

Influence of dusty micro-particles contamination on adhesive bond strength

A. Krofová* and M. Müller

Department of Material Science and Manufacturing Technology, Faculty of Engineering, Czech University of Life Science, Kamýcká 129, CZ-16521 Prague, Czech Republic; *Correspondence: krofovaa@tf.czu.cz

Abstract. A necessity for a bond creation is one of common attributes of production companies. An adhesive bonding technology is a method of a connecting. This method is suitable for workings with a single and serial production. Many research projects dealt with a preparation of adhesive bonds, degradation aspects etc. An area, which has not been properly investigated at present, is an influence of a contamination of the adhesive bonds by dusty micro-particles, e. g. from a ventilation of assembly shops, production hall etc. The research was focused on the evaluation of the influence of dusty micro-particles contamination of the two-component epoxy adhesive at the hardening process. The dusty micro-particles were gained from the filtering equipment used in a production hall. Sizes of gained dusty particles were analysed on sieves of dimensions 315 µm, 250 µm, 160 µm, 90 µm. Subsequently, these particles were added in various ratios into the mixture of the adhesive during its preparation. The adhesive bonds containing the dusty particles of the sizes 250 µm, 160 µm and 90 µm showed the fall of the adhesive bond strength. The adhesive bonds containing the dusty particles of the size 315 µm showed the mild increase of the adhesive bond strength. The failure area did not change owing to the contamination of the adhesive bond with the dusty particles.

Key words: adhesive bond, elongation, failure area, dusty micro – particles.

INTRODUCTION

A necessity for a bond creation is one of common attributes of production companies. An adhesive bonding technology is a method of a connecting. This method is suitable for workings with a single and serial production. Many research projects dealt with a preparation of adhesive bonds, degradation aspects etc. An area, which has not been properly investigated at present, is an influence of a contamination of the adhesive bonds by dusty micro-particles, e.g. from a ventilation of assembly shops, production hall etc. The adhesive bonds enable to substitute welding seams and mechanical connecting elements, help to decrease the material fatigue and failure around the heat-influenced area.

Undesirable changes of the adhesive properties occur not only when using products but they can already occur at own process of the working of adhesives and at their storing (Müller & Valášek, 2014).

The adhesive bonding is a modern method of connecting different materials. This method is still seldom spread in spite of its advantages just because of its low durability at exposing to unfavourable influences. An assumption for securing the service life of the adhesive bond is keeping basic processes at its creation. The essential factor is a cleanness of a working environment under given conditions under which the bond is created and also a character of degradation processes influencing strength of whole construction. This undesirable but inevitable factor has to be analysed.

The dust which can be found all in the air is a significant source of a rise of the dangerous working environment. It is a restriction which cannot be avoided in a common practice.

Many studies researching the air quality in various environments were performed in past (di Giorgio et al., 1996; Jones, 1999). Spaces in which people are moving when doing various working activities were also investigated (Bluyssen, 1996; Karwowska, 2003; Kic & Chládek, 2010; Kic & Růžek, 2014), farms and agricultural plants for poultry-farming too (Karwowska, 2005; Kic et al., 2007; Kic et al., 2012; Nimmermark et al., 2009). The dust is a general term for solid particles of a mean smaller than 0.075 mm. It consists of cells of a human skin, a small amount of a vegetable pollen, human and animal hair, textile fibres, paper fibres and many other materials which can be found depending on the particular environment (Nõu & Viljasoo, 2011). We can suppose on a basis of general pieces of knowledge that the pollution of adhesives will cause a fall of a resultant adhesive strength of the adhesive bond. Zhang et al. state in their work that the adhesive is more resistant to water and its strength is increasing when adding starch particles into the adhesive (Zhang et al., 2015).

By adding fractions into the macro-molecular materials (which are also polymers) it comes to changes of a chemical chain on which the properties of the polymers depend (Ducháček, 2006). Generally it can be said that the change of the molecular mass of the macro-molecular materials influences their mechanical properties in both ways-in positive as well as in negative one (Ducháček, 2006; Valášek, 2014).

However, the cohesion mechanism of the adhesive bond depends on an adhesion, a cohesion and a wettability of the adhesive bonded surface. During the hardening e.g. of two-component epoxy adhesives the epoxy resins react with hardeners and they create macro-molecules. The polymerisation process influences the resultant strength of the adhesive bond. It can be supposed that it comes to lowering of the resultant adhesive bond strength at the adhesive bond contamination during the hardening process (the polymerisation).

The aim of this work was to perform laboratory experiments focused on the strength of the adhesive bond polluted with the dust gained from the filtering equipment used in production halls using the information gained from available references and by means of recommended introduced standard processes. Results should serve for the evaluation of the influence of two-component epoxy adhesives contamination with dusty micro-particles on the adhesive bond and because of the fact that the bond cleanness is the main factor causing the failure of adhesive bonded constructions it is necessary to comprehend and explain their degradation processes. Pieces of knowledge will be subsequently used at the evaluation of properties of the adhesive bonds at their creation depending on the environment contamination.

MATERIAL AND METHODS

The research was focused on the evaluation of the influence of dusty micro-particles contamination of the two-component epoxy adhesive Glue Epox Rapid F (marked as a matrix in the text) at the hardening process. The dusty micro-particles were gained from the filtering equipment used in a production hall.

In the production hall there were e.g. these technologies: blasting cabin, a working place of connecting and dividing of materials. A source of higher dusty particles in the filtering equipment was obviously the blasting equipment.

Two-component epoxy adhesive Glue Epox Rapid F was prepared by mixing a part A (a low-molecular epoxy resin) and a part B (a polyamide hardener). A ratio for mixing the parts A : B was 100 : 45 of mass parts. A processing time of the adhesive bonded mixture at the temperature 22 ± 2 °C is max. 10 minutes. An exceeding of the processing time influences the adhesive bond strength in the negative way (it decreases) (Müller & Valášek, 2013).

Sizes of gained dusty particles were analysed on sieves of dimensions 315 µm, 250 µm, 160 µm, 90 µm (Fig. 1). The dusty micro-particles were of an irregular shape. The size of used dusty particles was given by a sieve analysis (that means by size of used sieves). The particles below 90 µm were not used. A reason was an impossibility to set the size.

Subsequently, these particles were added in various ratio into the mixture of the adhesive during its preparation.

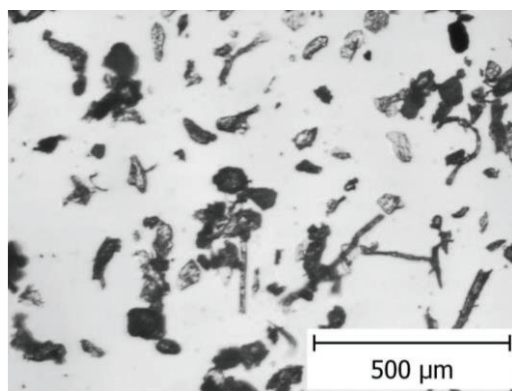


Figure 1. Dusty particles of size 90 µm.

The mass ratios of test specimens are following 100,000 mg of a matrix: 250 mg, 500 mg, 750 mg and 1,000 mg of dusty particles. The influence of the dusty particles contamination was investigated at the adhesive bonds according to the standard CSN EN 1465 (2009).

The basis of adhesive bonds laboratory testing was the determination of the tensile lap-shear strength of rigid-to-rigid bonded assemblies according to the standard CSN EN 1465 (2009) (Equivalent is BS 1465). The tests were performed using the steel S235J0 specimens of dimensions 100 x 25 x 1.5 mm.

The surface preparation is important and should guarantee good strength on the boundary of adherents. Steel adherents were firstly mechanically surface treated with blasting by a synthetic corundum (Al_2O_3) with a size of a fraction F80. Using the profilograph Surftest 301 following values were determined: $R_a 1.83 \pm 0.25$ µm, $R_z 10.42 \pm 0.88$ µm. Then the surface was cleaned and degreased using acetone P6401 and prepared to the application.

The adhesive bonds were created until 5 ± 2 minutes after adding the dusty particles into the matrix.

An even thickness of the adhesive layer was reached by a constant pressure 0.5 MPa. The lapping was according to the standard 12.50 ± 0.25 mm. The adhesive bonds were hardened for 48 hours at the laboratory temperature (22 ± 2 °C). The adhesive layer thickness in the adhesive bonds was 0.15 ± 0.15 mm. The real thickness of the adhesive layer was set on the basis of the picture analysis in the cut of the adhesive bonds by means of the microscope.

The tensile strength and the elongation tests were performed using the universal tensile strength testing machine LABTest 5.50ST (a sensing unit AST type KAF 50 kN, an evaluating software Test & Motion). A speed of the deformation corresponded to 6 mm min^{-1} .

The failure type according to CSN ISO 10365 (1995) was determined at the adhesives bonds.

RESULTS

The adhesive bond strength (without the contamination with the dusty particles) was 12.02 ± 0.76 MPa. It showed a fall at the adhesive bonds contaminated with the dusty particles of the sizes 250 μm , 160 μm and 90 μm . The fall was in the interval 0.79 to 18.85% (all concentrations). The adhesive bond strength increased at the adhesive bonds contaminated with the dusty particles of the size 315 μm . The strength increase was in the interval 4 to 5%. A graphic presentation of the results of the adhesive bond strength prepared by 3D graph with method of the smallest squares can be seen from Fig. 2.

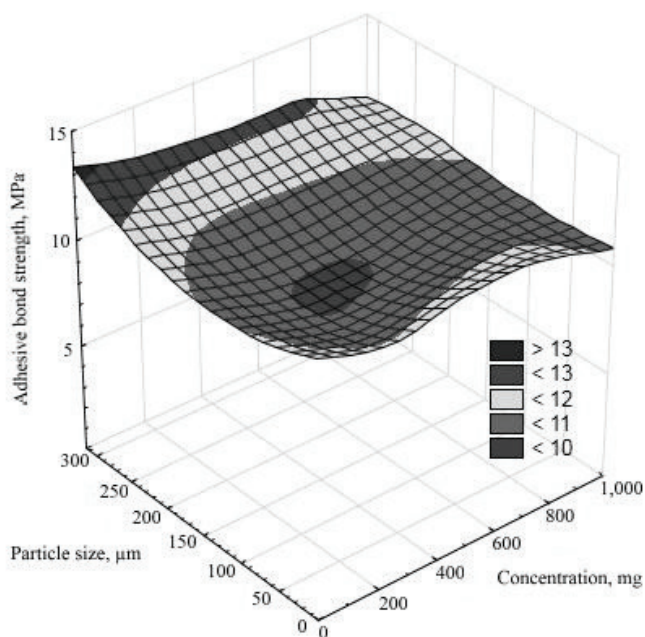


Figure 2. Influence of concentration and size of dusty particles on adhesive bond strength.

The elongation of the adhesive bond (without the contamination with dusty particles) was $1.27 \pm 0.10\%$. It showed a fall at the adhesive bonds contaminated with the dusty particles of the sizes $160\ \mu\text{m}$ and $90\ \mu\text{m}$. The elongation fall was in the interval 0.69 to 20.08% (all concentrations). The elongation of the adhesive bond increased at the adhesive bonds contaminated with dusty particles of the sizes $315\ \mu\text{m}$ and $250\ \mu\text{m}$. It can be supposed that it was not the impurity but the filler at this size.

The elongation increase was in the interval 9.84 to 75.50% .

A graphic presentation of the results of the elongation of the adhesive bond prepared by 3D graph with method of the smallest squares can be seen from Fig. 3.

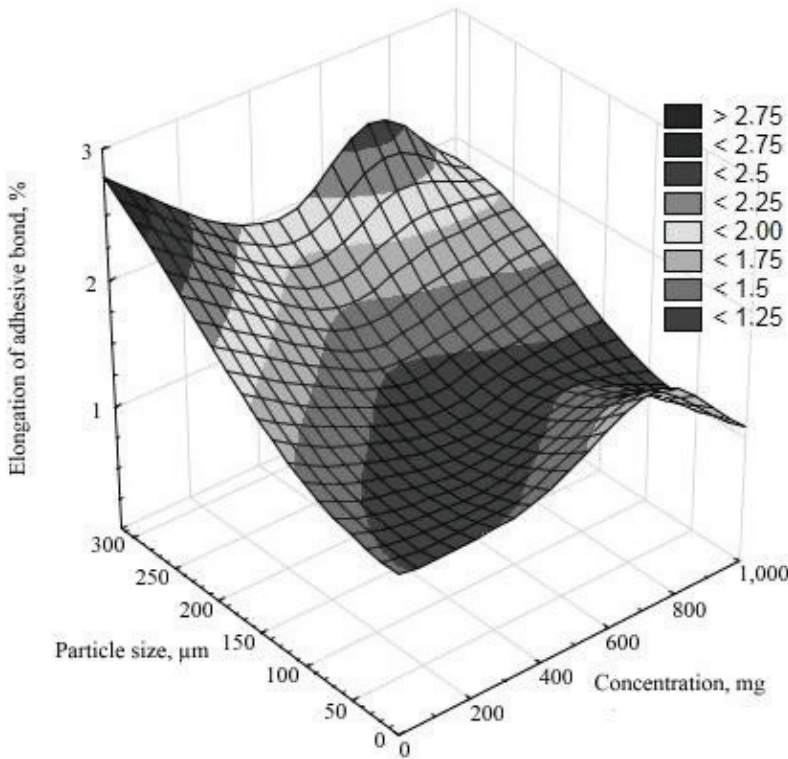


Figure 3. Influence of concentration and size of dusty particles on elongation of adhesive bond.

F-test is used for testing a significance of a difference of two dispersion variances. A value of the criteria p was gained by a calculation in the programme Statistica.

F-test was used for the statistical comparison. The zero hypothesis H_0 presents the state when there is no statistically significant difference ($p > 0.05$) among tested sets of data from their mean values point of view.

The results of F-test are from the point of view of the influence of dusty particles concentration on the adhesive bond strength following: the hypothesis H_0 was confirmed at the dusty particles of the size $250\ \mu\text{m}$ ($p = 0.3730$), so there is no difference among particular tested concentrations in the significance level 0.05 . The hypothesis H_0 was not confirmed at other sizes of the dusty particles: $315\ \mu\text{m}$ ($p = 0.0262$), $160\ \mu\text{m}$ ($p = 0.0016$)

and 90 μm ($p = 0.0046$), so there is a difference among particular tested concentrations of dusty particles in the significance level 0.05.

The results of F-test are from the point of view the influence of the size of the dusty particles on the adhesive bond strength following: The hypothesis H_0 was confirmed at the concentration of the dusty particles 1,000 mg ($p = 0.1991$), so there is no difference among particular tested sizes of the particles in the significance level 0.05. The hypothesis H_0 was not confirmed at other concentrations of the dusty particles: 750 mg ($p = 0.0061$), 500 mg ($p = 0.0003$) and 250 mg ($p = 0.0012$), so there is a difference among particular tested sizes of the dusty particles.

The results of F-test are from the point of view of the concentration of the dusty particles on the elongation of the adhesive bonds following: the hypothesis H_0 was confirmed at the sizes of the dusty particles 250 μm ($p = 0.0505$) and 160 μm ($p = 0.0656$), so there is no difference among particular tested concentrations in the significance level 0.05. The hypothesis H_0 was not confirmed at other sizes of the dusty particles 315 μm ($p = 0.0263$) and 90 μm ($p = 0.00009$), so there is a difference among particular tested concentrations of the dusty particles in the significance level 0.05.

The results of F-test are from the point of view of the influence of the size of the dusty particles on the elongation of the adhesive bonds following: the hypothesis H_0 was not confirmed at all concentrations of the dusty particles: 1,000 mg ($p = 0.0044$), 750 mg ($p = 0.0014$), 500 mg ($p = 0.0009$) and 250 mg ($p = 0.0056$), so there is the difference among particular tested sizes of the dusty particles in the significance level 0.05.

A composite comes into being at the contamination of the adhesive bond with the dusty particles. The resultant strength of the adhesive bond usually decreases at the composite materials filled with various particles. More significant changes of the strength occur by acting the created adhesive bond by degradation processes. Also a storing and a transport have similar influence on the creation of the adhesive bond (Müller et. al., 2009 & 2013; Balkova et. al., 2002; Doyle & Pethrick, 2009, Liljedahl et. al., 2007). The fall of the adhesive bond strength ranged in the interval 2 to 18% at the tested adhesive (Müller, 2013).

Exposing of the adhesive bonds to impurities, water, extreme temperatures or chemical stuffs can influence the process of the adhesive bond failure (Nolting et al., 2008). The fall of the adhesive bond strength is also connected with it. The change of the strength of the adhesive bonds caused by the adhesive bond production was smaller than 19%.

Sargent (2005), Doyle & Pethrick (2009) and claim that the changes in the environment can act both the way by which the physical properties of the adhesive change in time, and the strength of the boundary of the adhesive – adherent.

Undesirable changes of mechanical properties usually occur at the contamination by the particles at the adhesive bond creation (Brown, 1999; Ducháček 2006; Mleziva, 2008; Valášek 2014).

The adhesive bonds without the dash of the dusty impurities are of adhesive-cohesive failure area (Fig. 4). The failure area of the adhesive bonds contaminated with the dusty particles did not change (Fig. 5).

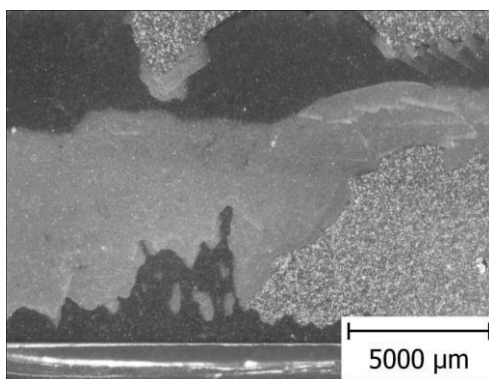


Figure 4. Adhesive-cohesive failure area – adhesive without dusty particles.

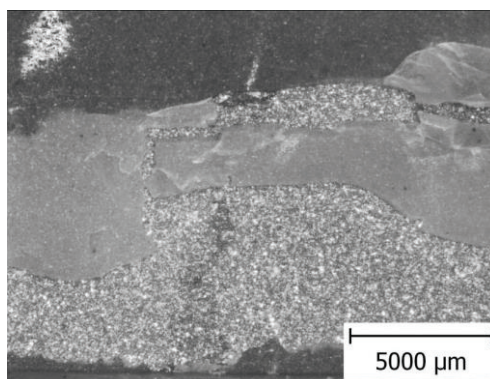


Figure 5. Adhesive-cohesive failure area – adhesive contaminated with dusty particles of size 315 μm and concentration 1,000 mg.

CONCLUSION

Following conclusions can be deduced from the research focused on the influence of the contamination with the dusty micro-particles on the adhesive bond strength:

- The adhesive bonds containing the dusty particles of the sizes 250 μm, 160 μm and 90 μm showed the fall of the adhesive bond strength. The strength fall of the adhesive bond did not exceed 18%. The adhesive bonds containing the dusty particles of the size 315 μm showed the mild increase of the adhesive bond strength.
- The dusty particles above 160 μm caused the increase of the elongation of the adhesive bonds.
- The failure area did not change owing to the contamination of the adhesive bond with the dusty particles.

ACKNOWLEDGEMENT. Supported by Internal grant agency of Faculty of Engineering, Czech University of Life Sciences in Prague no: 2015:31140/1312/3106.

REFERENCES

- CSN EN 1465. 2009. Adhesives. Determination of tensile lap-shear strength of bonded assemblies. Czech Office for Standards, Metrology and Testing, Prague, Czech Republic. (in Czech)
- CSN ISO 10365. 1995. Adhesives. Designation of main failure patterns. Czech Office for Standards, Metrology and Testing, Prague, Czech Republic. (in Czech)
- Balkova, R., Holcnerova, S. & Cech, V. 2002. Testing of adhesives for bonding of polymer composites. *International Journal of Adhesion & Adhesives* **22**(4), 291–295.
- Bluyssen, P.M. 1996. European indoor air quality audit project in 56 office buildings. *Indoor Air*. **6**(4), 221–238.
- Brown, R. 1999. *Handbook of Polymer Testing: Physical Methods*. (M. Dekker, Ed.). p. 845. New York: CRC Press.

- Di Giorgio, C., Krempff, A., Guiraud, H., Binder, P., Tiret, C., & Dumenil, G. 1996. Atmospheric pollution by airborne microorganisms in the city of Marseilles. *Atmospheric Environment* **30**(1), 155–160.
- Doyle, G. & Pethrick, R.A. 2009. Environmental effects on the ageing of epoxy adhesive joints. *International Journal of Adhesion & Adhesives* **29**(1), 77–90.
- Ducháček, V. 2006. *Polymers: production, properties, processing, use*. 2nd ed., p. 278. Prague: ICT Prague Press.(in Czech)
- Jones, A.P. 1999. Indoor air quality and health. *Atmospheric Environment* **33**(28), 4535–4564.
- Karwowska, E. 2003. Microbiological air contamination in some educational settings. *Polish Journal of Environmental Studies* **12**(2), 181–185.
- Karwowska, E. 2005. Microbiological air contamination in farming environment. *Polish Journal of Environmental Studies* **14**(4), 445–449.
- Kic, P., Hubený, M., Ledvinka, Z., Tumová, E., Giner, C.C., & Torres, C. M. 2007. Control of indoor environment in housing of laying hens. *Conference Proceedings – 3rd International Conference, TAE 2007: Trends in Agricultural Engineering 2007*. 212–214.
- Kic, P., & Chládek, L. 2010. Microclimate in tutorial and research brewery during winter season. *Conference Proceeding – 4th International Conference, TAE 2010: Trends in Agricultural Engineering 2010*. 311–315.
- Kic, P., & Růžek, L. 2014. The microbiological environment in specific rooms of a university campus. *Agronomy Research* **12**(3), 837–842.
- Kic, P., Růžek, L., Ledvinka, Z., Zita, L., & Gardianoba, I. 2012. Pollution of indoor environment in poultry housing. *Engineering for Rural Development* **11**, 480–483.
- Liljedahl, C.D.M., Crocombe, A.D., Wahab, M.A. & Ashcroft, I.A. 2007. Modelling the environmental degradation of adhesively bonded aluminium and composite joints using a CZM approach. *International Journal of Adhesion & Adhesives* **27**(6), 505–518.
- Mleziva, J. 2008. *Polymers – Production, Structure, Properties and Applications* (1st ed.). Prague: Czech Technical University Publishing House. (in Czech)
- Müller, M. 2013. Research of liquid contaminants influence on adhesive bond strength applied in agricultural machine construction. *Agronomy Research* **11**(1), 147–154.
- Müller, M., Chotěborský, R. & Hrabě, P. 2009. Degradation processes influencing bonded joints. *Research in Agricultural Engineering* **55**(1), 29–34.
- Müller, M. & Valášek, P. 2013 Assessment of bonding quality for several commercially available adhesives. *Agronomy Research* **11**(1), 155–162.
- Müller, M. & Valášek, P. 2014. The logistics aspects influencing the resultant strength of adhesives at practical application. *Agronomy Research* **12**(1), 285–290.
- Nimmermark, S., Lund, V., Gustafsson, G., & Eduard, W. 2009. Ammonia, dust and bacteria in welfare-oriented systems for laying hens. *Annals of Agricultural and Environmental Medicine* **16**(1), 103–113.
- Nolting, A.E., Underhill, P.R. & DuQuesnay, D.L. 2008. Variable amplitude fatigue of bonded aluminum joints. *International Journal of Fatigue* **30**(1), 178–187.
- Nõu, T., & Viljasoo, V. 2011. The effect of heating systems on dust, an indoor climate factor. *Agronomy Research* **9**(1), 165–174.
- Valášek, P. 2014. *Polymeric Materials*. p. 67. Czech University of Life Sciences Prague. ISBN 978-80-213-2489-3. (in Czech)
- Sargent, J.P. 2005. Durability studies for aerospace applications using peel and wedge tests. *International Journal of Adhesion & Adhesives* **25**(3), 247–256.
- Zhang, Y., Ding, L., Gu, J., Tan, H., & Zhu, L. 2015. Preparation and properties of a starch-based wood adhesive with high bonding strength and water resistance. *Carbohydrate Polymers* **115**, 32–37.