Drink -
88 CSD, beer, milk, etc..). The bottling area must be protected both from human access for safety
89 reason, and from external bodies and contamination for food safety and hygiene reasons: wide
90 perimetral protections prevent access to the filling zone from operators, while smaller
91 protections close to the nozzles can prevent the access of foreign bodies and other sources of
92 contamination. Inside the protected volume are performed, periodically, cleaning cycles called
93 Surface Cleaning (called in the common practice Cleaning-Out-of- Place or COP), that apply
94 acid and alkaline cleaning products, plus a certain amount of water for preparation and rinsing.
95 A protection type that realize a volume where bottle transfer and filling are performed, is shown
96 in Figure 1.

97



(a)

(b)

Figure 1 – example of protection: a) outer side; b) inner side (machine under construction).

The protection is composed of an inner casing, visible on the left in Figure 1b, which function is to separate the internal of the carousel from the region where the bottles are filled and that is subjected to the COP. The inner casing is made up of calendered, sheet metal sectors, in a number depending on the diameter of the carousel welded along the nip and reinforced by welded strap on the outer face. An outline of the inner casing and of the connection to the carousel is given in Figure 2; the inner casing is moving at the same rotational speed of the carousel. Mechanical and environmental actions are summarized in Table 1.

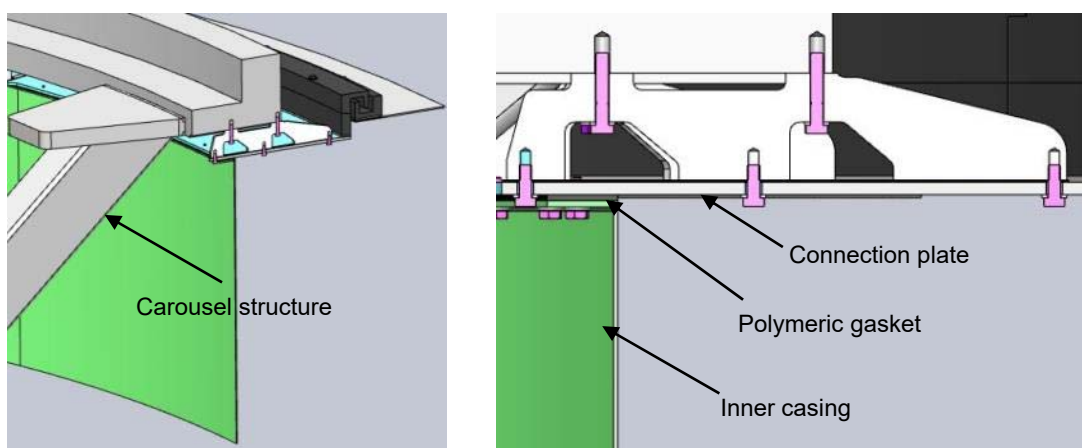


Figure 2 – Outline of the inner casing and detail of its joint with the connection plate.

Table 1 – Mechanical and environmental actions on the inner casing of RE machines.

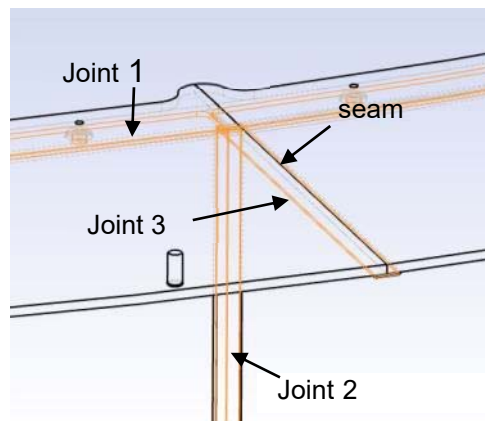
Action	Type
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Mechanical	Centrifugal body force in service
	Inertial body force in case of emergency stop
	Thermal eigenstress during sanitization
	Gravity body force
Environmental	Sanitization agents
	Sanitization temperature (max 40 °C)

114

115 The inner casing has three joint, shown in Figure 3. Joint 1 is a bolting to the carousel
 116 connection plate shown in Figure 2, with the interposition of a polymeric gasket. Joint 2 is
 117 instead a butt-strap welding carried out by TIG (Tungsten Inert Gas) technique. Joint 3 is a butt-
 118 strap TIG welding done in order to seal the seam between the connection plate sectors. The use
 119 of bonded joints would also eliminate the need of the polymeric gasket in Joint 1.

120



121 **Figure 3 – Joints in the inner casing to be substituted by bonding.**

122

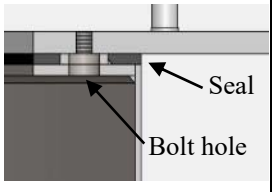



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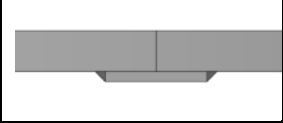
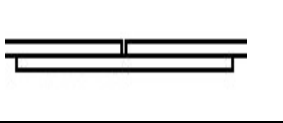
124 The three joints can be easily converted into butt or butt-strap bonded joints as outlined in Table
 125 2, minimizing the changes to drawings and to the manufacturing process and making the bonded
 126 solution applicable also in case of maintenance of existing machines.

127

128

Table 2 – Outline of joints in the inner casing and corresponding bonded solution.

Joint	Present solution	Bonded solution	Bonded area (cm ²)
1			3600
2			90

3			40
---	---	--	----

129

130 The joint 1 has also the structural function to support the weight of the casing and the actions
 131 originating from the rotation of the carousel to which the casing is connected. Although these
 132 actions do not achieve large values, they generate traction on the bond and it is therefore good
 133 to evaluate in advance the level of stress induced on the joint. The average stresses on the joint
 134 are:

135

$$136 \quad \sigma_z = \frac{P}{A} \quad (1)$$

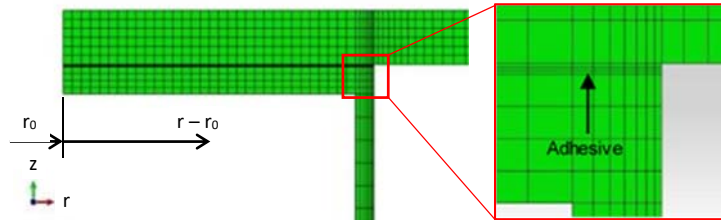
$$137 \quad \tau_{r\theta} = \frac{J \cdot \alpha \cdot R}{I_p} \quad (2)$$

138

139 where P and J are respectively the weight and the moment of inertia of the inner casing around
 140 the rotation axis, A and I_p the area and the polar moment of inertia of the joint and α the angular
 141 deceleration of the carousel during the emergency stop. Given the large bonding area (see Table
 142 2), the resulting average stresses assume extremely low values $\sigma_z = 3.3 \cdot 10^{-3}$ MPa, $\tau_{r\theta} = 0.4 \cdot 10^{-3}$
 143 MPa, so even considering that the real distribution may show stress concentration at the ends
 144 of the joint, that would hardly lead to critical values for the strength. To estimate more in detail
 145 the stress distribution, a finite element analysis of Joint 1 is done using the Abaqus software
 146 (Figure 4). In order to keep the modeling and computational effort at an acceptable level, only
 147 the connection plate (thickness 4 mm), the inner casing (thickness 2 mm) and the interposed
 148 adhesive layer (thickness 0.2 mm) are considered and modeled with axisymmetric two-
 149 dimensional (2D) finite elements. The model is loaded with body forces corresponding to the
 150 gravity and to the centrifugal acceleration produced by the rotation at 12 rotations per minute
 151 (rpm), while the emergency stop deceleration direction is out-of-plane and therefore could not
 152 be accounted for in the 2D model. The adhesive layer is modelled with two rows of four-node
 153 elements and the stresses are evaluated at its midplane. The analysis is linear elastic with the
 154 Young's modulus taken from Table 3 for Adhesive 1 and Adhesive 2, respectively, and a
 155 Poisson's ratio taken as a typical values found in epoxies $\nu = 0.4$. The low stiffness of Adhesive
 156 3 would give for sure a smoother stress distribution than with Adhesive 1 or 2 and Adhesive 4
 157 Young's modulus is intermediate between Adhesive 1 and 2, therefore the results would also
 158 take intermediate values. The analysis revealed that the gravity alone produces a peak of peel
 159 at the outside corner with a value $\sigma_z = 0.02-0.03$ MPa depending on the adhesive, while in

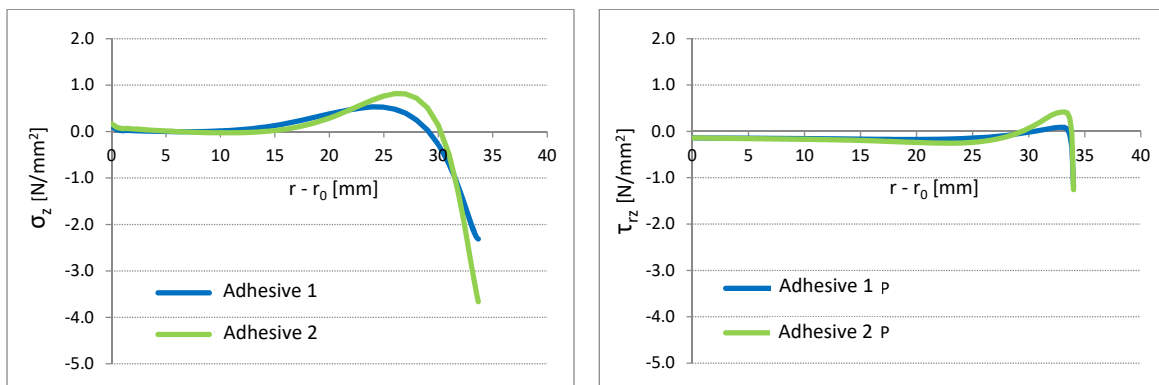
160 service a stress distribution, characterized by both peel and shear, is shown in Figure 5. Taking
 161 the maximum principal stress as uniaxial equivalent stress measure that account for the sign of
 162 peel stress, the most stressed point is located at the peak of peel stress, with a value $\sigma_{PRIN,MAX}$
 163 = 0.57 MPa for Adhesive 1 and $\sigma_{PRIN,MAX} = 0.88$ MPa for Adhesive 2.

164



165
 166 **Figure 4 – axisymmetric finite element model of Joint 1.**

167



168
 169 **Figure 5 – Peel (left) and shear (right) stresses in Joint 1.**

170

171 2.2 Adhesives

172 Technical datasheets of structural adhesives report generally the value of strength under
 173 standard test conditions and on specific materials and surface finishes, which are difficult to
 174 transfer to a generic case where joint shape, environmental conditions, materials and finishes
 175 strongly differ from those of a standard test. It is therefore necessary to carry out a design
 176 integrated by tests, i.e. the experimental evaluation of the behavior of the adhesive in the
 177 specific conditions envisaged for the joint to be made. To verify the performance of the selected
 178 adhesives, an experimental test campaign was therefore set up, verifying both the characteristics
 179 in terms of strength on the specific surface finish and the effect of chemical aging by the COP
 180 agents.

181 For a first experimental campaign, three adhesives were selected, which will later be referred
 182 to for simplicity as Adhesive 1, 2, and 3, respectively. The main features are:

183 Adhesive 1: LOCTITE® Hysol® 9466™ (currently rebranded as LOCTITE® EA® 9466™), a
 184 two-component thixotropic epoxy with high mechanical strength, reaches full strength after 24
 185 h and has an open time (work-life) of about 60 minutes, which allows precise assembly and
 186 possible adjustment of the position of the adherends;

187 Adhesive 2: LOCTITE® Hysol® 9492™ (currently rebranded as LOCTITE® EA® 9492™), a
 188 two-component thixotropic epoxy for high temperatures, reaches full strength after 24 h. It is
 189 declared to be resistant to some chemical agents of interest, such as NaOH and acetic acid, as
 190 well as to water and humidity.

191 Adhesive 3: TEROSON MS 9399, a two-component, flexible MS (Modified Silane) adhesive,
 192 cures completely in 24 h. It allows excellent adhesion on metals without pre-treatment, but
 193 cleaning and degreasing. A good chemical resistance is declared with acids and bases at the
 194 concentrations of interest in this study.

195 A second experimental campaign involved other two adhesives, which will be therefore called
 196 Adhesive 4 and 5, respectively, in the following. In these cases, the tests were conducted only
 197 for the 2B tumbled surface finish (see description of surface finishes in the following).

198 Adhesive 4: 3M Scotch-Weld™ 7240 FR B/A, a high performance, two-component, toughened
 199 epoxy adhesive that exhibits a good strength in contact with oil derivatives and salt fog.

200 Adhesive 5: Elantas Elan-tech® AS 50/AW 50, a two-component epoxy system loaded with
 201 non-abrasive fillers with chemical resistance to engine oil, petrol, acids and bases.

202 A third experimental campaign involved Adhesive 4 for which, unlike Adhesives 1-3 and 5, a
 203 specific primer was available (3M™ Surface Pre-Treatment AC-130) and it was applied after
 204 cleaning. In this way, the good resistance to chemical aging of the adhesive itself was combined
 205 with the improvement of the adhesion and chemical resistance of the interface provided by the
 206 primer. The basic mechanical properties of Adhesives 1-5 are listed in Table 3.

207

208 **Table 3 – Adhesives basic mechanical properties from suppliers datasheets (in MPa).**

Property	Adhesive 1	Adhesive 2	Adhesive 3	Adhesive 4	Adhesive 5
Lap shear strength*	23	12	2	24	17
Tensile strength**	32	31	3	N/A	50***
Young's modulus	1718	6700	3	3750	4000

209 *on stainless steel; **bulk adhesive; ***flexural strength

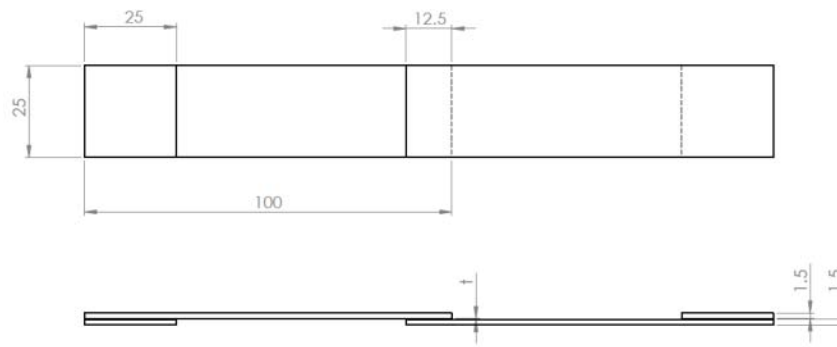
210

211 2.3 Specimen and test procedure

212 Since the geometry of the joints hypothesized in Sect. 2.1 for bonding of the inner casing is a
 213 butt-strap type, a single-lap joint specimen defined by the standard [15] and represented in
 214 Figure 6, is a reasonable choice to test the strength. Samples with an adherend thickness of 1.5

215 mm were used and tabs of the same thickness and material were applied to the ends of the
216 adherends to keep the alignment. The material is AISI 304 L (X2 CrNi 18-9). The thickness of
217 the adhesive layer 0.25 ± 0.01 mm, controlled by adding stainless steel spheres with a diameter
218 of 0.24-0.26 mm. In the case of Adhesive 3, a thickness of 1.5 mm was set, as typical for highly
219 elastic adhesives. At the same time, thicker tabs were also used to ensure the alignment of the
220 specimen.

221



222

223

Figure 6 - single-lap joint specimen used in the experiments.

224

225 Five specimens were tested for each condition examined as indicated in [15]. The specimens
226 were bonded in a PolyEthilene (PE) template to ensure the alignment of the adherends and the
227 length of the overlap. The tests were performed using an Instron 4467 electromechanical
228 machine with an acquisition rate of 10 Hz. As prescribed in [15], they were performed under
229 displacement control until the sample was broken. The displacement speeds used are 1.3
230 mm/min for joints with stiff epoxies and 10 mm/min for joints with flexible MS adhesive so
231 that, according to [15], the test lasts no longer than 65 ± 20 s in any case. The Chauvenet
232 criterion was applied to the results to identify any outliers with respect to a normal distribution
233 assumed for each batch of specimens.

234

235 2.4 Surface finish

236 The surface finish of the stainless steel sheets commonly used in food machinery are indicated
237 in Table 4 with the nomenclature according to the European standard EN 10088-2 and the
238 related value of roughness R_a ; the sandblasting treatment is also indicated for comparison. A
239 surface with a low roughness clearly limits adhesion, however mechanical, physical or chemical
240 surface treatments are hardly applicable in this case because of hygienic and safety risks
241 management at the manufacturing line, so that one can only resort to cleaning and degreasing
242 and, possibly, apply a specific primer or coupling agent.

243
244

Table 4 – Average values of roughness R_a of the adherends.

Surface finish type	R_a [μm]
2B bright (2R)	0.13
2B Scotch-Brite (2J)	0.29
2B (tumbled)	1.07
Micro shot-peened	1.54
Sandblasted	3.56

245

246 Comparative tests were then performed to evaluate the effect of these types of surface finish on
247 the strength of the joint and compared to a conventional sandblasting treatment. The MS
248 adhesive was not tested with the 2B bright finish or with the sandblasted one since for the other
249 three surfaces, tested initially, the failure was always cohesive meaning a substantial
250 insensitivity to roughness.

251

252 2.5 COP-SOP aging simulation

253 The cleaning cycle of the machine surfaces (Cleaning-Out-of-Place, COP) involves a first
254 washing with an alkaline, sodium hydroxide (NaOH) foaming solution and a subsequent
255 sanitization (Sanitization-Out-of-Place, SOP) with a PerAcetic Acid (PAA) solution. Rinsing
256 with process water is performed before and after each phase. The cycle is commonly between
257 daily and weekly and it is supplemented monthly and after each maintenance operation by
258 washing with an acid-based solution (HNO_3 or H_3PO_4) with a descaling function. This process
259 takes place at temperatures ranging from ambient up to 40 °C. Table 5 shows an indicative
260 recipe of the daily external washing cycle (COP and SOP).

261

262

Table 5 – Typical cleaning cycle for external surfaces (COP-SOP).

263

Step	Agent	Temperature (°C)	Duration (min)
Rinsing	Water	Ambient	10
Cleaning	Foaming with 2% NaOH	Max 40	10
Rinsing	Water	Ambient	10
Sanification	Foaming with 2% PAA	Max 40	10
Rinsing	Water	Ambient	10

264

265 Considering an average machine life of 20 years as a reference target and summing the minutes
266 of contact with the solutions, a total contact time of approx. 50 days (1200 h) is found.

267 The aging caused by contact with COP-SOP agents was simulated by immersing the joints in
268 solutions indicated in Table 6. To maintain the temperature at 40 °C, two thermostatic baths
269 were used in which containers with the solutions were immersed in hot water. The solutions

270 were renewed every 7 days, in order to keep them active, in particular this is important for the
 271 product VT70-Diverfoam which contains PAA which is known to degradate rapidly.

272
 273

Table 6 – COP-SOP chemicals (supplier: Diversey).

SOL.	Agent	Conc. [% vol.]	Active principle	Conc. of active principle in the agent (% vol.)	T [°C]
1	VE3 EnduroSuper	2	KOH EDTA Propanol	3-10 % 3-10 % 3-10 %	23-40
2	VE9 EnduroEco	2	H ₃ PO ₄ Propanol	30-50 % 3-10 %	23-40
3	VT70 Diverfoam Active	2	CH ₃ COOOH CH ₃ COOH H ₂ O ₂	1-3 % 3-10 % 10-20 %	23-40

274

275 For the conditioning temperature, a weekly cycle of 5 days at 40 °C, the maximum temperature
 276 recommended for external washing, was applied followed by 2 days at room temperature
 277 (corresponding to the thermostatic bath being switched off during the weekend for safety
 278 reasons). The total immersion time was set at 50 days (1200 h), corresponding to 7 cycles plus
 279 one day, that corresponds to the sum of the minutes of contact with the PAA sanitizing solution,
 280 that being an oxidant should be the most aggressive for adhesives. These conditions do not
 281 exactly repeat those present in the COP-SOP, as there is no continuous immersion but periodic
 282 daily contact followed by rinsing and drying. Keeping the specimens immersed for a period of
 283 several consecutive weeks will however favor the diffusion in the polymer up to the interface
 284 with the metal, affecting but cohesion and adhesion strength.

285 At least two groups of 5 specimens, made out of tumbled 2B surface finish sheet metal plates,
 286 were immersed and at least one group was picked up for testing before the end of the period in
 287 order to evaluate a possible damage trend. The values of strength obtained for the unaged joints
 288 were used as a reference of strength. The solution with VE3 (alkaline solution) was not used
 289 with the Adhesive 2, since the technical datasheet already indicated a strength of 115% with
 290 respect to the unaged one after 3000 h of immersion in a 4% NaOH solution.

291

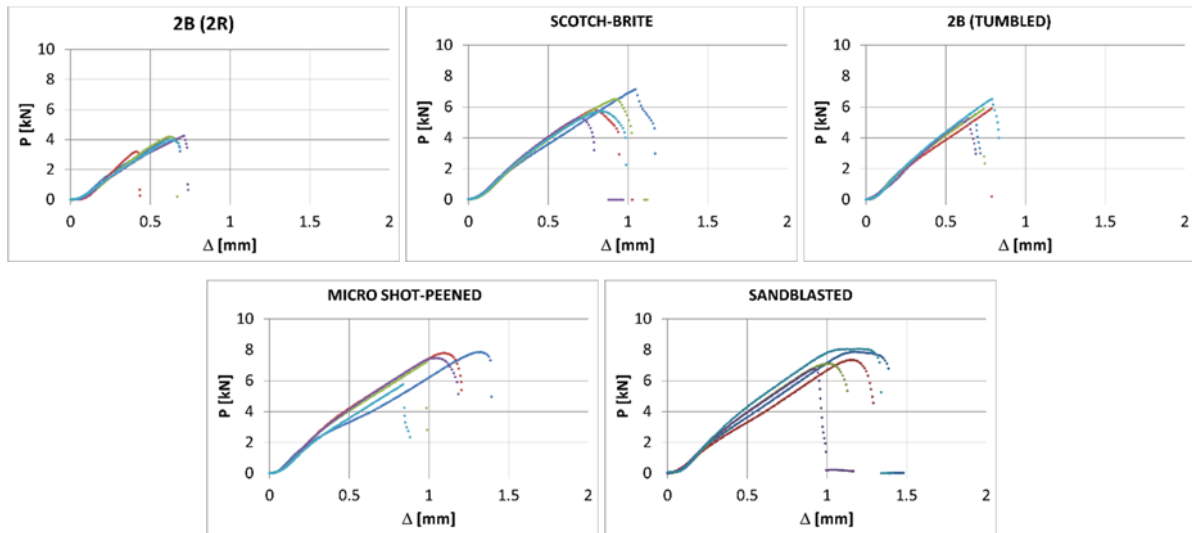
292 **3. Results and Discussion**

293 3.1 Adhesives 1-3: unaged strength and effect of the surface finish

294 Regarding adhesive 1, the force (P) vs. displacement (Δ) plots are shown in Figure 7 for the
 295 various roughnesses tested and the average shear strength is summarized in Table 7 along with
 296 standard deviation.

297

298



299

300

Figure 7 - Force vs. displacement plots of unaged Adhesive 1 at different surface finishes.

301

302 The strength of micro-shot-peened substrate is comparable to that obtained with sandblasting,
 303 so this surface is very favorable for bonding with Adhesive 1, probably due to its light but very
 304 uniform roughness. It can be noticed in both these cases that failure occurs in most of the
 305 specimens after reaching a force plateau, meaning that plastic yielding of the adherends is
 306 attained and the force cannot increase any longer.

307

Table 7 – Average shear strength τ and standard deviation $s(\tau)$ of Adhesive 1 as a function of surface finish.

309

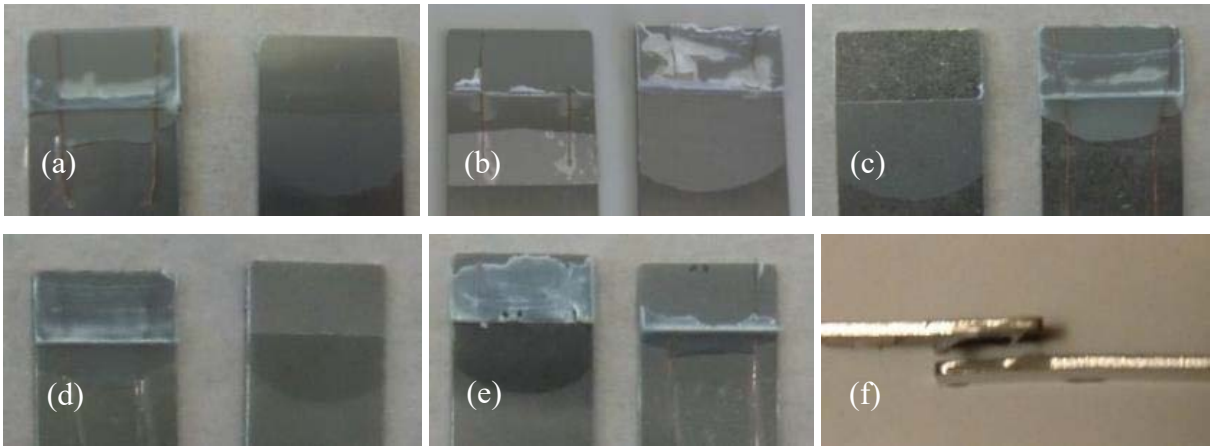
Surface finish type	Ra [μm]	τ [MPa]	$s(\tau)$ [MPa]
2B - (2R)	0.13	13.3	0.38
SCOTCH-BRITE	0.29	19.6	2.30
2B (tumbled)	1.07	18.6	1.51
Micro shot-peened	1.54	24.3	0.97
Sandblasted	3.56	23.8	1.71

310

311 Overall, even for fairly smooth surfaces (2B and above all Scotch-Brite) good strength values
 312 are obtained, while a consistently lower value is recorded on the less rough surface, as expected.
 313 Figure 8 shows the failure surfaces which are always totally or mainly adhesive even in the case
 314 of sandblasting. In this case, however, as in that of the micro-blasted surface, the value reached
 315 (24-24.5 MPa) seems to be the upper limit due to yielding of the AISI 304 sheet metal (Figure
 316 8f) that increases the shear deformation of the adhesive at the ends of the joint and causes in
 317 turn the failure to start from the most deformed interface.

318

319



320

321

322

323

Figure 8 – Failure surfaces of the specimens made with Adhesive 1 and 2B-2R (a), Scotch-Brite (b), 2B tumbled (c), Shot-peened (d) and Sandblasted (e) surface finish. The sandblasted and micro-shot-peened specimens are plastically deformed near the ends of the overlap (f).

324

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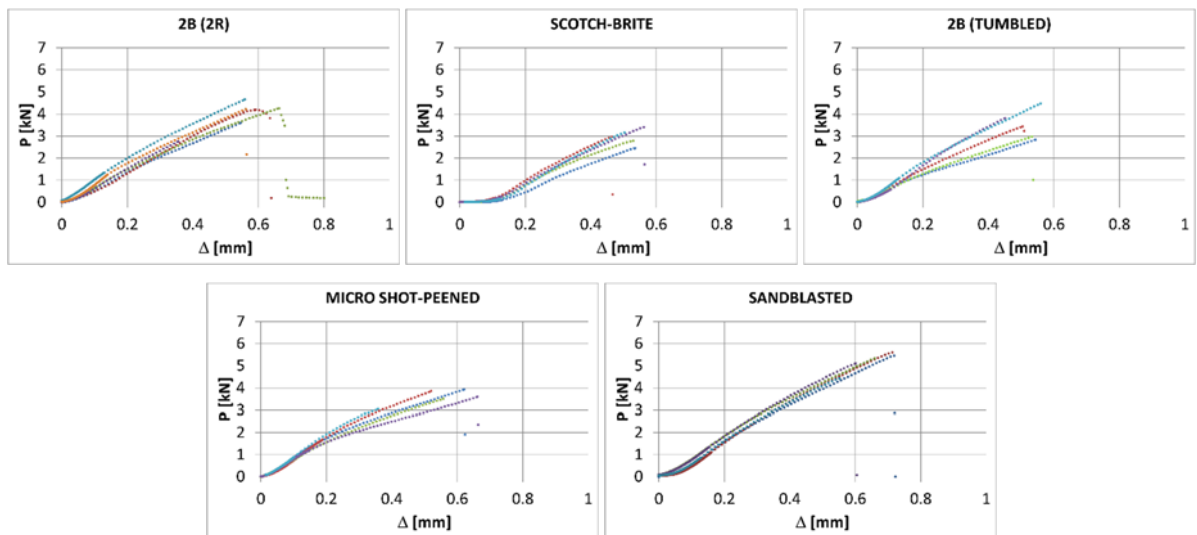
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331

332

In the case of Adhesive 2, the force vs. displacement data are shown in Figure 9. The force peaks, hence the shear strength values (Table 8) are lower than those of Adhesive 1 as could be expected from the data declared by the supplier in the respective technical data sheets, albeit referring to metal alloys different from AISI 304. It is interesting to notice that, different from Adhesive 1, the increase in performance is not uniquely related to the surface roughness, but quite overlapping values are found for all the surface treatments but sandblasting, that yields the higher joint strength.

333



334

335

Figure 9 - Force vs. displacement plots of unaged Adhesive 2 at different surface finishes.

336

337

338

Table 8 – Average shear strength τ and standard deviation $s(\tau)$ of Adhesive 2 as a function of surface finish.

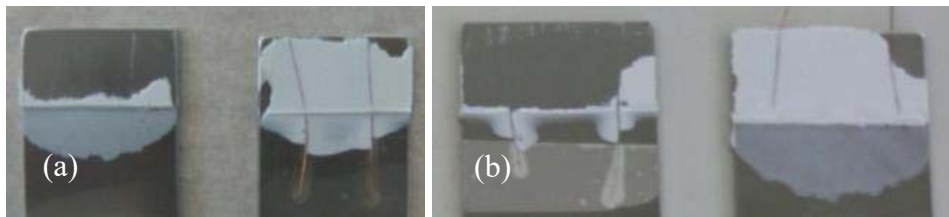
Surface finish type	Ra [μm]	τ [MPa]	$s(\tau)$ [MPa]
---------------------	----------------------	--------------	-----------------

2B - (2R)	0.13	13.4	1.20
SCOTCH-BRITE	0.29	9.5	1.17
2B (tumbled)	1.07	11.4	2.04
Micro shot-peened	1.54	11.9	0.64
Sandblasted	3.56	16.6	1.47

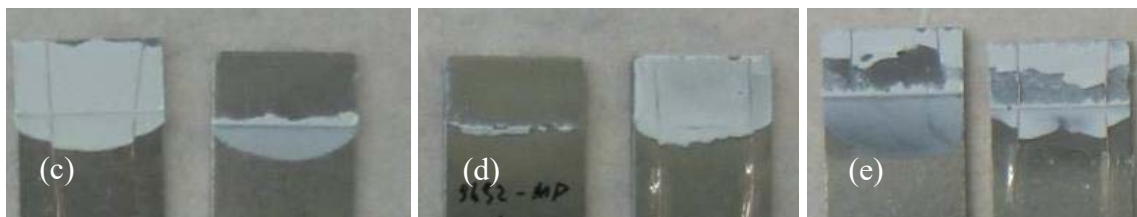
339

340 The failures are adhesive also in this case (Figure 10) with the only exception of sandblasting,
 341 in which the visual inspection detects a thin layer of adhesive on the surface in one of the two
 342 adhesions, so that the breakage occurs near the interface, but still cohesive.

343



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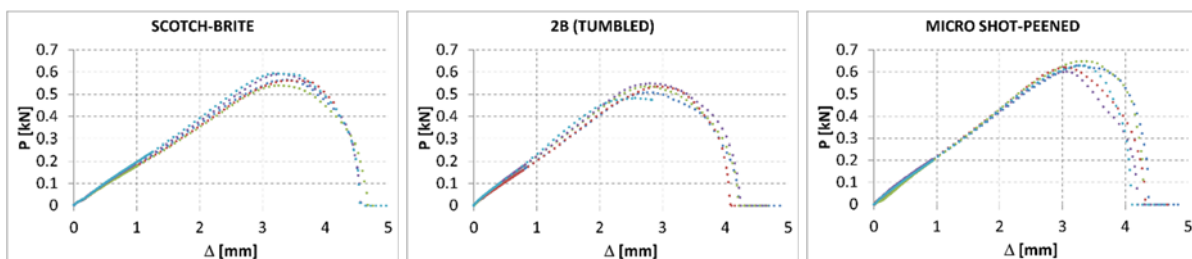
345

346 **Figure 10 – Failure surfaces of the specimens made with Adhesive 2 and 2B-2R (a), Scotch-Brite (b), 2B**
 347 **tumbled (c), Shot-peened (d) and Sandblasted (e) surface finish.**

348

349 Adhesive 3, being elastic, also has a much lower resistance in absolute terms than the others,
 350 but it has the advantage of being almost insensitive to roughness (see Figure 11 and Table 9)
 351 and always presents cohesive cracks (Figure 12), that is, it has a good adhesion to the surface
 352 relative to the performance provided.

353



354

355 **Figure 11 - Force vs. displacement plots of unaged Adhesive 3 at different surface finishes.**

356

357 The cohesive failure is reflected also by the ratio $s(\tau)/\tau$ lower than the other two adhesives that
 358 exhibited the interfacial failure, as this latter yields generally a higher scatter of the strength.

359
360
361

Table 9 – Average shear strength τ and standard deviation $s(\tau)$ of Adhesive 3 as a function of surface finish.

Surface finish type	Ra [μm]	τ [MPa]	$s(\tau)$ [MPa]
SCOTCH-BRITE	0.29	1.83	0.07
2B (tumbled)	1.07	1.67	0.08
Micro shot-peened	1.54	2.01	0.05

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Figure 12 – Failure surfaces of the specimens made with Adhesive 3 and Scotch-Brite (b), 2B tumbled (c) and Shot-peened (d) surface finish.

366

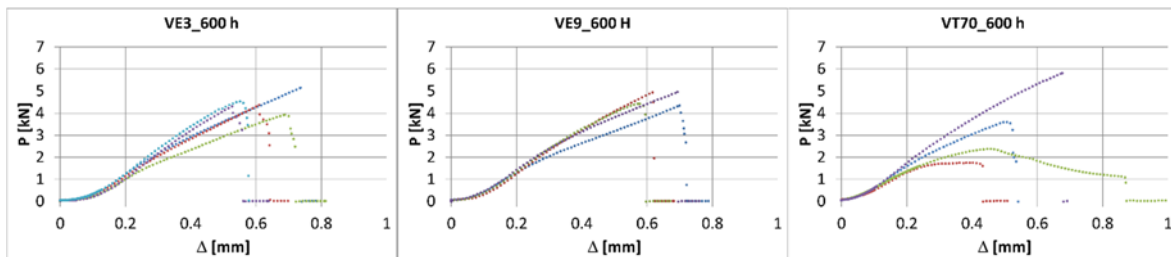
367 From the whole of the tests on the three adhesives it can also be said that the 2B (tumbled)
368 surface finish selected for the subsequent immersion tests in the COP-SOP solutions, which
369 represents also the most commonly used one, provide a value of strength intermediate among
370 those examined.

371

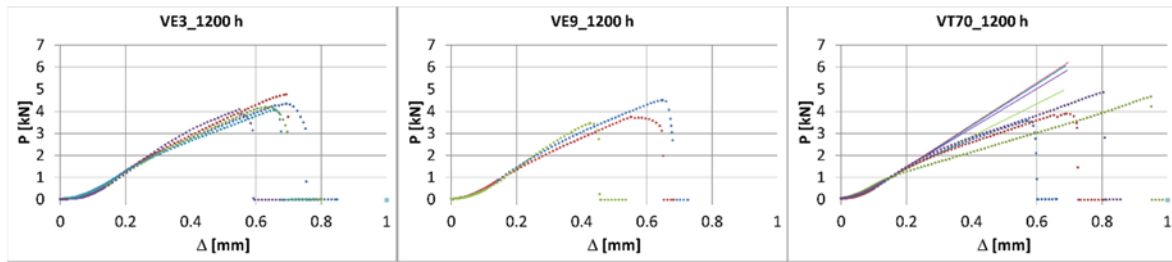
372 3.2 Adhesives 1-3: resistance to COP-SOP agents

373 With Adhesive 1, spontaneous debondings of some joints occurred in the first part of the
374 immersion period (at 14 and 25 days) in VE9, therefore the processes leading to debonding are
375 rather rapid during during the immersion period. The results obtained from the tests carried out
376 on the residual samples are reported in Figure 13, bearing in mind that ten samples were
377 immersed for each type of joint.

378



379



380

381 **Figure 13 - Force vs. displacement plots of Adhesive 1 aged in different chemicals and at increasing times.**

382

383 Table 10 summarizes the residual shear strength values and the related standard deviation, along
 384 with the percent residual strength with respect to the unaged samples. It can be observed that
 385 the decrease in resistance in VE9 solution is approximately linear and that that due to VE3
 386 varies little in the second half of the conditioning time. Although the residual strength is quite
 387 high both in absolute and relatively to the unaged condition and it would sufficient for the
 388 application under examination, the development of spontaneous failures in VE9 can make this
 389 adhesive critical in service.

390

391 **Table 10 – Average shear strength of Adhesive 1 with respect to aging time. The % indicates the residual**
 392 **strength with respect to the unaged joint.**

Aging time	600 h		1200 h	
Solution	τ [MPa]	$s(\tau)$ [MPa]	τ [MPa]	$s(\tau)$ [MPa]
VE3	14.7 (76.7 %)	1.426	13.4 (71.9 %)	0.390
VE9	15.0 (80.3 %)	1.045*	12.5 (67.3 %)	1.726*
VT70	12.0 (64.3 %)	2.41	10.6 (56.8 %)	2.690

393

*As three joints failed spontaneously before 600 h, of the remaining seven, three were tested
 394 after 600 h and four after 1200 h.

395

396 By evaluating the stiffness of the joint as the slope of the load-displacement values close to the
 397 origin of data it can be seen that this too undergoes a reduction due to aging, that is a symptom
 398 of a partial plasticization effect (Table 11). The reduction is relatively small, and it is due not
 399 only to modifications of the polymer, but probably also to effects on the edges of the bondline,
 400 where in many cases an adhesive debonding has begun during immersion.

401

402 **Table 11 – Reduction of joint stiffness with Adhesive 1 after immersion in the various solutions.**

Aging time	Unaged	600 h	1200 h
Solution	Stiffness [kN/mm]	Residual stiffness (%)	Residual stiffness (%)
VE3	10.507	86.6	87.8
VE9	10.507	93.5	90.3

VT70	10.507	77.7	88.3
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In the specimens tested after conditioning in VE3 there were no visible effects (with the exception of air bubbles under the excess adhesive film), and the adhesive failures are similar to those of the unaged one, Figure 14a-b. In the specimens conditioned in VE9 there was a slight "wrinkling" of the adhesive in excess at the end of the joint, Figure 14c-d.

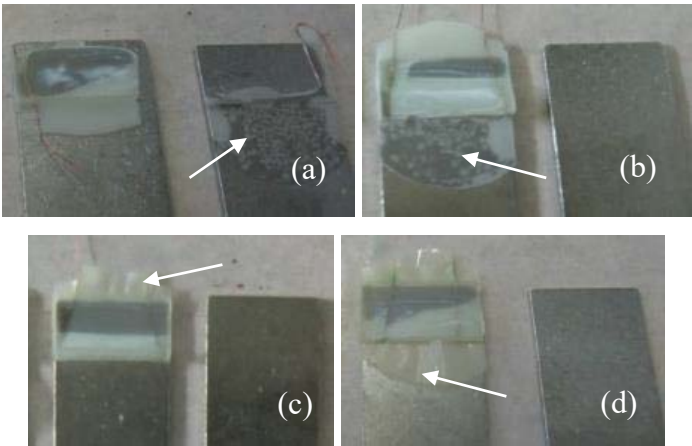


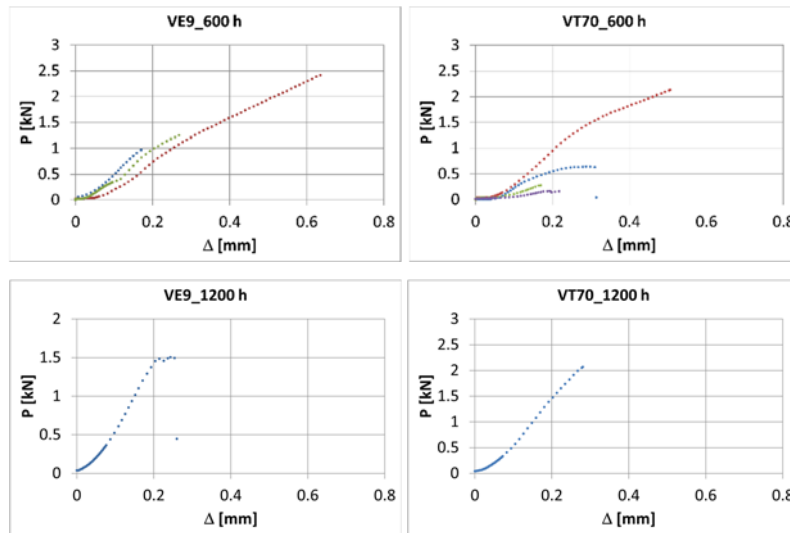
Figure 14 – Failure surface of Adhesive 1 aged in VE3 tested after 25 and 50 days (a and b, respectively) and in VE9 after 25 and 50 days (c and d, respectively). The arrows indicate the wrinkled excess adhesive.

In the samples conditioned in VT70, reddish-brown deposits were also found, that leads to suppose the presence of contaminating particles of dirt bound to the stainless steel microspheres used to keep the adhesive layer thickness, which have not in fact been cleaned before bonding. In the other samples with microspheres, however, there are no signs of corrosion and the failure is anyway adhesive, without spontaneous debondings during immersion.

Together with the described phenomenon, there is also a light brown patina on the adherend near the joint, with a consistency similar to powder, typical of a liquid solution with PAA that stagnates and then dries on a metal surface. It is not a sign of corrosion, but it is a phenomenon that is found also in machines, and which in this case is due to the liquid that remained under the excess adhesive at the ends of the joint. It is also noted that in almost all the samples the excess adhesive in the vicinity of the fillets has lifted away from the surface, which may have favored the aggression of the bondline.

With Adhesive 2, tested only with VE9 and VT70, a high rate of spontaneous debondings was detected, which occurred in even shorter times than those occurred in the samples with Adhesive 1, so much that at the end of the conditioning period just one sample for each solution was still integer, as it can be seen from Figure 15.

431



432

433 **Figure 15 - Force vs. displacement plots of Adhesive 2 aged in different chemicals and at increasing times.**

434

435 The results are summarized in Table 12. The residual strength is lower than Adhesive 1 and the
 436 number of spontaneous debondings before the end of immersion is even higher, making the
 437 data of Table 12 purely indicative: if the strongest specimen at 600 h was treated as an outlier,
 438 the residual strength would have been much lower and, treating as an outlier for coherence the
 439 only specimen that lasted 1200 h, it could be said that there was no residual strength after the
 440 maximum aging time.

441

442 **Table 12 – Average shear strength of Adhesive 2 with respect to aging time. The% indicates the residual**
 443 **strength with respect to the unaged joint.**

Aging time	600 h		1200 h	
	τ [MPa]	$s(\tau)$ [MPa]	τ [MPa]	$s(\tau)$ [MPa]
VE9	5.34 (47.0 %)*	*	4.82 (42.4 %)	**
VT70	6.83 (60.0 %)*	*	6.62 (58.3 %)	**

444

*Only the strongest specimen was reported for comparison with the one survived at 1200 h.

445

**it can not be calculated since only one specimen was left after spontaneous failures during immersion.

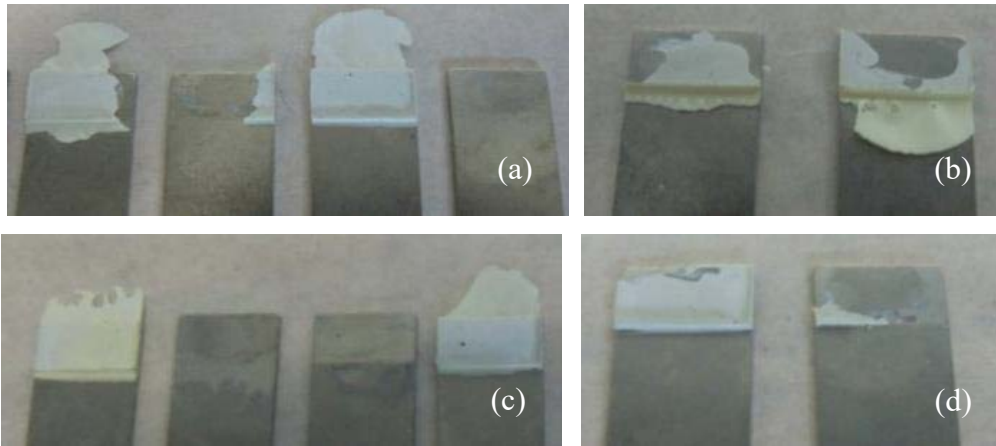
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448 The only difference found with respect to the unaged joint, were the yellowed bands in
 449 correspondence with where the excess adhesive on the outside of the joint was raised and
 450 probably provided a preferential attack path for the solutions (Figure 16).

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454

455 **Figure 16 – Joints made with Adhesive 2 immersed in VE9 and tested at the intermediate interval (a) and**
456 **at the end of aging (b); immersed in VT70 and tested at the intermediate interval (c) and at the end of**
457 **conditioning (d).**

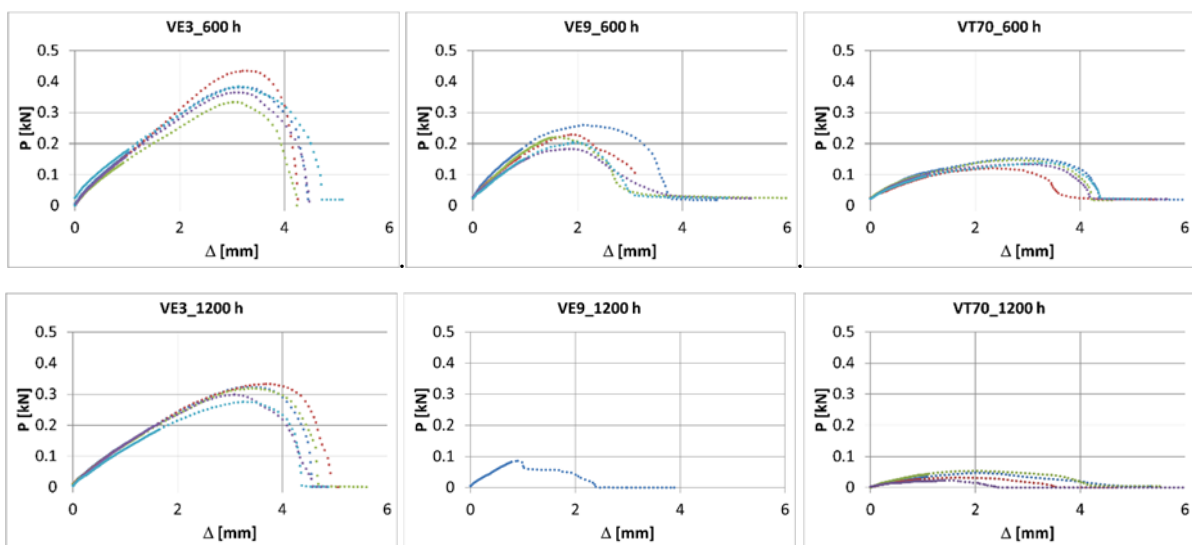
458

459 Concerning this adhesive, the main outcome is the poor adhesion after aging. The spontaneous
460 failures indicate that the combination of stainless steel surface without specific preparation
461 treatment does not allow a durable joint in aggressive environmental conditions with this
462 adhesive.

463 The force-displacement results of Adhesive 3 are presented in Figure 17. Spontaneous failures
464 under immersion occurred also for Adhesive 3, particularly in acid solutions. However, they
465 turned out to be much more gradual, i.e. the degradation and the beginning of debonding was
466 detectable, as also indicated by the samples immersed in VT70 which at the end of conditioning
467 appeared only partially debonded.

468

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470

471 **Figure 17 - Force vs. displacement plots of Adhesive 3 aged in different chemicals and at increasing times.**

472

473 However, it should be noted that in general the samples showed also a visible swelling and
 474 deformation of the adhesive. As can be seen from Table 13 and Table 14, this seriously
 475 compromised both the strength and stiffness of the joint. Furthermore, the failures have turned
 476 from cohesive in the unaged joint to adhesive after aging, therefore Adhesive 3 seems to be
 477 particularly sensitive to the chemical agents used in COP-SOP and therefore not very suitable
 478 for the application.

479

480 **Table 13 – Average shear strength of Adhesive 3 with respect to aging time. The% indicates the residual**
 481 **strength with respect to the unaged joint.**

Aging time	600 h		1200 h	
Solution	τ [MPa]	$s(\tau)$ [MPa]	τ [MPa]	$s(\tau)$ [MPa]
VE3	1.28 (76.6 %)	0.074	0.99 (59.3 %)	0.074
VE9	0.70 (42.0 %)	0.091	0.28 (16.5 %)	*
VT70	0.45 (26.8 %)	0.045	0.13 (7.60 %)	0.045

482 *it can not be calculated since only one specimen was left after spontaneous failures during
 483 immersion.

484

485 **Table 14 – Reduction of joint stiffness with Adhesive 3 after immersion in the various solutions.**

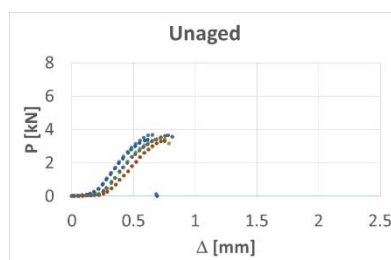
Aging time	Unaged	600 h	1200 h
Solution	Stiffness [kN/mm]	Residual stiffness (%)	Residual stiffness (%)
VE3	0.216	82.12	58.93
VE9	0.216	71.33	35.58
VT70	0.216	30.66	-

486

487 3.3 Adhesive 4-5: unaged strength and resistance to COP-SOP agents

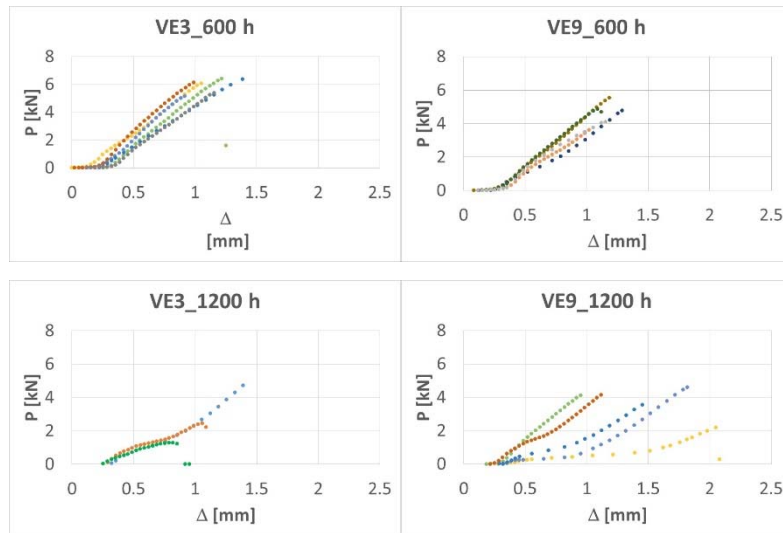
488 Since none of the first three adhesives tested provided both a sufficient residual strength and
 489 full integrity after immersion in chemical agents, a second campaign was carried out on
 490 Adhesive 4-5 under unaged condition and after immersion in VE3 (alkaline) and VE9 (acid)
 491 solutions. The force-displacement plots of Adhesive 4 are reported in Figure 18 at different
 492 aging times. It is immediate to notice that some tests are missing at 1200 h because there were
 493 spontaneous debondings also in this case.

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Figure 18 - Force vs. displacement plots of Adhesive 4 unaged and aged in different chemicals and at increasing times

500 Looking at the values of average shear strength in Table 15, a net increase is visible after the
 501 first 600 h of immersion, that may be the beneficial result of a post-curing of the adhesive that
 502 overrides aging. After 1200 h instead, the strength gets lower or similar to the unaged one, as a
 503 result of chemical aggression. Most important 3 over 6 specimen failed spontaneously in VE3
 504 and 1/6 in VE9, and those that resisted show a quite large scatter of the results, therefore there
 505 is a long-term reliability problem also for Adhesive 4 under these aging conditions.

506

507 **Table 15 - Average shear strength of Adhesive 4 with respect to aging time. The% indicates the residual**
 508 **strength with respect to the unaged joint.**

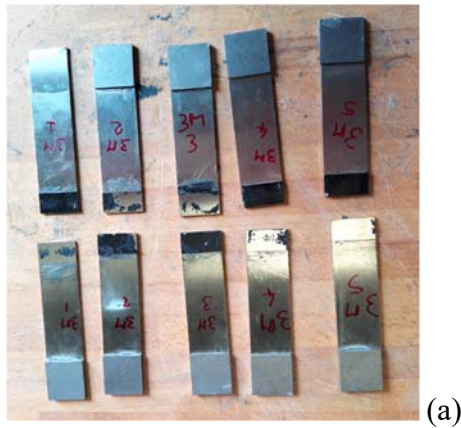
Aging time	600 h		1200 h	
Solution	τ [MPa]	$s(\tau)$ [MPa]	τ [MPa]	$s(\tau)$ [MPa]
VE3	18.7 (169%)	1.67	8.98 (81.4%)	5.67
VE9	14.2 (129%)	2.15	11.7 (106%)	2.97

509

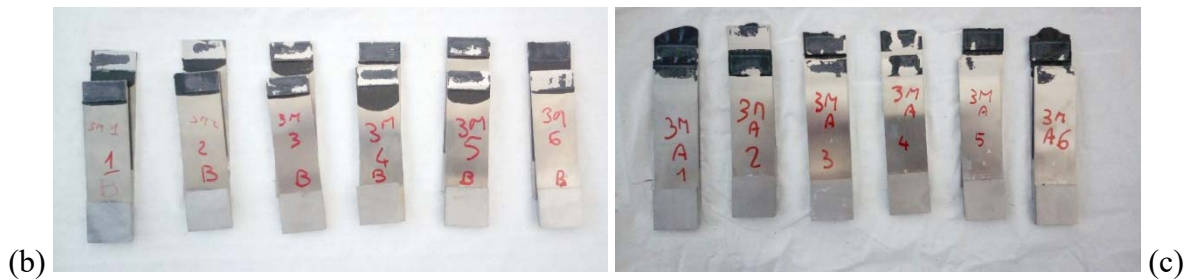
510 Figure 19 reports the failure surfaces of Adhesive 4 at different aging stages in VE3 and VE9.
 511 The rupture is mixed cohesive-adhesive, especially after 600 h in VE3, or adhesive alike
 512 Adhesive 1-3. However, mixed or adhesive failure was already present in the unaged joints due
 513 to the low surface roughness and the absence of surface pretreatment but degreasing, therefore
 514 aging does not change fundamentally the failure mechanism.

515

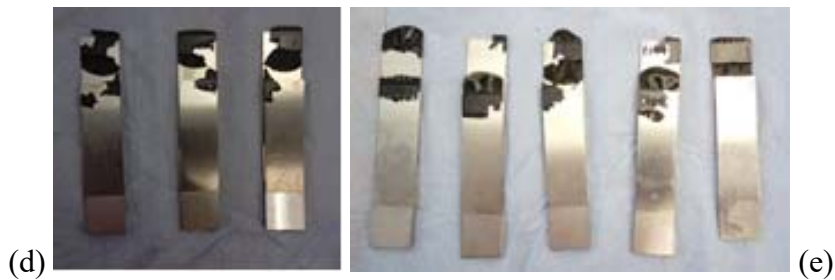
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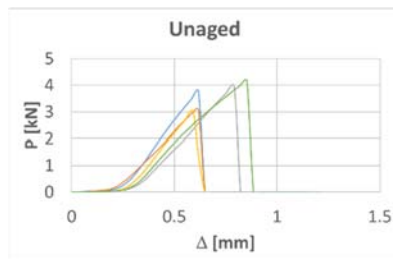


519 **Figure 19 - Failure surface of Adhesive 4 unaged (a), aged in VE3 tested after 25 and 50 days (b and c,**
 520 **respectively) and in VE9 after 25 and 50 days (d and e, respectively). In b-e the adherends are overlapped**
 521 **each other so that the failure surfaces are shown one over the other.**

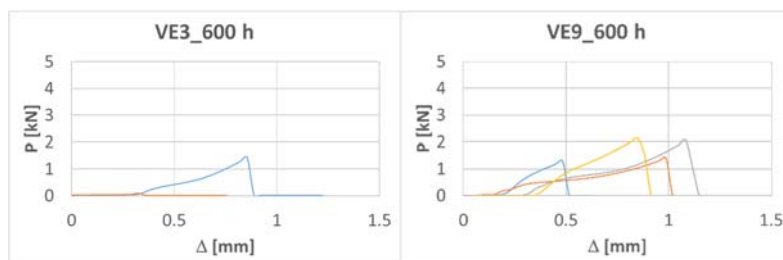
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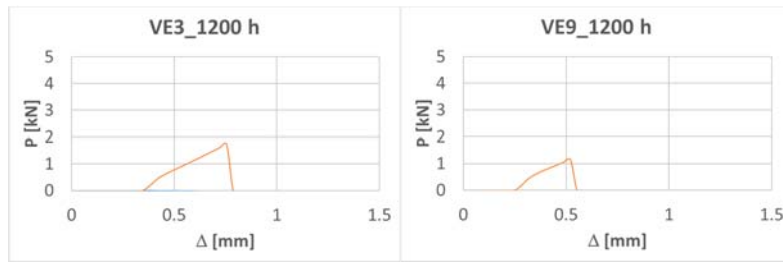
523 From the force-displacement plots of Adhesive 5 reported in Figure 20, it is immediately visible
 524 that several tests are missing after aging, because of spontaneous failures during immersion.
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529 **Figure 20 - Force vs. displacement plots of Adhesive 5 unaged and aged in different chemicals and at**
530 **increasing times**

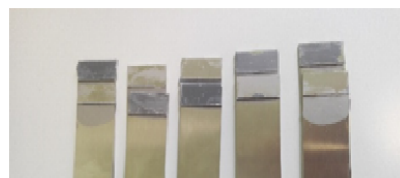
531
532 As also testified by the values of average shear strength and the related standard deviation in
533 Table 16, Adhesive 5 seems to be particularly affected by the chemicals used in COP-SOP,
534 thereby little suitable for the application.

535
536 **Table 16 - Average shear strength of Adhesive 5 with respect to aging time. The% indicates the residual**
537 **strength with respect to the unaged joint**

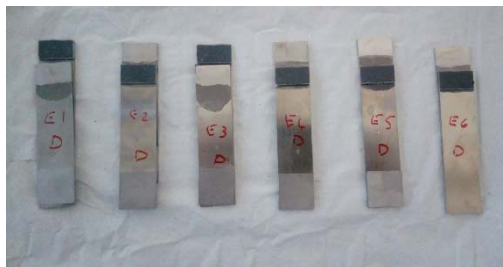
Aging time	600 h		1200 h	
Solution	τ [MPa]	$s(\tau)$ [MPa]	τ [MPa]	$s(\tau)$ [MPa]
VE3	4.494	-*	5.528	-*
VE9	5.416	1.381	3.694	-*

538 *it can not be calculated since only one specimen was left after spontaneous failures during
539 immersion

540
541 The failure surfaces (Figure 21) exhibit always interfacial debonding while the unaged failure
542 was mixed, therefore in this case there is an evident deterioration with respect to the initial
543 conditions.



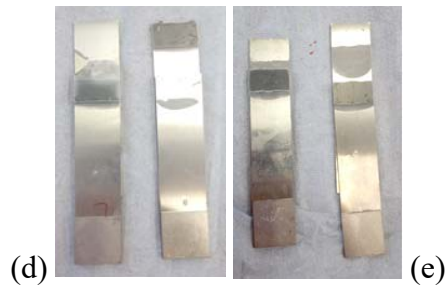
544 (a)



545 (b)



(c)



546

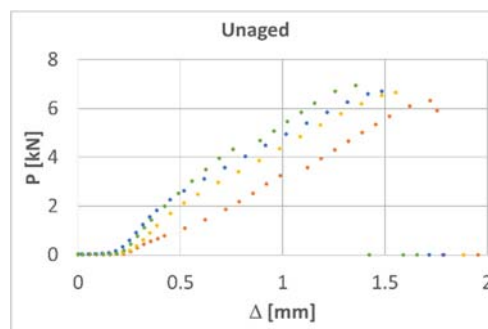
547 **Figure 21 - Failure surface of Adhesive 5 unaged (a), aged in VE3 tested after 25 and 50 days (b and c,**
 548 **respectively) and in VE9 after 25 and 50 days (d and e, respectively). The adherends are overlapped each**
 549 **other so that the failure surfaces are shown one over the other.**

550

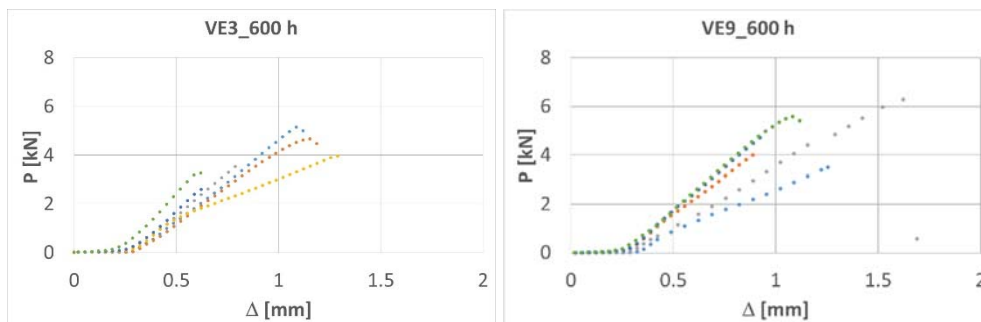
551 3.4 Adhesive 4 with chemical surface pretreatment: unaged strength and resistance to COP-
 552 SOP agents

553 Since also Adhesive 4-5 alone failed to provide a complete reliability in terms of both residual
 554 strength and integrity after aging a third campaign was done on Adhesive 4 plus primer under
 555 unaged condition and after immersion in VE3 (alkaline) and VE9 (acid) solutions; alike the
 556 second experimental campaign, tests were done after 600-800-1000-1200 h of immersion. The
 557 force vs. displacement diagrams are collected in Figure 22 while Figure 23 and Figure 24 show
 558 the average values of the shear stress at failure and the relative standard deviation for the
 559 solutions VE3 and VE9 respectively.

560

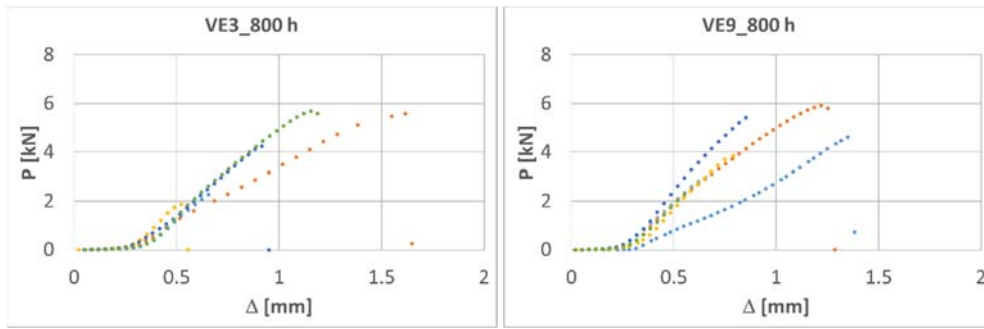


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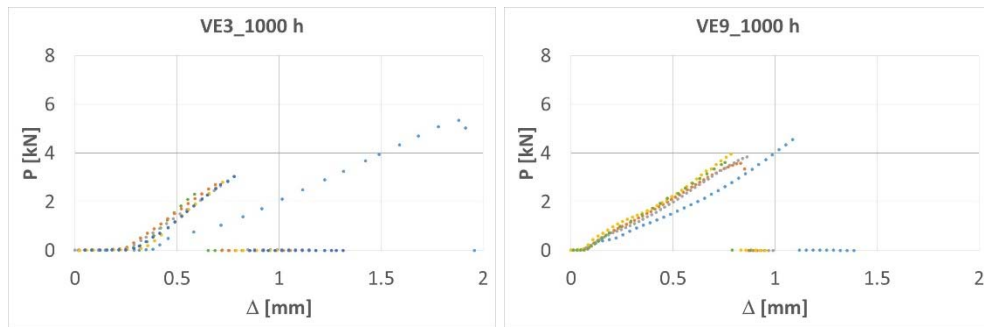


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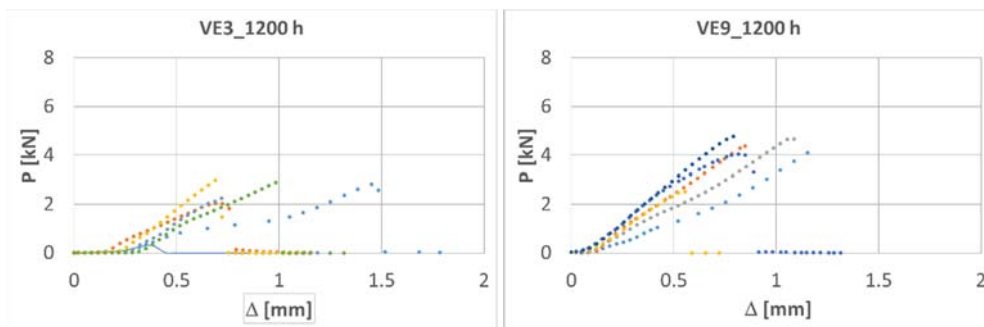
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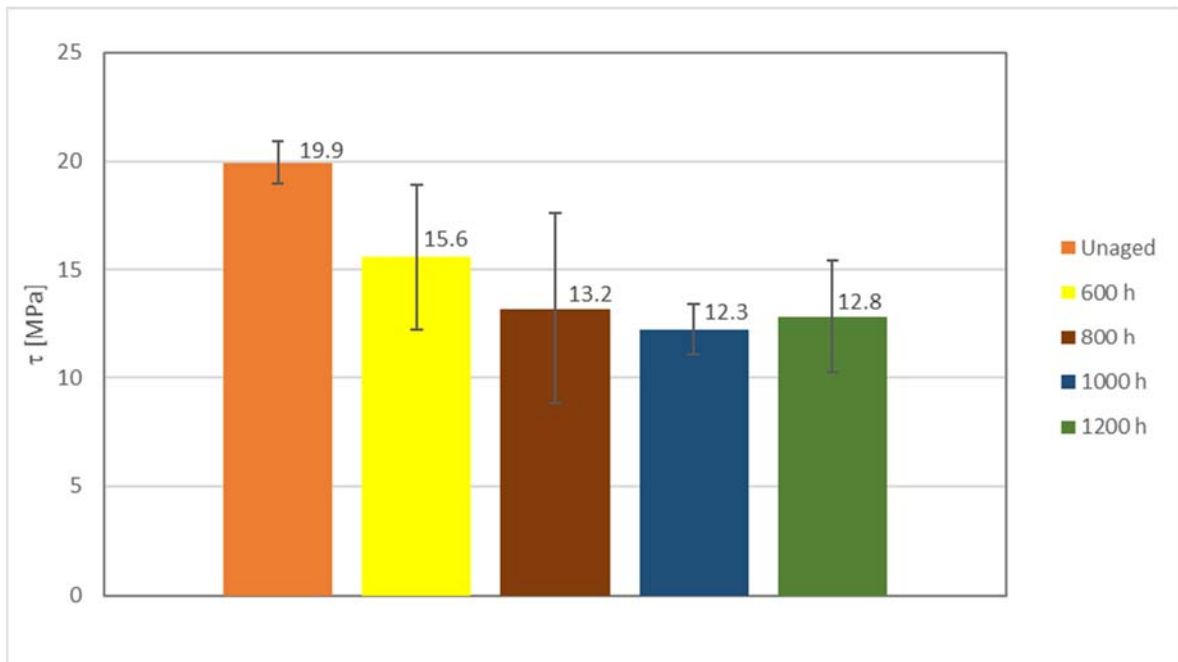
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Figure 22 - Force vs. displacement plots of Adhesive 4 unaged and aged in different chemicals and at increasing times

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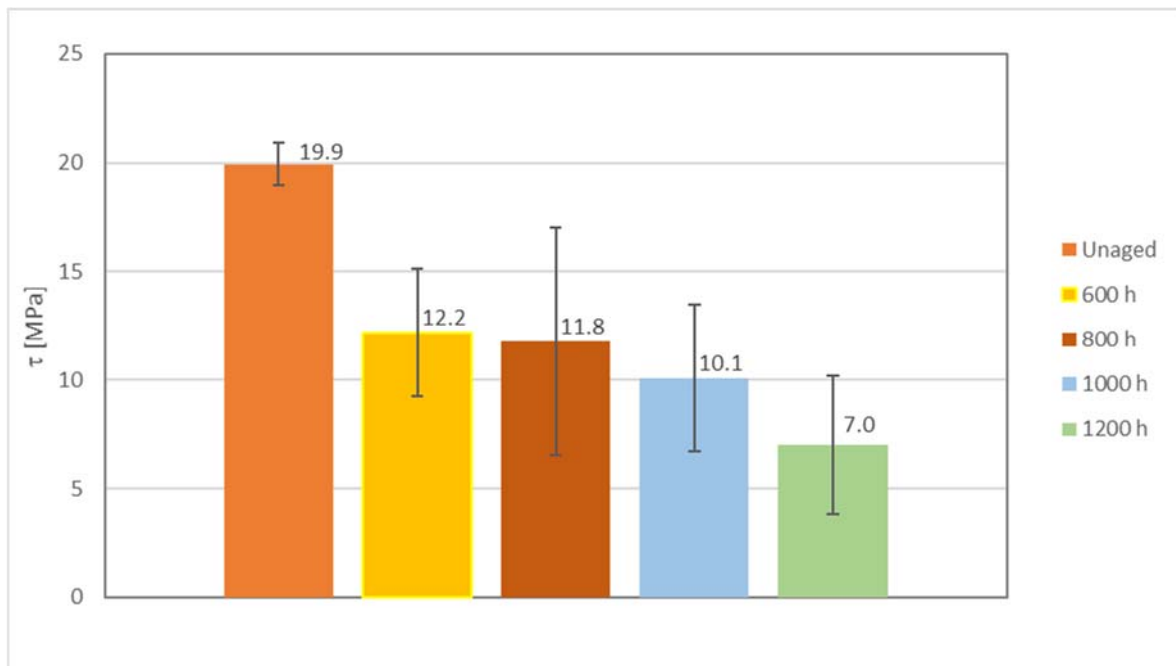


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Figure 23 – Average shear strength of Adhesive 4 + primer with respect to aging time in VE3.

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It can be noticed that in the alkaline solution (VE3) the strength undergoes a degradation, stabilizing around 65% of the unaged value starting from 800 hours of immersion. In the acid solution (VE9), on the other hand, it tends to progress, reaching a residual strength equal to 35% of the non-aged at the end of the test. Even though these values are comparable or even lower than Adhesive 1, the presence of a primer protected the stainless steel adhesive interface improving long-term adhesion such that a sufficient strength is preserved over time and no spontaneous failures were recorded. For this reason, Adhesive 4 can be preferred for the application in beverage filling machines. A feature of Adhesive 4 common also to the other adhesives tested is a relatively high coefficient of variation (std. dev./average); this is related to the low surface roughness, as found also in unaged joints, and also to the aging process that promotes further the interfacial failure especially in the case of Adhesive 3. However, as the stress on the parts to be joined are very low, a good reliability can be achieved anyway.

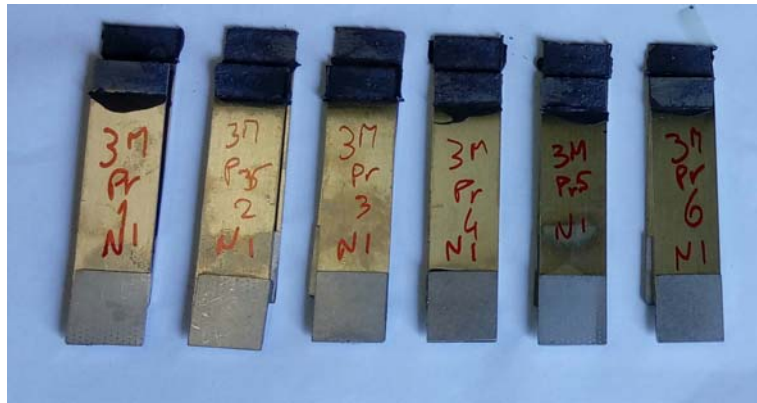


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Figure 24 – Average shear strength of Adhesive 4 + primer with respect to aging time in VE9.

588 In Figure 25 the failure surfaces of the specimens used in the non-aged and end-of-aging tests
589 in the two solutions are shown. The failure appears cohesive before aging, testifying the
590 beneficial effect of the primer, while at the end of aging partially cohesive failures occur which
591 are reflected in the degradation of strength recorded in the tests.
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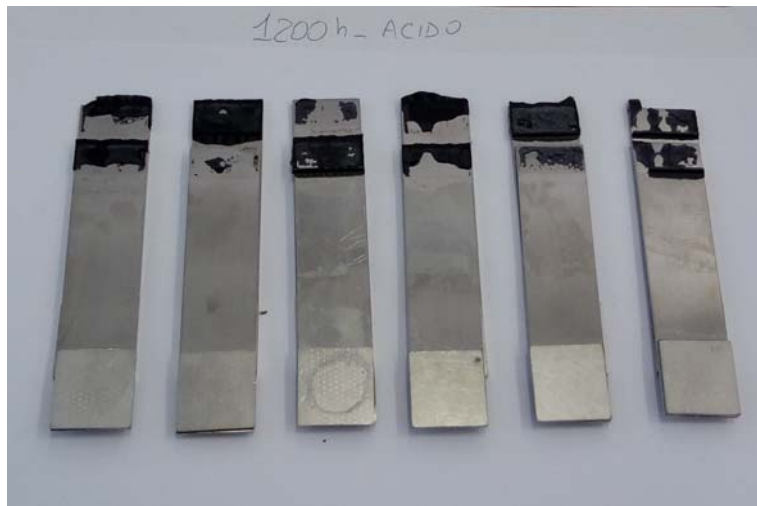
(a)

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(b)

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(c)

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Figure 25 – Failure surfaces of Adhesive 4 plus primer specimens unaged (a), aged 1200 h in VE3 (b) and aged 1200 h in VE9 (c).

598

599 4. Conclusions

600

The introduction of bonding technology in place of welding in the assembly of beverage filling

601

machines was approached by the experimental identification of an adhesive appropriate for

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bonding low surface roughness stainless steel joints subject to chemical aging due to cleaning

603

and sanitization. Three epoxy adhesives, two without and one with a primer, and one modified

604 silane were pre-selected as potentially suitable for application on the AISI 304 stainless steel
605 sheet with different surface finishes and in contact with the acid and alkaline solutions used in
606 COP-SOP.

607 The effect of surface finish evidenced that bonding to just degreased surfaces yields an average
608 shear strength equal or lower than that obtained by sandblasting, as expected, even though in
609 general the value can be sufficient for practical applications without aging.

610 The identification of aging effects required a specifically designed experiment. Joints with 2B
611 tumbled surface finish were immersed in thermostatic baths for aging up to 1200 h
612 corresponding to 20 years of overall contact with the chemicals. A too high rate of spontaneous
613 debondings during immersion characterized Adhesives 2 and 3, making them unreliable for the
614 application. Adhesive 1 showed a good residual strength but also some spontaneous
615 debondings, therefore a second campaign was done on other two epoxy adhesives (Adhesive 4
616 and 5). The latter again showed a substantial number of debondings, while Adhesive 4 yielded
617 results qualitatively similar to Adhesive 1. Adhesive 4 was finally tested also in conjunction with
618 a chemical surface pre-treatment that was made available by the supplier. The results showed
619 no spontaneous debondings after the end of aging so that, even though the residual strength was
620 lower than Adhesive 1 (but still high enough for the application), Adhesive 4 plus primer was
621 the combination that satisfied the demanding aging conditions along with low surface roughness
622 of the material adopted, giving the necessary reliability for the application on this long-lasting
623 type of machinery.

624

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629 macchine e impianti per l'industria alimentare" (New paradigms for the design, construction
630 and operation of machines and plants for the food industry) – CUP D92I16000040009.

631

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