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FATIGUE TESTS OF THE METAL-TO-METAL ADHESIVE BOND USED IN DENTAL IMPLANTOLOGY INCLUDING ADHESIVE DRYING TIME

KEYWORDS

Adhesive, adhesive connection, RelyX, dental implant, fatigue.

ABSTRACT

Fatigue tests carried out on the dental implant experimental adhesive connection proved that the adhesive drying time is one of the factors responsible for adhesive higher durability. So far this factor has not been taken into account during experimental studies. Specimens of adhesive connection with RelyX dental adhesive were allowed to dry for 1, 3 and 8 days. After each drying time the fatigue tests were carried out. As the adhesive drying time increased, its durability also increased. After 3 days of drying, the adhesive durability were twice higher than the adhesive durability with 1 day of drying. Specimens with 8 days adhesive drying revealed the unlimited durability for load level applied during the study. Research has shown that the drying time of the adhesive is an important factor that has so far been neglected. Allowing the adhesive to dry for at least 3 days increases the chances of successful implant treatment. In order to avoid the upper plate of the machine hitting the sample, the tests were carried out in forces from Fmax = -1N to Fmin = -50N. The frequency with which the test was carried out was set at f = 1Hz. The abutment roughness were measured with the MarSurf PS 10 (5667) measuring device with the average roughness Ra was 1.8 μm. The metal surface prepared in this way bonds better with the dental adhesive.

1. Introduction

Dental implants have existed about 2500BC [1]. World has heard more about dental implant just about 30 years ago by Per-Ingvar Brånemark paper [2]. He was the first person who touched the aesthetics aspects of teeth loss. Over time it turned up that just aesthetics is not enough anymore. Biological and mechanical aspects started to develop [3]. Discovered biomaterials with perfect properties for implant surface, as titanium or zirconia [4–10]. Currently the most common research concern the osteointegration process, which also Brånemark was the first person who discovered it. This is the process of accreting the implant titanium screw with the bone [11–15]. The parameters which have influence on this process are the problem. FEM analysis is very popular method to investigate the behavior of dental implant in the mechanical way and to assess the long-term implant stability and level of treatment success [16–21]. Other research are connected with the screw and abutment connection strength [22, 23]. Unfortunately, there are still no effective research results for the upper part of dental implant system (adhesive and crown part), especially for the non-standard treatment cases.

Experimental research presented in this paper can help to assess the durability of dental adhesive used in crown connection [24]. It will eventually permit to obtain the adhesive durability with the various range of drying time. The aim of this paper is to define the fatigue durability of dental implant adhesive connection with the prosthetic tooth crown taking into account the adhesive drying time.

1.1. State of the art

With the age of each person, individual parts of the body slowly stop working as efficiently as at times of youth. Wearing out the joints or loss the teeth is the natural way of life. Unfortunately, no human on the Earth is born with “the spare parts package”, which could be replaced. There is no possibility to buy the new ones at the store and no matter how much we take care of ourselves, after some

time the eyesight is already failing, the joints are wearing out and the heart doesn't beat so strong anymore. For this reason, medicine and prosthetics development initiated the work on spare parts for people in need. Designing the dentures has begun. Improving denture functions improves patients satisfaction [25–27]. The artificial dentures can be implanted into the human body replacing the worn ones. Prosthetics elements very often take over functions of replaced organs and work just as well as the original parts [28, 29].

The one and only natural guarantee time applies to our teeth and more specifically, to the primary teeth. Unfortunately, the nature doesn't pamper people and guarantees teeth replacement during childhood, not in the old age and not every teeth but only twenty of them. In the place of primary teeth, permanent teeth erupt, which are not covered by any natural warranty [30]. Teeth are one of the most important organ in human body and – as well as joints – wear out the most. Teeth loss causes the aesthetic disturbance, might affect self-esteem and is the reason of bone loss, which leads to face shape deformation. In very extreme cases the teeth loss may lead to problem with grinding food, malnutrition and in the end – even to death [31]. This is the reason why dental implantology came into being. Dental implants are not natural teeth [32] but ensure the aesthetic effect and also prevent resorption of the alveolar bone [33, 34]. An exemplary dental implant and a dental implant system with connected parts are showed in the Fig. 1.

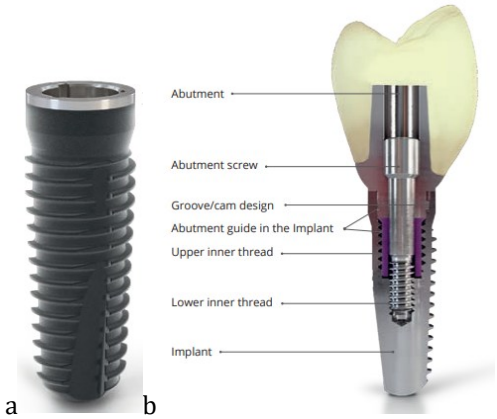


Fig. 1. Dental implant, where: a – implant, b – dental implant system [35]

1.2. Motivation of the work

Despite the fact that implantology is a quite new direction of development, the safety of use is very high. Titanium implants simulating human proper teeth are also the comfort and the aesthetic effect never seen before. Anyways, it doesn't mean that the method has been thoroughly tested and is free of defects. The mean problem is connected to adhesive not enough strong and prosthetic crown falling out. Under the influence of daily activity, the forces acting inside the mouth and the microgaps appearing between the dental implant screw and the implant abutment [36], adhesive is crumbling and the crown is usually falling out. This is especially true of non-standard cases that go beyond standard procedures of dental implant treatment. Research was carry out with a maximum load of 50 N. The frequency was set at f = 1 Hz. The abutment roughness were measured and the surfaces were prepared using implantologists methods to ensure the comparable bonding conditions.

2. Materials and Methods

The experimental studies were carried out in the Science and Technology Park in Opole, using the local strength machine – ElectroForce 5500 equipped with an electromagnetic inductor and operating in the range of 200N [37] (Fig. 2).

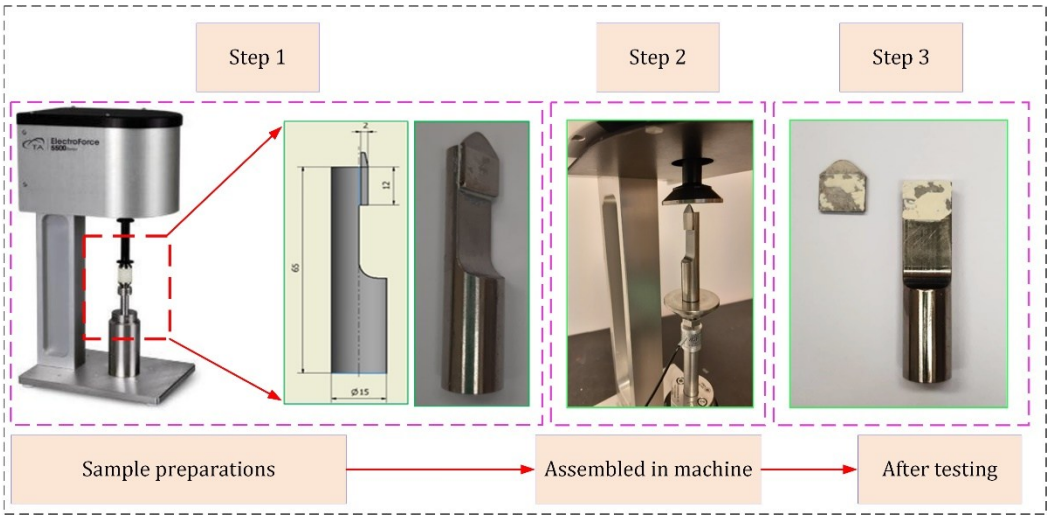


Fig. 2. Experimental procedure adopted in current work

The main material is RelyX Temp NE dental adhesive, also known as composite cement. It is the most commonly used adhesive for setting the prosthetic crowns on the abutments of dental implants, for placing the entire dental bridges and temporary restorations. It can also be used in filling cavities in natural teeth resulting from treated caries [38].

The adhesive consists of two components:

- Base paste
- Catalyst

First of all, composite cements must be biocompatible, but their strength and abrasion resistance are also important. They cannot dissolve in saliva and food liquids, need to have an appropriate plasticity phase [39], and also adhere to both – prepared and unprepared material surfaces [40]. Due to the lack of standards or clear principles of conduct during experimental tests of adhesive connection in dental prosthetics, it was decided to follow the guidelines, rules of conduct and the personal experience of the cooperating dental implantologist. Dosage and adhesive mixing was performed as stated in the operating manual in a 1:1 ratio of base and catalyst. Bonding was carried out at room temperature. The preparation of the adhesive consisted of the following steps:

- Cleaning the surface of specimens on holder and plate with salicylic alcohol
- Pressing the right amount of catalyst and base paste onto the adhesive mixing block included in the package
- Mixing the above two ingredients for 30 seconds using a thin, metal tip
- Transferring the adhesive to the holder, applying and pressing the plate
- After about 5 minutes, the excess glue, which was pressing out by pressure, was scraped off and dried at the periphery of the specimen.

Each specimen was submitted to the same procedure. Four specimens were bonded at one time. The fourth sample was always a spare. After testing, the specimen elements were cleaned and bonded again. The adhesive was the bonding cement for the two metal surfaces made of stainless steel. Such a steel material which the specimen elements are made of, reflects the material that abutments in implant systems and the base of prosthetic crowns are manufactured. Premium stainless steel 1.4305 was selected for the tests, named after the chemical composition X8CrNiS18-9 and with the symbol 303 according to ASTM standards. The chemical composition of the material is given in Table 1. The correct choice of material prevents the development of various types of corrosion between metal elements and the physiological environment in the human mouth [41].

Table 1. Chemical composition of steel 1.403 [42]

composition:	C	Si	Mn	P	S	Cr	Ni	Cu	N
content [%]	0 – 0,1	0 – 1,0	0 – 2,0	0 – 0,045	0,15 – 0,35	17,0 – 19,0	8,0 – 10,0	0 – 1,0	0 – 0,11

Sample ready for testing consisted of three elements:

- one metal holder,
- one metal plate,
- the layer of cement which connected both metal parts.

The figures of the whole assembled and ready for testing sample, its geometry and actual appearance are presented in the Fig. 2.

The metal-to-metal connection was chosen because of the actual reflection of the most popular connection in dental implantology – a glued connection, where one element is a metal abutment of the implant system, and the other is a prosthetic crown with a metal base. The tests were carried out according to the drying time of the adhesive: 1 day, 3 days and 8 days. The samples were tested individually. Specimen assembled in the machine is shown in Fig. 2. The preparation of metal surfaces was carried out in accordance with recognized standards.

The decision was made to carry out fatigue tests with a maximum load of 50 N. Such a load is reflected in the literature, e.g. in [43], where whole polymer blocks were tested for fatigue. The frequency with which the research was conducted was assumed to be at f = 1 Hz. This is the average frequency with which a person grits their teeth during a day.

3. Results

3.1. Fatigue results

Fig. 2 also showed the appearance of an exemplary sample after destruction with cement residues on the sample holder and on the plate. Table 2 presents the results of fatigue tests of the adhesive connection divided into different adhesive drying times. Each drying day obtained different cycle number. Considering the fact that the test frequency was 1 Hz, the cycles number is corresponding the number of seconds that adequate specimen gained.

Table 2. Results

Adhesive drying time	Specimens designation	Cycles
1 day	S1_1	210 029
	S1_2	148 580
	S1_3	175 053
3 days	S3_1	402 922
	~~S3_2~~	~~6 967~~*
	S3_3	247 511
	S3_4	1 000 000
8 days	S8_1	1 000 000
	S8_2	1 000 000
	S8_3	201 050

* error – sample rejected and excluded from further calculations.

The error is immediately noticeable with the result of the sample with the designation S3_2. The test result was excluded from further calculations. The error was caused by a temporary lack of electricity, which resulted in the machine stopping during the test and jamming. An additional test was performed on the adhesive, which had dried for 3 days prior to testing. The result of sample S3_4 also differs from the others, but the result was taken into account in further calculations, due to the specificity and nature of the cement test and the fact that the result was not influenced by any external factors. The results presented in Table 2 show that the drying time of the adhesive affects its durability. The longer the adhesive connection dried without exerting any load on it, the longer the fatigue test took. With a result of 1,000,000 cycles, the machine turned off automatically, assuming that the adhesive connection had achieved unlimited life. Using the Least Squares Method, analogy and a slightly modified formula 1, the single-logarithmic regression equation was calculated for the obtained results, which after the transformations obtained the proposed form:

$$T = A + BX, \quad (1)$$

where:

T – adhesive drying time in days

$X = \lg N_f$ – durability logarithm

Using equation 1 and test results values, a trend line equation was obtain in the form:

$$T = 3.653 - 0.669 \cdot \lg N_f \quad (2)$$

Fig. 3 shows a modified Wöhler fatigue curve based on the adhesive drying time. The chart shows the number of cycles to failure and shows a trend line based on the equation obtained. The single-logarithmic plot was determined on the basis of the least squares method and the modified regression equation (2).

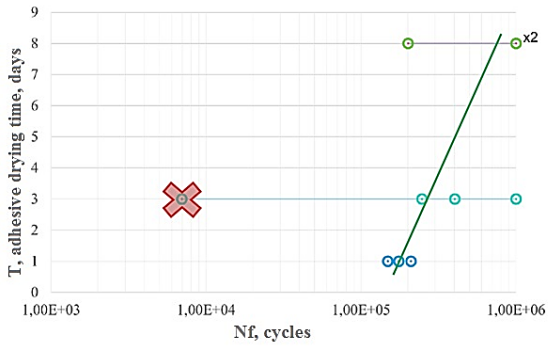


Fig. 3. Modified Wöhler fatigue curve based on the adhesive drying time

The chart shows all points obtained as a result of experimental tests. The point marked with a red cross was not taken into account during the tests, due to the resulting external factors that disturbed the tests (lack of electricity). It was sample S3_2 which was removed from further research and calculations.

3.2. Testing conditions and analysis of software

Due to the changing conditions in the oral cavity, apart from even the influence of saliva, temperature or the influence of food on the teeth, as well as on dental implants, there are variable loads inside over time. They result from the daily work of the oral cavity and the entire stomatognathic system, which, through biting, grinding food, talking or as a result of various occlusal parafunctions, generates variable stresses in biomaterials. Variable stresses directly affect the reduction of the strength of a given material, so we are dealing with the phenomenon of material fatigue. Just as often various types of machine elements are destroyed much faster at lower stresses than their immediate strength, the same structural elements of the implant system may wear out faster due to time-varying stresses than would result from their durability guaranteed by the manufacturer. The factors that directly affect strength are:

- the shape of the element and the action of the notch,
- quality and processing, i.e. surface roughness of the material,
- dimensions of the element, i.e. its thickness.

The sample was placed in the machine, then pressed between the handles to get rid of the effect of the machine's handle (plate) hitting the sample – in this way, the clearances between the machine and the sample were reduced. Impacts on the sample could in themselves cause distortion of the results and disturbance of the tests due to the generated force, vibrations and, ultimately, the adhesive chipping. Before starting the fatigue tests, a simple strength test was performed. It was checked at what size of the maximum load the glued joint will unglue. The same ElectroForce 5500 testing machine and a static tensile shear test were also used for this. In this way, it was possible to properly select the load for fatigue tests. The force at which the joint failed in the static tensile shear test was less than 60 N. On this basis, a decision was made to carry out fatigue tests with a maximum load of 50 N. Such a burden is also reflected in the literature, e.g. in work [43], where whole polymer blocks were tested for fatigue. In order to avoid the upper plate of the machine hitting the sample, the tests were carried out in the range of forces from Fmax = -1 N to Fmin = -50 N. The forces are given with negative values due to the compressive nature of the test. The frequency with which the test was carried out was set at f = 1 Hz. It is the average frequency with which a person clenches his teeth during the day, often used for various studies since ancient times. In the literature, the frequency of 1 Hz is also most often used for testing the elements of the implant system. In the Fig. 4 shows a screenshot of the table with the basic parameters that have been entered into the machine's control and measurement software mentioned earlier in this chapter. In “Conditional Statement” section there is a programmed condition ensuring correct execution of tests. If the force turns out to be higher than -1 N, the machine will turn off and the results will not be taken into account (then there is a possibility of the machine plate hitting the sample and crat-

ing additional disturbances.). Fig. 5 shows an example of a sinusoidal cycle generated during the testing of one of the samples where the time is shown and the load from -1 N to -50 N with displacements. The displacement of the specimen metal plate is about 0,028 mm. Fig. 6 shows an exemplary hysteresis loop generated during the testing of one of the samples, which shows the dependence of the current state of the tested system on the states at earlier moments. It can be said that it is a delay in reaction to an external factor.

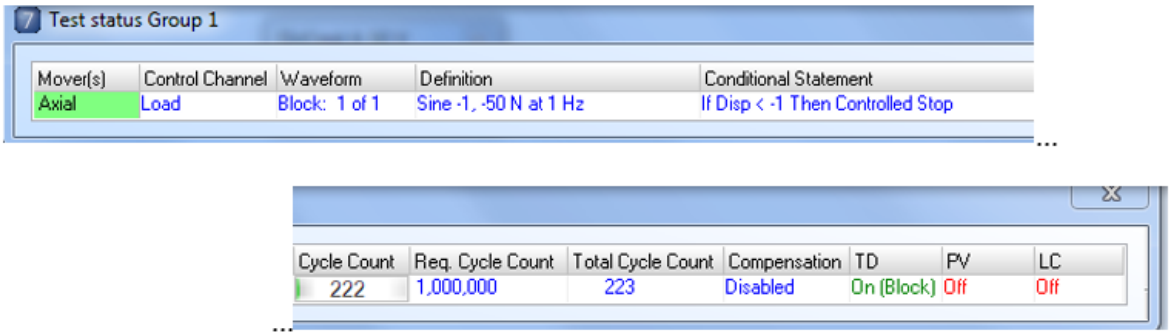


Fig. 4. Dialog window of the testing machine control program with test parameters

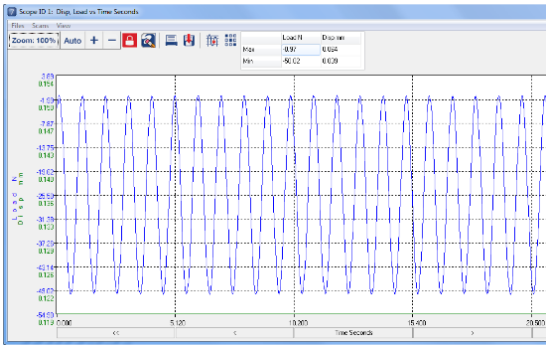


Fig. 5. Part of the dialog window of the testing machine control program showing the course of the sinusoidal cycle

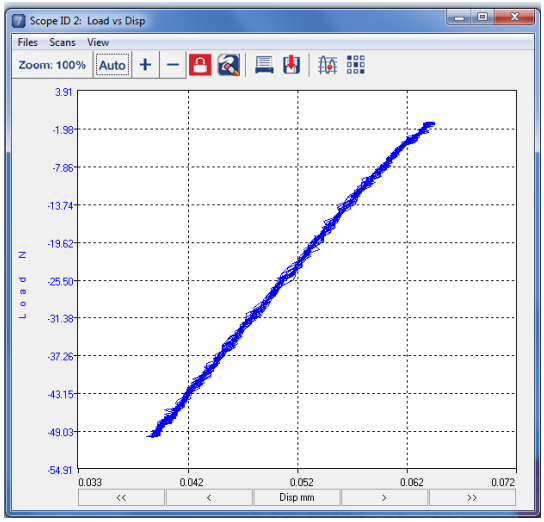


Fig. 6. Dialog window of the machine control program showing the hysteresis loop

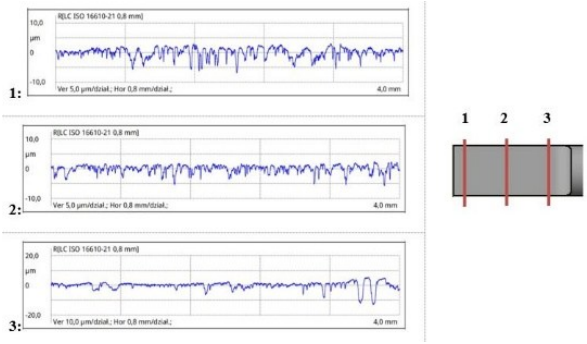
3.3. Abutment roughness analysis

Roughness is a very important factor influencing the implant system fatigue life. Starting with the implant itself – its surface must additionally have the appropriate shape and roughness. This increases the contact area of the implant with the bone, the strength of their connection, and also accelerates the processes of osseointegration. Osteointegration is a direct structural and functional contact between the bone and the implant surface. Roughness is achieved by double acid etching or applying a titanium plasma aerosol. The latter solution is abandoned due to reported complications in patients. Gluing is the joining of at least two components with a thin layer of a joining material that does not affect the parameters of the glued element. Adhesive color is similar to the color of tooth tissues, which increases the aesthetics of the work. They also usually ensure sufficient connection strength with standard crowns and tightness. Adhesive tests are quite problematic due to the behavior of the adhesive itself, which reacts differently in different conditions and on different surfaces, which results in a large dispersion of the obtained results. That's why the metal plates surface have to be prepare properly. The preparation of metal surfaces was carried out in accordance with recognized standards. The abutment is filed with a fairly coarse file to roughen the material, which guarantees a better bond with the dental adhesive. The obtained roughness is not tested and its value is not known for implantologists. Therefore, to reflect reality, the handle and the glued plate were

treated with abrasive cloth with P100 gradation, suitable for metal surfaces. Roughness process was measured in three places of the handle: 1, 2, 3 in the vertical and horizontal direction marked by lines in figures (Fig. 7 and Fig. 8). Examples of roughness courses were measured with the Mar-Surf PS 10 (5667) measuring device equipped with

$$\lg\tau_\alpha = A + m \cdot \lg\bar{X}, \quad (3)$$

PHT 350 cap. The average roughness Ra was 1.8 µm, which is the seventh class of accuracy – rough grinding. The metal surface prepared in this way bonds better with the dental adhesive.



1 day adhesive drying time: $\mathbf{lg\tau_\alpha = 44.78 + 8.7 \cdot lg\overline{N_{f1}}}$, (4)

3 days adhesive drying time: $\mathbf{lg\tau_\alpha = 48.43 + 8.7 \cdot lg\overline{N_{f3}}}$, (5)

8 days adhesive drying time: $\mathbf{lg\tau_\alpha = 49.32 + 8.7 \cdot lg\overline{N_{f8}}}$. (6)

Fig. 7. Roughness courses measured in three places of the handle: 1, 2, 3 in the vertical direction

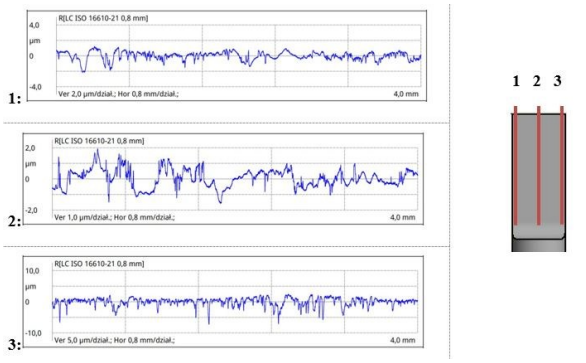


Fig. 8. Roughness courses measured in three places of the handle: 1,2,3 in the horizontal direction

4. Discussion

Three specimens in each group of adhesive drying time are the required minimum. Results are similar to each other or even repetitive, so the number of specimens has been recognized as sufficient. The main factor influencing the number of samples was the long testing time – even about a month for a single sample whose adhesive connection had dried for 8 days. The test frequency was 1 Hz, which strongly influences the duration of the test. In addition, an appropriate hierarchy of the tested samples had to be maintained – the adhesive could not dry for any longer or shorter than the assumed test conditions.

Often in fatigue tests, tests are performed using the influence of various factors, e.g. temperature or size. Using the calculated material factor of m = 8.7 borrowed from [44], where the dental adhesive was tested under variable load, the durability of the adhesive for individual drying days can be compared. For this purpose, a double logarithmic equation (3) is used taking into account the magnitude of the shear stress amplitude and the average number of cycles to failure for each compartment separately:

where:

$\lg\tau\alpha$ – the amplitude of the compression stress logarithm

A – constant value

m – material factor

$\lg\bar{X}$ – mean value of cycles to destroy logarithm.

Using the adhesive connection drying time test results and the formula (3), curves are calculated (4–6) and showed in the Fig. 9. Calculated curves are plotted on a double logarithmic graph described by the amplitude of the compression stress.

The curves in the diagram show the increase in durability of dental adhesive as a result of increasing its drying time.

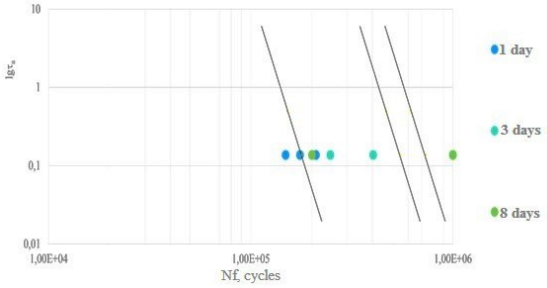


Fig. 9. Curves in double logarithm graph

It can be concluded from the research that the drying time of the adhesive before starting the tests, or in the case of an implantologist's work – before starting further work on a metal abutment – affects its durability. The behavior of the cement under load clearly showed a tendency towards higher durability with the longer drying time taken into account. The adhesive which dried for only one day had the lowest durability among all the tested samples, and the number of cycles to failure did not exceed 210 029, which means a maximum of 2.4 days of continuous operation of

the adhesive connection. In this case, taking into account the fact that, according to scientists' calculations, the average time of a healthy person biting teeth without any occlusal para-function is only 17.5 minutes during one day [45], it can be easily calculated that the adhesive, which withstood 210 029 cycles to failure at 1Hz, has a lifetime of 200 days. This means that a statistically healthy person could enjoy his adhesive connection between a prosthetic crown and a dental implant abutment for 200 days. Assuming a constant maximum load of 50N on this connection for 17.5 minutes a day.

In the case of an adhesive that was allowed to dry and remained for the next 3 days, the durability increased, as shown by the trend line. Excluding the sample with error, the maximum number of cycles to failure during the test was 402 922. This is practically twice as much as for the adhesive with a drying time of 1 day and translates into a durability of over 384 days (with the same assumptions as the previous case). The minimum number of failure cycles for 3 days of glue drying is 247 511, which is comparable to the 1 day drying cement durability. During additional tests, which had to be performed in place of the deleted result with the error, it was possible to obtain a result in which the sample withstood up to 1,000,000 cycles and fell within the unlimited life range. This may suggest that even 3 days of glue drying can significantly increase its durability and may be sufficient for the patient to enjoy his new design of the dental implant even until the end of his days, without worrying about the decementation of the crown. In the case of the adhesive that dried for 8 days, only one sample did not achieve unlimited durability. The adhesive left for over a week can serve the patient for a very long time without the necessity to visit the dentist again and fix the crown. The research showed an increasing tendency for durability, taking into account the longer drying time of the dental adhesive. This means that leaving the prosthetic work for a minimum of 3 days may increase the probability of correct and permanent bonding of the prosthetic crown to the connector of the implant system. Professional practice of implantologists requires refraining from eating and drinking for 2.5 hours after surgery. The research proved that with extreme caution and not moving the prosthetic work for at least 3 days, its durability can be significantly extended.

5. Conclusions

Following conclusions are drawn from current investigations:

- The reconstruction of single teeth with the use of an implantological system is undoubtedly a good method of treatment, giving quick and permanent aesthetic results. Despite the continuous development of implantology, practice shows the necessity to conduct a lot of research and experiments, as well as the existence of many unusual situations. The basic problems relating to implants are those related to their strength and durability, obtaining appropriate models for research and those that appear in everyday dental and implantology practice.
- The tests were carried out in forces from Fmax = -1N to Fmin = -50N to avoid the upper plate of the machine hitting the sample. The frequency was set at f = 1Hz. The abutment roughness were measured with the MarSurf PS 10 (5667) measuring device with the average roughness that was 1.8 μm.
- Experimental studies have shown that with the lengthening of the adhesive drying time, its durability increases. Using the new approach to research with dental adhesive proposed in the this paper, assuming taking into account different days of adhesive drying, it was possible to calculate the trend line.
- The adhesive which had dried for 1 day showed the lowest durability. For specimens where the adhesive dried for 3 days, a minimum durability equal to twice that of the adhesive which dried for 1 day was noted. This means that extending the drying time of the adhesive directly affects its durability.
- Adhesive dried for 8 days withstood a majority of 1,000,000 cycles for and is now within the indefinite life range. Longer drying time of the cement significantly increases the chance of successful implantation treatment.
- Therefore, an extremely important element in the treatment process is the gentle use of the rebuilt system in the first days after its installation.

Ethics declarations

Conflict of interest

The authors have no relevant financial or non-financial interests to disclose.

Ethical approval

This article does not contain any studies with human participants performed by any of the authors.

Research involving human participants and/or animals

This research did not involve human participants and/or animals.

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