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ANALYSIS OF ADHESION CHARACTERISTICS OF LINER DENTAL MATERIALS

Adhesive characteristics of materials used in dental practice are determined by indirect methods, by measuring mechanical properties of liner materials. In that procedure, the adhesion is defined using the measured mechanical properties of the bond material-test sample, which has several shortages. The focus of the present study was based on multi-component composite materials, which have both organic and inorganic components in their structures. The direct measure of material-dentine bond was used in order to quantify the adhesion properties of the investigated liner materials. Artificial saliva was used as the media for inducing the liner-dentine bond destruction. Destruction measurements were made by applying the quantification of visual information methodology. The obtained results were used to calculate the adhesion coefficient of the liner materials. The results were correlated with the mechanical test. There are no references on comparative testing of adhesion mechanical properties of dental material with the presented methodology in the literature. The methodology proved to be useful for the functional quality ranking of dental materials.

Keywords: adhesion, dental materials, bonding, quantification of visual information.

Modern prosthodontics widely uses metal free ceramic restorations, in order to achieve maximal esthetic and biocompatibility. Due to their optical, but also mechanical properties, metal free restorations have to be luted to sound teeth tissues with composite cements, by means of adhesive cementation. Composite cements are widely used for adhesive cementation of inlays, onlays, veneers and full crowns made of indirect composites or full ceramic materials [1].

Adhesion is an indicator of bond quality between the liner and dentine. It is a very important characteristic of any dental material used in the practice. Bonding adhesive resin to the phosphoric acid etched enamel is regarded as a reliable procedure and it was first time introduced in 1955. Relatively hydrophobic adhesive resins can flow into the large conditioned enamel prism (5-7 mm diameter) and sometimes into the submicron sized porosities within the etched enamel prisms. A long-lasting enamel bond can be achieved by means of micro-mechanical interlocking between the resin and enamel. The acid-etch modification of the enamel surface allows the formation of an intimate micromechanical bond between enamel and the resin component of the composite [2].

The composite restoration should, at all times, be considered as an adhesive restoration. The advantages are manifold, but principally the reliance on adhesion rather than retention helps to conserve tooth structure, to improve the strength of the tooth crown and to provide a barrier to marginal leakage. It is therefore important that these materials are used only in situations where a good quality adhesive bond can be achieved.

However, bonding to dentin is far more challenging because dentin is a composite of apatite crystal "fillers" embedded in a collagen matrix. Dentin contains approximately twenty percent protein (mainly collagen) and dentinal tubules, which communicate with the pulp, and contain interstitial fluid. Material bonding to dentin is basically bonding the mineral phase and organic phases of dentin. The most im-

portant factor for the dentin adhesion under clinical conditions is the permeation of resin into the inter-tubular dentin.

This paper presents the development of adhesion quantification methodology for the liner dental materials using combination of:

• accelerated aging and
• quantification of visual information.

Simultaneous measurements of mechanical characteristics of materials were done in order to correlate mechanical properties of materials and obtained adhesion coefficients (Figure 1).
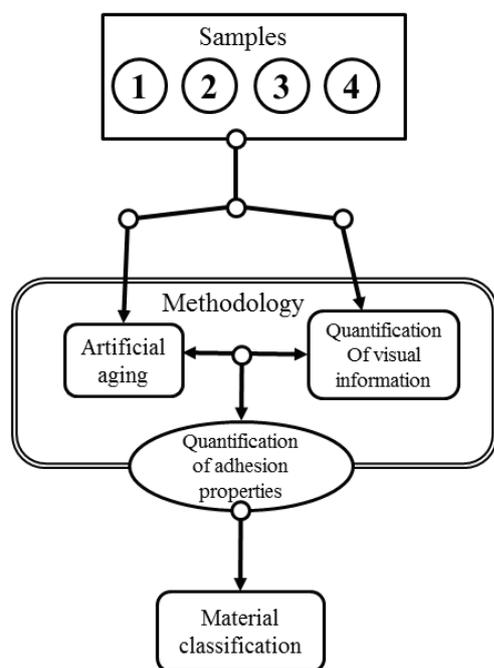

Figure 1. Testing scheme.

Adhesion to tooth substance distinguishes the differences between enamel and dentin. Enamel is composed mostly of hydroxyapatite crystals, containing a small amount of protein and water. Enamel is the most densely calcified tissue of the human body, and is unique in the sense that it is formed extracellular. It is a heterogeneous structure, with mature human enamel consisting of 96% mineral, 1% organic material and 3% water by weight (89%, 2% and 9% by volume, respectively). The mineral phase is made up of millions of tiny crystals of hydroxyapatite $[Ca_{10}(PO_4)_6(OH)_2]$, which are packed tightly together in the form of prisms, held together by an organic matrix. To bond external material to the enamel, the focus must be on the mineral component (hydroxylapatite) of enamel [3].

MATERIALS AND METHODS

Commercial liners Aqua Ionobond Ionoseal, Life and Cegal NV were used as test materials. The materials were tested in extracted teeth with carved Class V cavities. The tested materials were spread into the cavities and exposed to accelerated aging in the artificial saliva solution. Artificial saliva solution was used to provoke destruction of the material-tooth bond. Materials were prepared according to the manufacturers' instructions, and placed into prepared cavities. Six tooth samples were prepared for every used dental liner material. The reference sample was not treated by aging solution, and the others were analysed after 7, 14, 21, 28, and 35 days of treatment with the artificial saliva solution. Samples for visual analysis were prepared by cutting the tooth through the middle of the filling cavity, in the vestibule-oral direction. The sample surface was polished. The level of the material-dentine bond destruction was measured with the quantification of visual information methodology. Measured values of the bond destruction were used for the adhesion coefficient calculation.

The values of the adhesion coefficients enabled calculation of the adhesion gradient during aging. The adhesion gradient is a material characteristic that represents the material property better than the breaking force level of the material-test sample bond.

Bond quality between liner and dentine was measured with quantification of visual information using the OZARIA programme package for quantification of obtained visual data [4]. It is a complete dedicated system (Figure 2), where the microscope is connected to quantification of visual information's assemble, consisting of an image generator (CCD camera), computer and adequate software package.

However, in research practice, the images are frequently used as the presentation of some phenomenon or used solely for illustration. If the images are used for the quantification of some structure or phenomenon, they must be quantified - separated and transformed into image ingredients, objects.

Visual information quantification is a process consisting of several independent procedures. Procedures that are performed in the quantification process of visual information can be presented schematically (Figure 3). The quantification focus is based on the obtained set of visual (image) data. However, the underlying analysis and mathematical apparatus encompasses more general frameworks. Before the beginning of quantification, it is necessary to determine the quantification parameters in order to get the best feedback for analysed property [4]. In the case of

Figure 2. Advanced system for the quantification of visual information - Zeiss Axiom system [3].
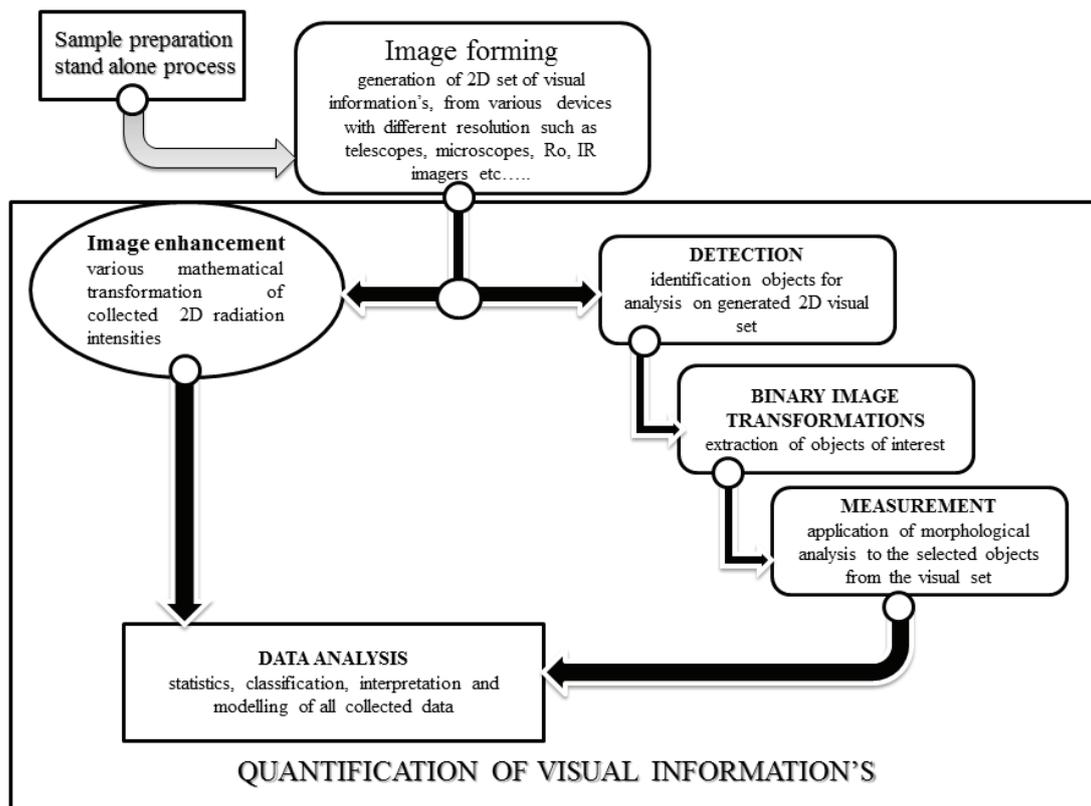

Figure 3. Schematic diagram of the quantification of visual information process.

quantifying dental adhesion, the representative morphological characteristic was the length of bond destruction between liner material and dentine, during time. It was measured on a prepared specimen tooth cross section. Symbolically, the ensemble of such sets of visual data can be presented as:

$$f = f(t, x) \tag{1}$$

where t is the index of the visual set, image, which could be the time stamp when the image was generated, x is the pixel location, and f is the gray level of image t at pixel location x [6].

In order to measure adhesion, and get the quantitative data of dental liner material adhesion pro-

perties, the methodology was developed based on morphology measurement principles. This methodology is based on measurement of the ratio between the length of broken bond and total bond length of the bond tooth-material over time (Figure 4).

The length ratio, L_1/L_0, practically presents the share of a bond broken under the influence of an external agent or due to some defect inside the material. In the case of a material with ideal adhesion, this share is equal to zero, and in the case of material with poor adhesion characteristics, the share is one. It can be presented as:
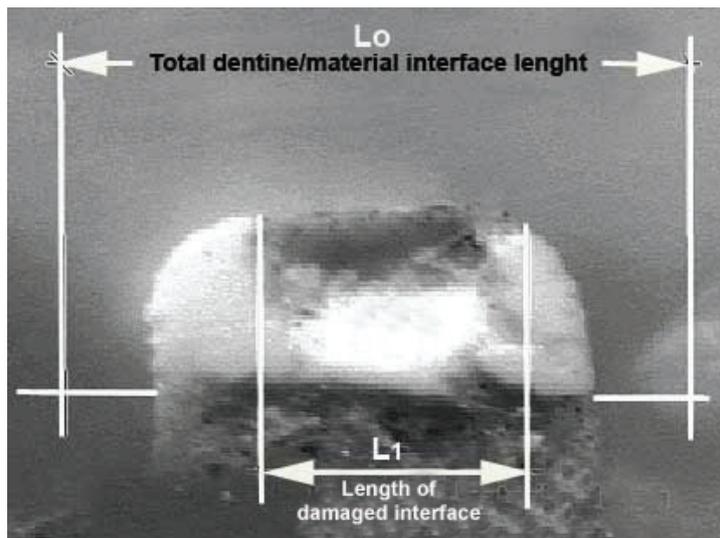
$$K_a = 1 - L_1 / L_0 \tag{2}$$

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Figure 4. Adhesion quantification method.

where K_a is the adhesion coefficient that has value of one (1) for an ideal adhesion material, and value of zero, for a material without adhesion properties. Applying this model, we can obtain the adhesion properties of analysed dental liner materials as the level of exception from ideal material.

Hypothetical changes of the adhesion coefficient with time can provide additional data about the material (Figure 5).

From the analyses of K_a change with time, it is possible to get material-tooth bond destruction gradients, which can be indicators of the material quality for the liners. Inadequate materials will have a high gradient, meaning that the material-tooth bond is more subjected to the destruction. If we standardize

testing conditions, adhesion coefficient, obtained with the presented methodology, could become the significant material quality indicator.

The calculation of the adhesion coefficient is based on the visual analysis of the material surface (Figure 6). According to stereology principles, developed by Saljtnikov [5], it is possible to extrapolate the obtained 2D results to the entire material-tooth volume surface contact.

RESULTS AND DISCUSSION

The ideal liner has to be biocompatible, thermo-isolating and indissoluble in the oral liquids. It is expected to have a mild bacteria-static effect, to have adhesive ability to the dentine and the other resto-
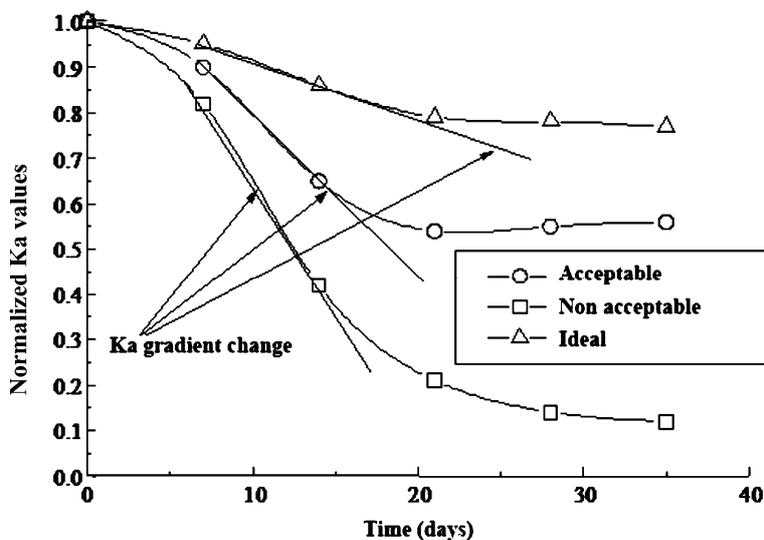

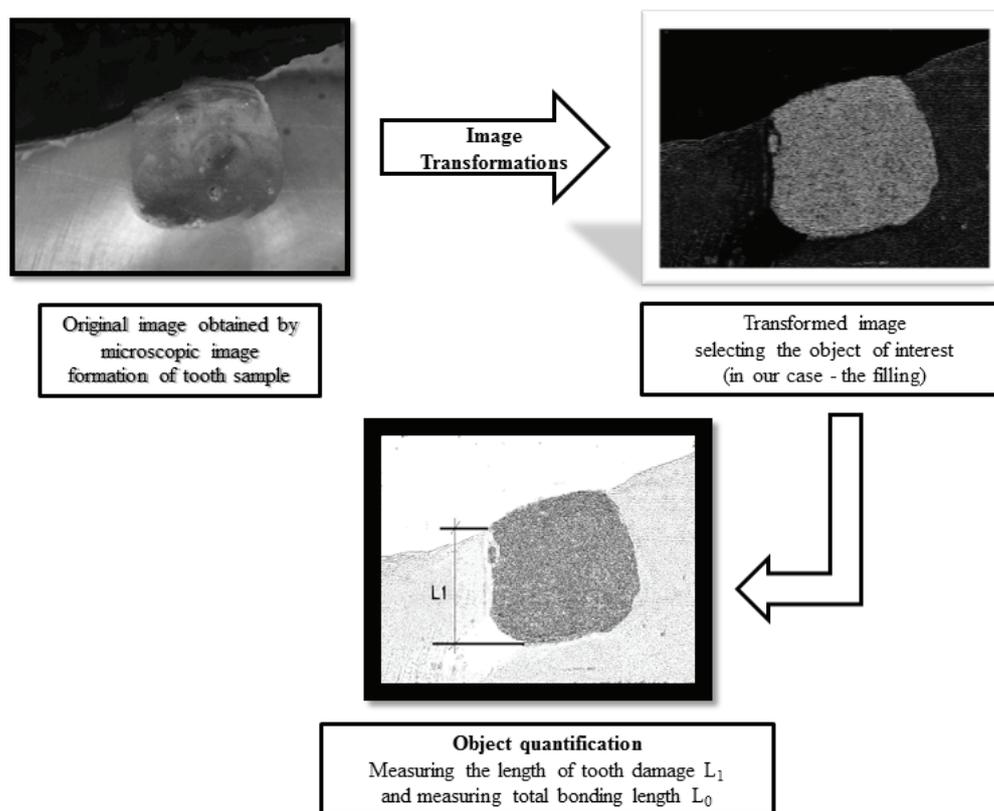Figure 5. Hypothetical adhesion coefficient changes.

Figure 6. Steps of adhesion coefficient measurements (Kₐ = L₁/L₀).

rative materials, and to not interfere with polymerization of the other dental filling materials.

Adhesiveness is a material property that depends upon numerous structural and surface property factors of one material. Adhesion characteristics analysis becomes more complex when dentine characteristics are taken as the variables.

Due to all these facts, material adhesion measurements and quantification are not simple. In most cases, the analogy with measurements of glue quality and using standards for such testing can be used. In the case of glue testing, the sample is placed between two holders, and tied to each of them. The force needed do break the ensemble is measured and considered as the adhesion force of breaking. There are many flaws in this kind of adhesion measurement. First, for testing characteristics of the material-holder bond, the choice of testing speed is an important factor. The second step is choosing the material liner tests. The use of polished tooth surface on one side and a metal cylinder on other side as surfaces for the liner material contacts during testing, according to the standard methodology, does not simulate the real conditions of material use in dental practice.

This was the main reason for developing an alternative methodology for quantification of adhesion properties. The idea was to use the ratio between the damaged bond length and total length of the material-tooth bond during aging. This will measure and quantify the level of destruction of the material-tooth bond, under the effect of the aging agent, in this case, the artificial saliva. The obtained destruction level could be presented as the adhesion coefficient. What are the advantages of this kind of adhesion testing?

First, dental materials are tested in an environment similar to the real mouth conditions. Second, the material adhesive properties are measured directly on the sample, without using indirect measurements. Indirect property determination is not desirable in practice, especially not for the most significant material function characteristics.

As it was described, experiments were performed by combining the quantification visual information and the artificial aging [7]. The results are shown in Figure 7.

The results of these tests are compatible with the results from the data of material mechanical tests, which are reflected in quantifying destruction tendencies of material functional characteristics [7].
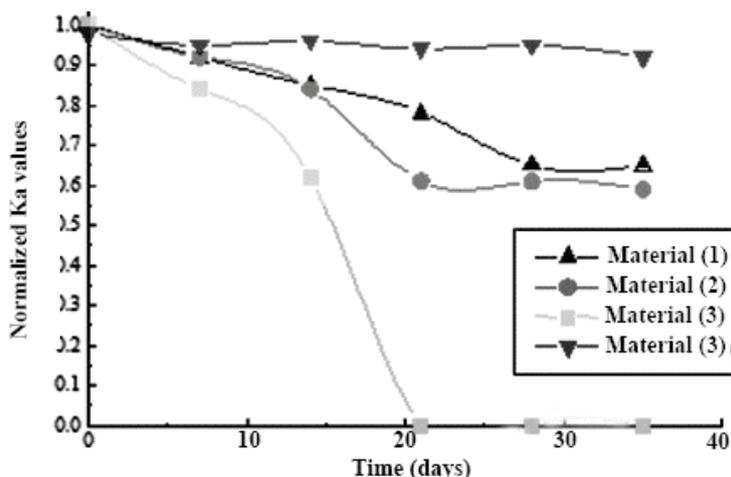
Figure 7. Measurement results of the adhesion coefficient, for tested materials, in total:
1) aqua Ionobond; 2) ionoseal; 3) life; 4) cegal NV.

Life dental liner material shows the largest changes in adhesiveness after 14 days; the same situation happens with mechanical characteristics of the same material. Aqua Ionobond liner and Ionoseal liner show the tendency of adhesiveness decrease after 21 days of treatment with artificial saliva solution.

The presented methodology of material-tooth bond level destruction quantification could be used for determination of material adhesiveness level. The material-dentine bond and the level of bond destruction are measured directly. The adhesion coefficient is a number that is directly related to material adhesiveness level, so it could be used for material comparisons. The method must be standardized in order to eliminate factors that disturb adequate adhesion coefficient determination.

The methodology limiting factors are: material preparation and adequate selection of tooth samples, standardization of the cavity generation, and the preparation of liner material. It is necessary to standardize the testing conditions. In that case, it will be possible to rank all the materials under same conditions.

Figure 8 presents the adhesion coefficient gradient changes of tested dental liner materials. Gradient values show that Life has the highest adhesiveness decline speed. This is an indication that this material passes the highest level of transformations during the testing period of artificial aging.

The results show that adhesiveness can be predicted by measuring material mechanical characteristics (strength, elasticity module), because they have the same tendencies.
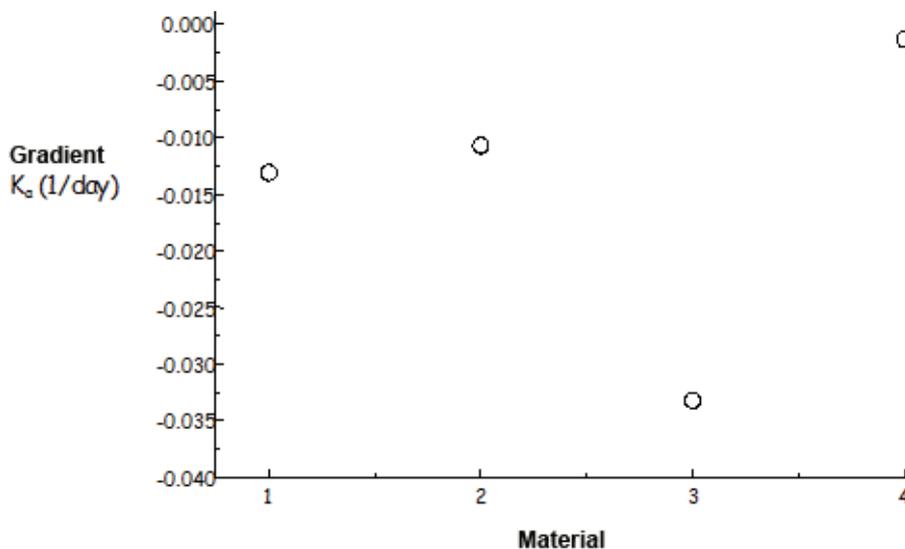

Figure 8. Gradient of change, K_a, for tested materials: 1) aqua Ionobond; 2) ionoseal; 3) life; 4) cegal NV.

The lack of a practical method could conditionally be brought down to the impossibility of defining the "standard tooth sample". Theoretically, every tooth is different, so every adhesion coefficient determination is an individual experiment. However, the results show just the opposite. The compatibleness of tendencies, from the results from mechanical testing and the corresponding adhesion coefficient, prove that this method can be used as an alternative to adhesiveness definition by measuring the breaking force.

As far as the tested materials are concerned, we could analyse results for aqua ionobond ionoseal, life and cegal NV. Aqua ionobond and ionoseal are acceptable from the adhesiveness aspect, but show the tendency of decreasing adhesiveness under the effect of artificial saliva. Cegal NV, of inorganic structure, presented minor destruction changes. The mechanical characteristics of this material presented the same level of changes. The Life liner must be rejected for the designed purposes.

The assumption is that these liner materials interact with some of the saliva's active components and organic material matrix. The interaction is directed towards the destruction of material characteristics, so the adhesiveness decreases. From a practical point of view, the contact of liner materials and saliva must be avoided. Otherwise, the process of material destruction will begin.

The question of the correlations between mechanical characteristics and adhesiveness remains open. In order to check the correlation, regression analysis between the gradient of change K_a and the elasticity module was performed. The results of this analysis are presented in Figure 9. The regression illustrates a linear tendency with a relatively high cor-

relation coefficient ($r^2 = 0.956$). At this research stage, the results indicate the existence of correlation between mechanical characteristics of liner materials and adhesiveness quantified by the presented methodologies.

The methodology applied for material adhesiveness testing can equally be used with the existing method. The results for the tested materials are compatible with literary quotes for new materials [8].

Minor changes of the adhesion coefficient appeared when testing zinc phosphate cement, and major changes in testing liner. Aqua Ionobond and Ionoceal materials did not have significant adhesiveness decrease during exposure to artificial saliva. They resulted only in surface changes, due to material reaction with active saliva components. Major changes were noticed for Life liner material, which practically disappeared after 14 days of treatment with artificial saliva solution. This material can be used for therapy or in combination with other liner materials. Zinc phosphate cement Cegal NV kept almost all of its characteristics during testing.

CONCLUSION

The purpose of research was to analyze the adhesion properties and test methodology using liner materials. The dentine-adhesive interface is very complex and depends not only on the dentine surface parameters, but also on the dental liner properties and the parameters of the applied test [9].

The presented methodologies are not standard in dental practice. Based on the conducted experiments and results, the simulation of conditions in mouth, by the artificial saliva, as the method of acce-
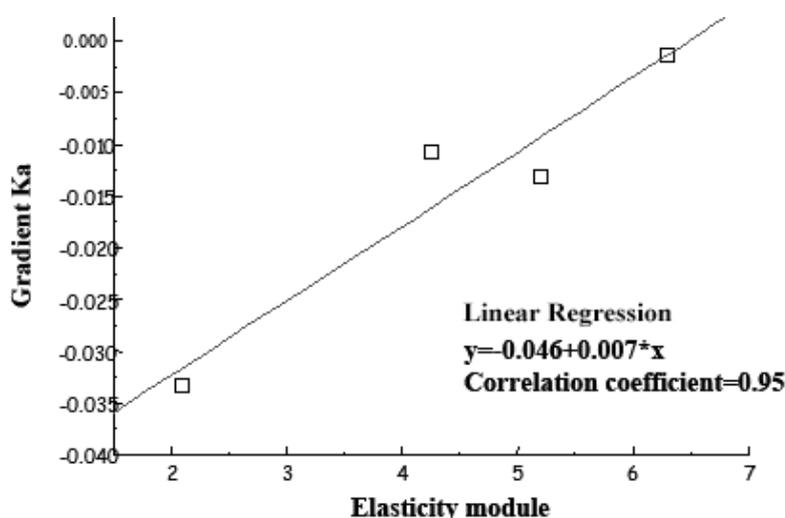

Figure 9. Regression analysis of the adhesiveness measurement results and mechanical characteristics of materials (y - gradient K_a, x - elasticity module).

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lerated aging, was adequate and applicable for testing this type of dental materials. The methodology of determination of adhesion coefficient is compatible with already existing and perennially used methods. The results proved that the adhesion coefficients and gradients of their changes could be used for the material characterization [10].

Transformation from traditional dentistry to modern adhesive dentistry is accompanied by an evolution in concepts as well as in materials, with the occurrence of composites of different viscosities and increasingly numerous new advance methodologies and clinical protocols. The main problem of composite resin fillings lies in the separation of the material from the walls of the cavity following the polymerization shrinkage of the resin matrix, opening the door for the invasion of bacteria and tooth decay. Understanding the interfacial mechanisms associated with the progress made in the field of dentine adherence has helped considerably to improve the reliability of care. The knowledge of these mechanisms helps to determine the choice of a surface treatment, adhesive, bonding agent and an associated adherence accelerator.

Acknowledgements

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NAUČNI RAD

ANALIZA ADHEZIONIH SVOJSTAVA MATERIJALA ZA PODLOGE KORIŠĆENJEM METODOLOGIJE KVANTIOFIKACIJE VIZUELNIH INFORMACIJA

Adhezivna svojstva materijala koji se koriste u stmatološkoj praksi određuju se posrednim merenjima sile kidanja osnove i ispitivanog materijala. Na taj način adhezija se definiše merenjima mehaničkih svojstava veze materijala i test uzorka. Takva metodologija ima nekoliko nedostataka. Prvi je ukoliko se radi o višekomponentnom kompozitnom materijalu koji u svom sastavu ima o organsku i neorgansku kompnentu koje mogu biti različitog agregatnog stanja. Drugi problem je jasna definicija sile kidanja kod ovakvog ispitivanog ansambla. Pretpostavlja se da je sila kidanja sila razdvajanja organske komponenete i osnove. Rad prikazuje istraživanja metodologijom kvantifikacije vizuelnih istraživanja kombinovane sa veštačkim starenjem za određivanje adhezivnih svojstava podloga koje se koriste u stomatološkoj praksi. Dobijeni rezultati su pokazali visok stepen korelacije sa merenjima sile kidanja, odnos klasičnim načinom određivanja adhezivnosti.

Ključne reči: adhezija, stomatološki materijali veza, kvantifikacija vizuelnih informacija.