Degradation processes influencing bonded joints

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Abstract: Degradation processes cause huge material and economic losses all over the world. On one hand, in today's globalised society, various climatic changes occur affecting products in different ways. On the other hand, different specific degradation media exist in specific branches of the human activity. Due to the influence of the degradation environment (media), destruction can set in. The aim of the laboratory experiments was to evaluate the influence of the degradation environment occurring mainly in agriculture. The adhesive bond strength properties as well as the influence on adhesive bonded materials were evaluated. For the degrading environment tested, a natural and an artificial fertilisers, machine oil, and water were selected. Also, the influence of corrosion was tested on the bonded material. The surfaces of adhesive materials debased by corrosion inevitably stop fulfilling their functions. The degree of the corrosion effect depends mainly on the type, the material structure, and of course, on the corrosion environment.

Keywords: bonding; bonding technology; degradation; testing of adhesive bonds

Bonding technology is one of the methods of connecting various materials. Its use is limited by two basic circuits of factors. Negative technological factors, such as the unsuitable integrity of bonded surfaces, unsuitable selected film of adhesive in the joint etc., can be eliminated already in the bonded joint preparation and formation.

On the contrary, the factors of the outside medium influence the joint especially by changes in temperature and moisture, by the contact with water and chemicals, and by atmospheric corrosion. It is necessary to consider these factors as practically uncontrollable, however, in the bonding technology application their action is relatively significant.

Although both above-mentioned factors influence in complexity the bonded joints properties, above all their strength, partial examination of each single factor by laboratory tests is important, namely with regard to the concrete adhesive type and to the bonded material. For the bonding technology limit determination in the conditions of agriculture, it is necessary to analyse the specific degradation conditions appearing just in this field. Fertilisers are a specific and, at the same time, a very aggressive category of degradation mediums. This is the reason for laboratory experiments performed in this direction (Vahab 2000).

For the objective evaluation, it is necessary to focus not only on the pertinent changes of stress but also on the bonded material. The degradation medium also influences, more or less, the bonded material. In the extreme, the destruction can occur of the bonded material owing to corrosion. After a certain time, the bonded material surfaces debased in such way stop fulfilling their function. The rate of the corrosion attack depends mainly on the sort and structure of the bonded material and, of course, on the corrosion medium (Arnott *et al.* 1992).

Corrosion is an undesirable change of the material surface, caused by electrochemical and chemical influences of the surrounding medium. We define it as the material degradation whose final result is a partial or total material disarrangement (Mohyla 1995; Pošta 2002).

Optimisation of the corrosion prevention issues from the corrosion processes and from the mechanism of the corrosion protection. The way of the optimal system planning follows from the specification of the function demands and from the medium corrosiveness determination. Economical

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demands, which must be taken into consideration, are an integral part. On the basis of all these input parameters is it possible to propose and determine the system of protection.

MATERIAL AND METHODS

The aim of the laboratory experiments presented were was the evaluation of the degradation medium influence on the mechanical properties of bonded joints taking in consideration the bonded material corrosion. The experiments were carried out according to the standard ČSN EN 1465 (1997) which determines the tensile lap-shear strength of rigid-to-rigid bonded assemblies. The substance of the test is the determination of maximal force which acts parallel with the bonded surface and with the principal axis of the assembly up to the failure. This method corresponds to the operational stress. The force measured at the bonded joint failure is the test result. The tested assemblies were prepared by bonding two adherents of dimensions 100 ± 0.25 × × 25 ± 0.25 × 1.6 ± 0.1 mm. The specified overlapping was 12.5 ± 0.25 mm (ČSN EN 1465 1997).

Laboratory tests were carried out using the standardised test specimens made according to the standard ČSN EN 1465 (1997) from the constructional plain carbon steel S235J0. The chemical composition is presented in Table 1.

Prior to bonding, the surfaces of the bonded specimens were blasted using Al_2O_3 of F24 grain size. Using the profilograph Surftest 301, the following values were determined: *Ra* 2.4 μm (the arithmetic mean of the departures of the profile from the mean line), *Rz* 15.3 μm (the average of maximum peak-to-valley length of five consecutive sampling lengths), and *Rt* 20.2 μm (maximum peak-to-valley length within the assessment length).

For bonding, the two-component epoxide adhesive Bison epoxy universal was used. The components were mixed in the ratio 1:1, the mixture being treatable up to 120 min. The heat stability is from –50°C up to +180°C. The adhesive is suitable for bonding metals, ceramics, and plastics. The perfect curing comes after 24 hours. The orientation strength of the bonded joint is 17 MPa (Firm Materials 2007).

The bonding was carried out using a glass panel. The adhesive was applied on one specimen so that the whole surface was evenly coated in the designated length (12.5 mm). In this layer, two distance wires of 110 μm diameter were placed. The distance wires were laid down parallel to the load force direction of the tensile strength test. Then the second specimen (steel sheet) was put to so that the overlapping of 12.5 mm was reached (according to the standard).The upper specimen was laid using a sheet of equal thickness (1.5 mm) and the assessment was aligned according to the longitudinal axis. Then the assembly was loaded using the weight of 720 g and left in the laboratory for the time prescribed by the instructions (24 hours) for curing at the temperature of 22 ± 2°C (laboratory temperature).

After curing, the marking of the individual assemblies and placing them in relevant mediums followed. As the degradation mediums, water bath, slurry, fertiliser LV15 (granulated, 15% N), and engine oil were used. The slurry composition is very different. During storage, the nitrogen organic acids decompose and lose the action ability in this way (Richter *et al.* 2002).

The strength values of the bonded joints as influenced by these four basic degradation mediums occurring in agriculture were compared with the values measured under laboratory conditions. The degradation processes were evaluated continuously during determined time intervals. The time period between the individual intervals was 15 days starting from the day of total curing. The last measuring was carried out after 90 days in the degradation medium provided that the strength occurred in the measurable range.

Each cycle was finished by the destructive testing of the bonded joints using the universal tensile-strength testing machine. After the joint rupture the maximal force was read, the overlapping length was measured with an accuracy of 0.05 mm, and the rupture type was determined according to ČSN ISO 10365 (1995). Then the overlapping surface (1) and the strength of the bonded joint (2) were calculated (ČSN ISO 10365 1995).

$$S = l_u \times b \tag{1}$$

where:

S – surface of the bonded joint (ČSN EN 1465 1997) (mm^2)

l_u – overlapping length (mm)

b – overlapping width (mm)

Table 1. Chemical composition of the bonded material

Element	Fe	C	Mn	Cr	Ni	Al	Cu	Nb	Ti
Weight (%)	99.5	0.047	0.24	0.076	0.017	0.065	0.039	0.007	0.016

$$\tau = \frac{F}{S} \quad (2)$$

where:

τ – shear strength (MPa)

F – loading power (N)

S – surface of the bonded joint (ČSN EN 1465 1997) (mm^2)

Further the corrosion was evaluated of the bonded material, which was without any corrosion prevention. The corrosion loss was determined by weighing, using at first the balance weighing machine Chirana P3/200 of 0.1 g accuracy. The determined orientation weight served for the adjustment of the balance weighing machine WA type PRL T A14 of 0.00001 g accuracy. For the inaccuracy reduction, the measuring was carried out repeatedly and then evaluated.

RESULTS

The results of the laboratory experiments are presented in Figure 1. The experiments were carried out according to the standard ČSN EN 1465 (1997). Single bonded joints were destructively tested using the universal tensile-strength testing machine. From the results presented in Figure 1 the influence of various degradation mediums is evident. The time behaviour is illustrated by the polynomial function of the second degree which was derived from the correlation field of the measured points showed in the XY graph.

For the correct evaluation, it is also important to determine the dependence intensity of the given dependence. It is the task for the correlation analysis. The closeness of the dependence is measured by the use of the determination index whose values can vary from 0 up to 1. According to the statistical evaluation, the closeness R^2 for all the evaluated degradation mediums is very high. For laboratory environment the closeness R^2 is high.

The orientation strength declared by the producer was reached. After curing, the strength of the bonded joints was about 18.9 MPa. According to the course of the curves, it is possible to state that the strength decrease of single joints is expressive. By the comparison of the curve which represents the laboratory conditions with other curves which represent the degradation mediums, it is clear that a considerable danger of the stress decrease exists. This dangerous phenomenon should direct towards the prevention from the access of the degradation mediums mentioned or at least towards the limitation of their action time.

The real course of the measured values is better expressed by the line chart presented in Figure 2,

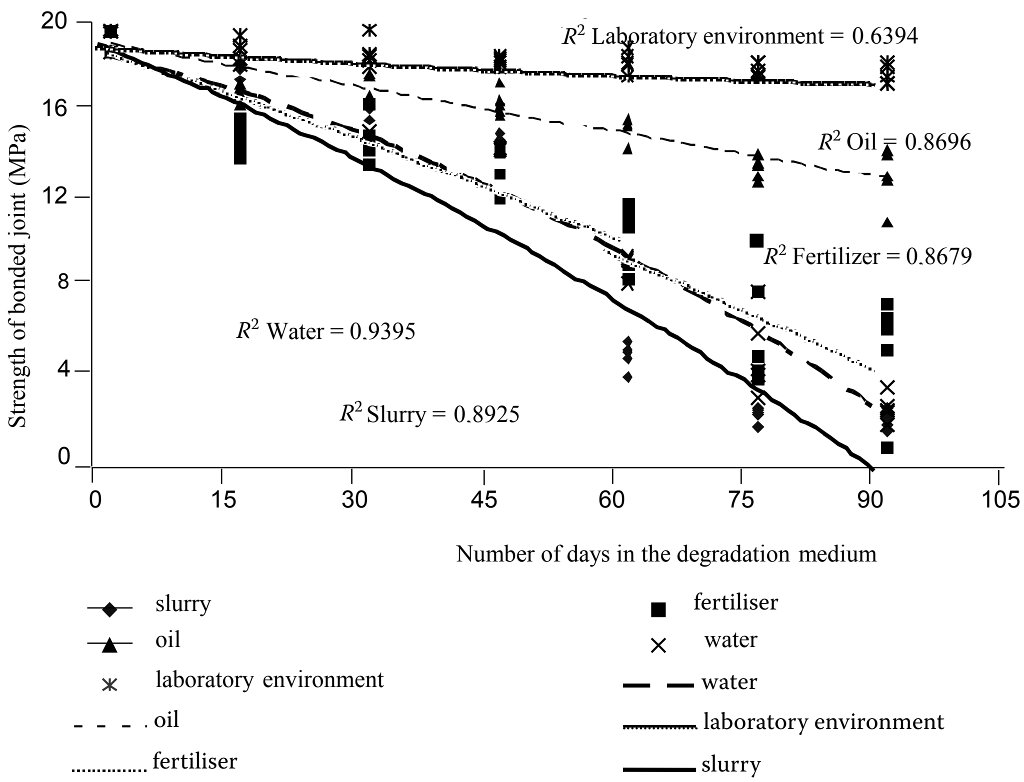

Figure 1. Influence of degradation medium on the bonded joint strength

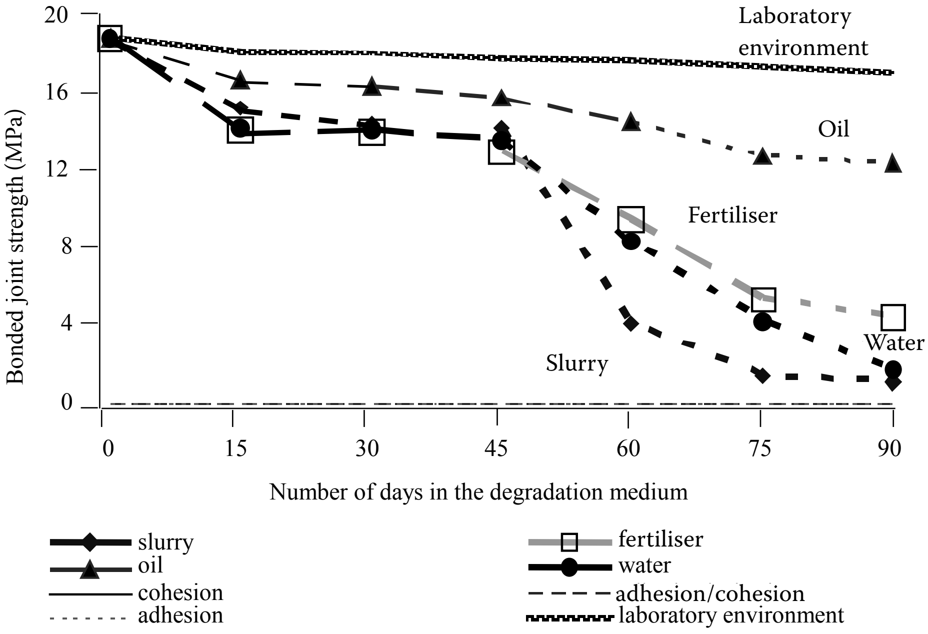

Figure 2. Influence of degradation mediums on the bonded joint strength and the failure types

showing the influence of the degradation mediums (water bath, fertiliser LV 15, and slurry). Using these mediums, a rapid strength decrease occurred after 45 days. Not only the fracture type was determined (also presented in Figure 2), but also the diffusion of the degradation mediums into the bonded joint. In this way the functional surface continuously decreased and a very fast strength reduction occurred.

On the basis of the picture analysis, the failure type was determined. The cohesive fracture type was found with the specimens placed in the laboratory. Fracture types of the specimens placed in four degradation mediums were different. In the first phase, the fracture type of all joints was cohesive. Later, the fracture type changed in the dependence on the degradation medium (see Figure 2). When the adhesive failure was identified, corrosion under the adhesive layer was found at the same time and due to this the adhesive strength decreased. This ascertainment offers the explanation for the bonded joints fast strength reduction. Not only a decrease occurred of the cohesion strength by the diffusion of moisture and chemical substances, but also a decrease of the bonding power – adhesion.

The degradation of the bonded material (steel S235J0) showed itself by its weight loss and the outward change. For the corrosion loss evaluation, the measured bonded specimen surface of about 10 750 mm^2 was taken into consideration.

Figure 3 presents the measured values. The corrosion behaviour of the bonded material was considerably influenced by the degradation medium. The highest values of the tested assemblies weight loss were determined with the exposition in slurry. The closeness R^2 of the curves was determined in the interval from 0.933 up to 0.985, thus very high.

The fertiliser dissolved in water bath was determined as the second aggressive medium. Here the increase of the corrosion weight losses stopped after 60 days. This phenomenon was caused by the creation of a continuous layer on the surface.

An even corrosion layer was found with slurry, fertiliser, and water bath. An uneven corrosion layer occurred in the test assemblies placed in the oil bath and in the laboratory.

CONCLUSION

On the basis the experiments carried out, it is possible to state that the resulting strength of bonded joints decreases with increasing time of exposition to the active surrounding medium. The strength decrease depends on the surrounding medium specific conditions.

In laboratory conditions, that is at 22 ± 2°C and 45 ± 5% relative humidity, the average strength decrease of 9.5% was found after 90 days. However, a significant strength decrease occurred after the first 15 days, when the strength decrease as compared with the values obtained after 24 hours (time necessary for perfect curing) was 3.7%. (0.7 MPa). The following strength decrease of 1.17% (0.22 MPa) after each 15 days was stable. On the basis of the standard deviation calculation, the average variance

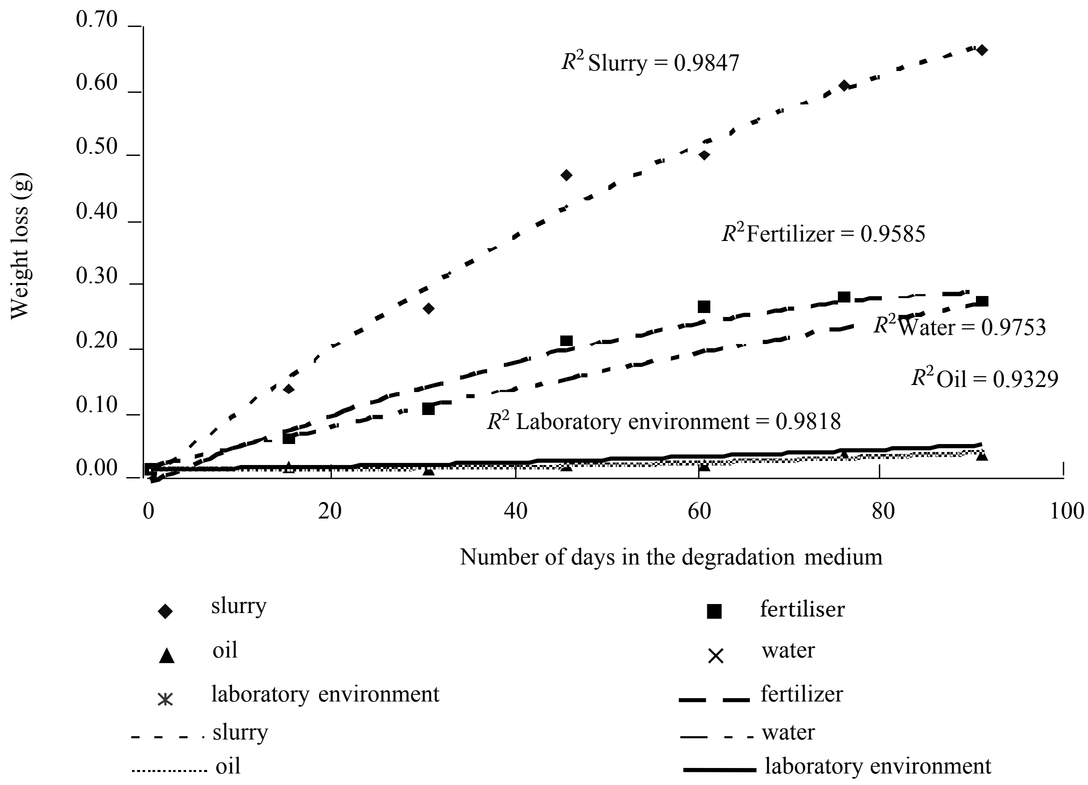

Figure 3. Corrosion weight losses at the degradation mediums attack

of 0.37 MPa was determined. The almost minimal strength decrease of the bonded joint was perceptible under laboratory conditions. Only the cohesive failure type occurred.

Accordingly, with all bonded joints exposed to various degradation mediums a significant strength decrease occurred in the first 15 days, namely in slurry by 19.1% (3.62 MPa), in fertiliser LV 15 by 24.9% (4.72 MPa), in engine oil by 12.26% (2.33 MPa), and in water bath by 24.14% (4.57 MPa).

With bonded joints exposed in slurry, a considerable strength decrease occurred already after 45 days, namely by 53.25%, and also the failure type changed to the adhesive one.

The bonded joints exposed in fertiliser LV 15 were also very much degraded. After 90 days, the strength decrease was by 76.2%. A significant decrease occurred after 45 days. The failure surface evaluation revealed a change in colour from transparent to yellow over the whole cross section. This fact attests to the penetration of the fertiliser dissolved in water into the adhesive.

In the engine oil, almost continuous degradation occurred, in average by 4.39% after each 15 days. After 90 days, the total strength decrease was 34.2%. The failure surface was completely of the adhesive type, the engine oil was contained in the whole cross section.

The last degradation medium was the water bath, which is used very often. After 90 days, the strength decrease by 90.3% occurred. As well as with the fertiliser and slurry, the critical strength decrease occurred after 45 days.

The environment, and also the degradation mediums, are significant factors influencing the bonded joints quality. Many times the bonded joints spontaneous destruction can occur after a very short time if these parts are exposed to the degradation mediums.

References

Arnott D.R., Wilson A.R., Rider A.N., Lambrianidis L.T., Farr N.G. (1992): Studies of the degradation of metal-adhesive interfaces with surface analysis techniques. Applied Surface Science, **70/71**: 109–113.

ČSN EN 1465 (1997): Adhesives – Determination of Tensile Lap-shear Strength of Rigid-to-rigid Bonded Assemblies. Czech Standard Institution, Prague. (in Czech)

ČSN ISO 10365 (1995): Adhesives – Designation of Main Failure Patterns. Czech Standard Institution, Prague. (in Czech)

Firm Materias (2007): Epoxy Adhesives BISON.

Mohyla M. (1995): Technology of Metal Surface Treatments. 1. Ed., VŠB, Ostrava. (in Czech)

Pošta J. (2002): Machines Serviceability. CULS, Faculty of Engineering, Prague. (in Czech)
Richter R., Hlušek J., Ryant, P., Lošák T. (2002): Organic fertilizers and their position in the agriculture. Available at http://www.mendelu.cz/user/ryant/publikace/Organicka_hnojiva_02.htm. (accessed June 26, 2008)
Vahab M. (2000): On the use of fracture mechanics in designing a simple lap adhesive point. Journal of Adhesive Science, **14**: 851–858.

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Abstrakt

Müller M., Chotěborský R., Hrabě P. (2009): **Degradační procesy ovlivňující lepené spoje**. Res. Agr. Eng., **55**: 29–34.

V celosvětovém měřítku způsobují degradační procesy značné materiální, ale i ekonomické ztráty. Na jedné straně dnešní globalizované společnosti stojí různé podnební a klimatické změny působící odlišně na výrobky. Na straně druhé jsou to různá specifická degradační média vyskytující se v konkrétním odvětví lidské činnosti.Vlivem působení degradačního prostředí/média může dojít, až k destrukci. Cílem laboratorních experimentů bylo hodnocení vlivu degradačního prostředí vyskytujícího se převážně v zemědělské výrobě. Hodnoceny byly změny pevnostních vlastností spoje, ale rovněž vliv na spojovaný materiál. Experimentálně hodnocenými degradačními prostředími bylo přírodní a umělé hnojivo, strojní olej a vodní lázeň. U spojovaného materiálu byl hodnocen rovněž vliv koroze. Korozní znehodnocené povrchy spojovaných materiálů po určité době nevyhnutelně přestanou plnit rovněž svoji funkci. Stupeň korozního působení je hlavně závislý na druhu, struktuře materiálu a samozřejmě na korozním prostředí.

Klíčová slova: degradace; lepení; laboratorní experimenty lepených spojů; technologie lepení

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