Comparative *in vitro* Study of the Tensile Bond Strength of Three Orthodontic Bonding Materials

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The bonding materials used in orthodontics must resist to masticatory and active orthodontic forces. The aim of this study is to evaluate and compare the tensile bond strength of three orthodontic bonding procedures: acid etching and chemically cured macrofilled composite (Evicrol, SpofaDental, Jičin, Cehia), glass ionomer cement Ketac Cem (3M ESPE AG, Seefeld, Germany) and self-etching primer with light cure adhesive bond material (Transbond Plus Self Etching Primer + Transbond Plus Color Changer Adhesive, 3M Unitek, Monrovia, California). Moreover, after removing the brackets, the Adhesive Remnant Index (ARI) has also been measured. For this purpose 30 recently extracted human premolars were used, grouped randomly in three groups, 10 teeth for each tested material. After bonding the brackets, these were torn off from the teeth surfaces with a tensile testing machine (Instron 1195). The ultimate tensile strenght (UTS) and the ARI has been measured. Our conclusion was that glass ionomer cement fails first to the tractioning forces, while light cured composite has proven to resist best to streching and traction. Although light-cured composite has failed to lower forces than the chemically cured one, the frequency of ARI score 3 has been the highest, which means that its adhesion is the best.

Key words: tensile bond strength, bonding material, adhesive remnant index

The properties of an adhesive agent used in orthodontics must be understood from the adhesion structural and molecular mechanisms point of view. One of the basic questions of physico-chemistry is: why do materials cohere? The molecular theory affirms that due to the attractive forces in and between different molecules. These forces produce bindings with different resistances (resistance = the energy needed to dissolve the binding) from covalent and ionic to hydrogen and other weaker intermolecular bonds.

The majority of solid materials, including dental tissues and the metallic base of brackets, do not cohere due to the simple touching of their surfaces. A liquid agent has to be introduced between the two solid surfaces facing each other. The fluid waters equally the two surfaces and enters the porosities of the solid material. Therefore, the most important properties of the liquid from this viewpoint are the angel of contact and viscosity [1, 2].

To resist in time all the possible disrupting forces, the fluid has to be transformed into solid phase. This convertion can be phisical (ex: cooling process) or chemical. In case of adhesive materials used in dentistry, chemical convertion takes place: polimerization of monomers for composites and neutralization for cements [2, 3].

Bonding is the most suitable expression to characterize and classify the binding mechanisms. Bonding means adhesion and attachment. These two terms reflect the nature of the binding mechanisms that take place: *adhesion*, the chemical and *attachment* [5], the mechanical mechanism. Adhesion is realized by hydrogenic bindings, London forces and other Van der Waals type bondings. Under attachment is understood when a solid substance which is rigid but porous - the conditioned enamel - makes possible the penetration of the adhesive and the solidification of this „in situ". The bonding between the sole of the bracket and the adhesive is based merely on the mechanical component because of the high retentivity of the bracket base [3-5].

Wetting capacity, penetration and tixotropy are the most important and desired properties of adhesive materials used in orthodontics. It is preferred the bonding time to be short, volumetric contraction small and water absorbtion minimal [6].

The brackets bonded with the adhesive should resist masticatory and orthodontic forces. Therefore durability is a key question, but in the same time after finishing the treatment, the components of the fixed orthodontic appliance should be removed together with the adhesive [7]. At this phase the risk of enamel lesion grows.

The aim of this experimental study is to evaluate and compare the resistance to traction of three adhesive materials: autopolimerising composite (Evicrol), glass ionomer cement (Ketac-Cem) and self-etching primer with fotopolimerising composite (Transbond Plus Self Etching Primer + Transbond Plus Color Changer Adhesive). Moreover, after removing the brackets, we have also measured the Adhesive Remnant Index (ARI).

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Experimental part

We have used 30 newly extracted human upper and lower premolars. After the extraction, they have been deposited in neutral 10% solution of formalin at room temperature for maximum 48 h. The selected premolars have fulfilled the criteria of intact enamel:

- absence of caries
- not have been treated with any deteriorative agent (ex. Hydrogen peroxid)
- no microfissures due to the process of extraction

The teeth have been arranged randomly into three groups each containing 10 premolars.

Before applying the adhesive materials they have been kept in distillated water for 24 h. After this, their surfaces have been prepared with a cleaning paste (not containing fluoride) and a rubber bell for 10 s. On the labial surface of each tooth we bonded a classical orthodontic edgewise metallic brackets (Microarch, GAC, Bohemia, NY) for premolars. Their contact surface equals 11.62 mm^2.

The three groups were the following:

Group I: chemically cured composite (classical) - Evicrol (SpofaDental, Jičin, Czech Republic)

Group II: glass ionomer cement - Ketac Cem (3M ESPE AG, Seefeld, Germany)

Group III: self-etching primer + light cured composite - Transbond Plus Self Etching Primer (3M Unitek, Monrovia, California) + Transbond Plus Color Change Adhesive (3M Unitek, Monrovia, California)

Group I. – Evicrol:

The previously cleaned tooth surfaces have been conditioned with a 37% gel of ortho-phosphoric acid for 30 seconds, after which they have been rinsed with a jet of water for 25 s. Next, they were dried until the area treated with acid became white-chalky. The two components of the composite – liquid and powder - were mixed together respecting all the indications of the manufacturer. The brackets have been fixed to the tooth surface applying a gentle pressure. The excess material has been removed with a buccal spatule.

Group II. - Ketac Cem:

The preparation of the bicomponent manual dosing glass ionomer cement respected strictly the ratio of liquid/powder and the indications of the producer. The next step was attaching the brackets to the buccal tooth surfaces applying a gentle pressure on them. The excess material has been removed before the setting has completely taken place, trying not to displace the bracket and to not interfere with the setting of the material [8, 9].

Group III. - Transbond Plus Self Etching Primer (TP SEP) + Transbond Plus Color Change Adhesive (TP CCA):

The enamel was treated with the self-etching primer for 5 seconds, after which using a light jet of air this was dispersed on the tooth surface forming a thin and uniform layer. The composite material has been applied on the sole of the bracket, which has been attached to the pre-conditioned tooth surface using a gentle pressure. After removal of the excess material fotopolimerization followed for 20 seconds from two opposite directions.

The premolars have been embedded into a matrix of autopolimerized acrilate in such a way that their buccal surfaces were parallel with the floor and the force to be applied to remove the brackets was perpendicular to the base of these (fig. 1). The teeth fixed this way into the acrylate have been fastened into an anchoring key manufactured by us for this purpose (fig. 2). The brackets have been tied with a wipla wire which made the connection with the testing machine. With the help of this machine the brackets were torn off with a constant speed

Fig.1. The embedded premolars

Fig. 2. The anchoring key

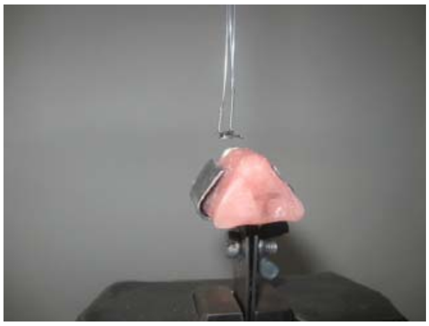

Fig. 3. Tearing off the brackets

from the tooth surface (fig. 3). In fact this is a dynamic test of the adhesive material. The results show the level of the traction force to which the adhesive fails.

After tearing off the brackets, the surface of each premolar has been evaluated with an optic microscope at a 10 times magnification and described conform to the adhesive remnant index (ARI) scores established by *Artun and Bergland* [10]*:*

0 – if remains no adhesive material on the enamel

1 – if the amount of the remnant adhesive material is less than 50% of the area where the bracket has been applied

2 – if the remnant material lies on more than 50% of the surface

3 – if the whole amount of adhesive remained on the enamel

Results and discussions

The results of the measurements can be seen in table 1. After obtaining these data we have calculated the average force of each group (fig. 4). For chemically cured composite this was 123.7 N, for glass ionomer adhesive 33 N and for the self cured composite 74.5 N.

The *ultimate tensile strength (UTS)* (table 2) has been calculated after the following formula:

$$R_m = F_m/S_0$$

R_m – ultimate tensile strength;

Table 1
THE MEASURED FORCES EXPRESSED IN N

Number of measurement	Force applied (N)		
	Evicrol	Ketac Cem	TP SEP + TP CCA
1	185	21	56
2	84	51	86
3	96	50	81
4	130	20	75
5	133	23	89
6	170	30	69
7	90	25	60
8	100	47	77
9	109	36	80
10	140	27	72

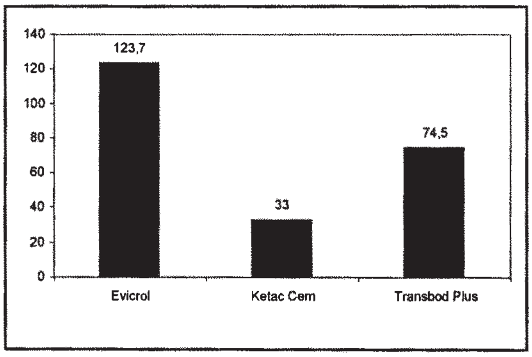

Fig. 4. Graphic representation of the average forces expressed in N

Table 2
ULTIMATE TENSILE STRENGTH EXPRESSED IN MPa

	Results (MPa)	Deviation (MPa)
Evicrol	10.64	± 4.34
Ketac Cem	2.84	± 1.33
TP SEP + TP CCA	6.41	± 1.42

F_m - the tractioning force;
S_0 - the cross section of the analyzed body (in this case the surface of the bracket sole, which is 11.62 mm^2)

Although there are some differences – which might be due to the different circumstances of work and to the fact that only the same class of adhesives have been used not the same commercial products, with the exception of TP SEP + TP CCA - between the measurements obtained by us and those found in the literature, the final results are similar: the highest resistance to stretching and traction has been registered in the case of chemically cured composite and the lowest in the case of glass ionomer cement [11- 13] (table 3, fig. 5).

The evaluation of ARI scores after removing the brackets is important for verifying the amount of adhesive material which remained on the enamel surface. The more the

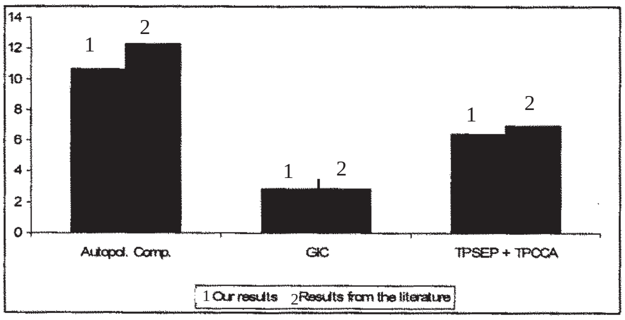

Fig. 5. Graphic representation of the comparison of our results for the ultimate tensile strength measurement with those found in the literature

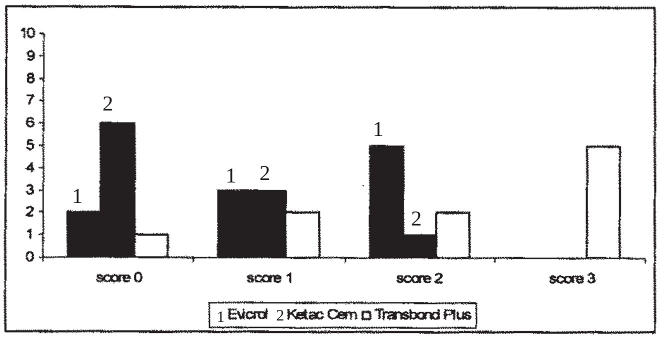

Fig. 6. Graphic representation of the ARI scores

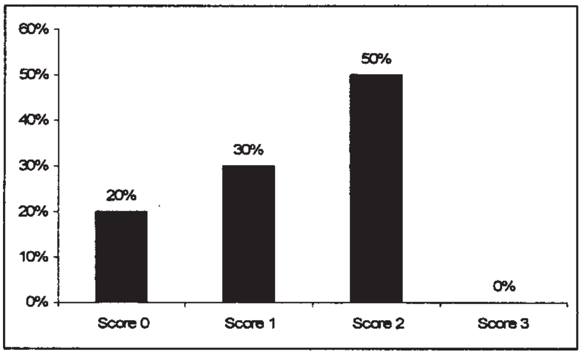

Fig. 7. Graphic representation of the percentage of ARI scores for Evicrol

remnant material is (ARI score 3), the stronger the material's adherence. In this study the majority of fractures – 21 of 30 cases - has taken place at the interference of bracket/adhesive material and in 9 cases no adhesive material has remained on the enamel surface (ARI score 0) (table 4, fig. 6). In case of the chemically cured composite, most of the fractures has taken place at the level of bracket/adhesive interference (fig. 7).

From the three adhesive materials, in case of the glass ionomer cement Ketac Cem has been recorded most frequently scores 0 ARI. This means that this kind of

Table 3
COMPARISON OF OUR RESULTS FOR THE ULTIMATE TENSILE STRENGTH WITH THOSE FOUND IN THE DEPARTMENT LITERATURE

	Evicrol (MPa)	Ketac Cem (MPa)	TPSEP + TP CCA (MPa)
Our results	10.64(±4.34)	2.84 (±1.33)	6.41(±1.42)
Jacob Daub et al. (2005)	12.29 (±3.01)		
Matheus Melo Pithon et al.(2006)		2.84 (±1.18)	
Ascensión Vicente et al. (2009)			6.93 (±3.34)

Table 4
ARI SCORES

	Total nr. of teeth	Score 0	Score 1	Score 2	Score 3
Evicrol	10	2	3	5	0
Ketac Cem	10	6	3	1	0
TPSEP + TPCCA	10	1	2	2	5

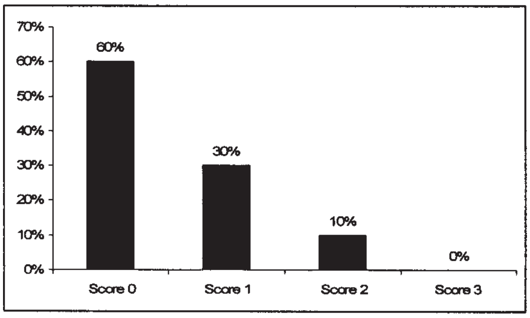

Fig. 8. Graphic representation of the percentage of ARI scores for Ketac Cem

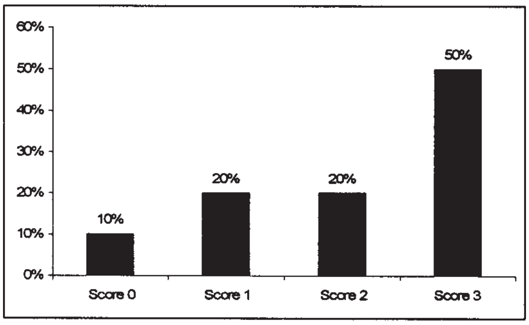

Fig. 9. Graphic representation of the percentage of ARI scores for TP SEP + TP CCA

adhesive material does not assure sufficient bonding to the enamel (fig. 8). The most frequent score 3 ARI has been noticed at the TP SEP + TP CC adhesive system, representing an efficient bonding to the enamel (fig. 9).

Matheus Melo Pithon et al [13] reported that the mean shear bond strength of glass ionomer bonding material without enamel conditioning was significantly lower (2.8 MPa) than the bond strength obtained after etching (11.94 MPa). The mean bond strength without enamel conditioning was substantially lower than the corresponding group in the present study and is below the minimum clinically acceptable bond strength. However the mean shear bond strength of group with enamel conditioning was significantly higher than the group tested in the present study. The evaluation of the ARI score showed that the group with etching had majority of the fractures at the bracket adhesive interface and group without etching had failure in the enamel adhesive interface. In Barshad's (2009) study [14], the bond failure was at bracket adhesive interface in all the groups. These variations may be attributed to the difference in sample teeth, methodology, cross head speed, curing time and attachments used in the study. Some authors [13] used bovine mandibular incisor which were bonded in vitro as compared to the in vivo bonding of premolar bracket in the present study. Bovine teeth are unsuitable for in vitro testing because of variation in enamel configuration compared to human teeth. When comparing debonding forces measured in vivo and in vitro, Pickett et al. [15] found that bond strengths in vivo were significantly lower than those measured in vitro. Most of the studies we found in literature were in vitro studies and further evaluations are needed in order to explain the differences of bracket and bonding behaviour in the two mentioned circumstances.

Conclusions

The use of autopolimerizing composite requires a lot of attention; the process consists of more steps which must be strictly respected. The chemically cured composite materials have proven to be the most efficient adhesives from the resistance to stretching, traction and compression point of view, altogether their resistance to the masticatory and orthodontic forces is the highest.

From our comparative study results that glass ionomer cements are the most inefficient adhesive materials. For this stands the fact that in 6 cases out of 10, the ARI score was 0 and there has not been any ARI score 3 cases.

Handeling the light cured composite is simple. The process of conditioning confers a high level of adhesion of the material to the enamel surface. The combination of self-etching primer and light cured composite has proven to be efficient in the long term attaching of brackets. Although the light cured composite has failed to lower forces than those measured in case of the chemically cured one, the frequency of ARI score 3 has been the highest: 5 cases out of 10.

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