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Final report of scientific *in vitro*-study:

Retentive strength of different cements in all ceramic zirconium crowns after long term water storage.

Introduction:

Tooth-colored, all-ceramic crowns have become the main interest in prosthetic dentistry for the restoration of single teeth in recent years due to their highly aesthetic appearance and metal-free structure.

Zirconium oxide is today's focus due to its optimal material properties, which makes this material even suitable for posterior-fixed partial dentures. According to its high fracture resistance, conventional cementation is possible in most cases. Of course, conventional cementation is limited to preparations showing sufficient friction-surfaces. The cement or luting material seals the remaining gap between the tooth's hard tissue and a fixed prosthesis and provides certain retention to the restoration.

The retentive strength of a conventional cement mainly comes from its physical strength and a kind of microretention of its filler particles, entangled with the rough surface of the prepared tooth, and not from an adhesive phenomenon. While in cast fixed prosthodontics the main retentive strength comes from friction, in all-ceramic restorations – due to the minor influence of friction – the adhesive interface is the determinant factor in the retention. Therefore, resin cements need to be bound adhesively in order to establish a tight interface to the tooth structure. These adhesive systems are mainly based on phosphoric acid etching or self etching systems. Despite the fact that adhesive cementation provides the best marginal seal of all-ceramic crowns, the possibilities of an adhesive cementation are limited to those cases where sufficient contamination control is possible to obtain

adequate adhesion. In the clinical situation, a combination of both friction and adhesive strength will result in the overall retentive strength of even an adhesively fixed all-ceramic restoration. Determining factors therefore are the retention surface, the convergence-angle and the physical properties of the luting agent.

Bond strength to ceramic is influenced by the polymerization mode of the luting resin cement (VLC-, dual, or dark curing) as well as the determination of bond strength after thermocycling and water storage, and of course the luting cement itself.

To determine these bond strength data, shear bond strength tests are mostly used for the determination of bond strength but do not represent the clinical reality due to multiple variables influencing the performance of the different materials such as C-factor and film thickness.

Therefore, to determine a clinically relevant statement on retentive strength, the retentive strength has to be investigated by means of a pull-off test of real crowns from extracted human teeth. This testing procedure is very complex and technique-sensitive, but is suitable for delivering true information on the retentive performance of a material.

Aim of the study

The aim of the present study was to determine the retentive strength of zirconium all-ceramic crowns after cementation using self adhesive resin cement systems.

In contrast to an already performed study (Ernst, C.-P., Cohnen, U., Stender, E., Willershausen, B.: In vitro retentive strength of zirconium oxide ceramic crowns using different luting agents. J Prosthet Dent 93 (6), 551-558 (2005)), a more pronounced preparation-angle was considered in this study, as already published in a different research (Ernst, C.-P., Aksoy, E., Stender, E., Willershausen, B.: Influence of different luting concepts on long term retentive strength of zirconia crowns. Am J Dent 22 (2) 122-128 (2009)).

Due to the fact, that longevity of the retention is a main concern in prosthetic dentistry, long term water storage will be considered in the new study design as well.

Material and Methods

Tooth preparation

240 freshly extracted caries-free teeth were stored in a 1% Chloramine-B-hydrate solution for a week and kept wet afterwards using tap water. No interaction with restorative materials or with the tooth's hard tissue when using Chloramine-B-hydrate has yet been reported yet.

The teeth were randomly distributed into 8 groups of 20 teeth each. Undercuts were prepared in the teeth's roots. The teeth were then embedded in film capsules of 24 × 36 mm slide films and fixed with Paladur PMMA resin (Heraeus Kulzer, Hanau, Germany).

A standardized 20° conus preparation ($\alpha/2 = 10^\circ$) was carried out (all the preparations within dentin, no core-reconstruction) in a customary manner by means of a dental lab Degussa F1 parallel milling machine (Degussa, Hanau, Germany) to ensure exactly the same preparation angle for each

individual tooth. To obtain this preparation-angle, custom made diamond preparation instruments were constructed and manufactured by Komet/Brasseler, Lemgo, Germany.

The instruments used were 125 µm and 30 µm torpedo-shaped as well as 125 µm and 30 µm conically-shaped diamond burs. All the resulting stumps had the same height (3 mm) and a flat top surface. New preparation-diamonds were used for each group of 10 teeth. The determination of the tooth preparation surface was carried out according to the method described by Dahl und Oilo (*Dahl BL, Oilo G. Retentive properties of luting cements: an in vitro investigation. Dent Mater 1986;2:17-20*) by correlating the weight of a tin foil wrapped around the prepared surface to a standardized square-cm piece of tin foil.

After preparation, the teeth were again stored in tap water. Polyether-impressions (Impregum Penta Soft, 3M ESPE, Seefeld, Germany) of all teeth were taken and conventional type stone models (Fujirock, GC, Japan) were made.

LAVA (3M ESPE) all-ceramic- crowns (caps) for all the teeth were manufactured in a milling centre (Geiger-Dentaltechnik GmbH, Mrs. Brunner, Gottlieb-Daimler-Str. 3, 73529 Schwäbisch Gmünd, Germany). The Lava caps were constructed with outer macro-retention bars to obtain retention in a low shrinkage resin material.

The cements to be investigated were:

1. Panavia 21 (Kuraray)
2. RelyX Unicem Aplicap (3M ESPE)
3. RelyX Unicem Clicker (3M ESPE)
4. SFCem 44 (now marketed as RelyX Unicem 2 Automix) (3M ESPE)
5. Ketac Cem (3M ESPE)
6. Zinc phosphate cement (Hofmanns)
7. Meron Plus (VOCO)
8. Maxcem Elite (Kerr Hawe)
9. iCEM (Heraeus)
10. QuickCem (now marketed as SpeedCem) (Ivoclar)
11. Bifix SE (VOCO)
12. SECure (Sun Medical)

All LAVA caps were sand-blasted (Rocatec Pre, 110 µm, 3M ESPE).

In the SeCure group, the tooth's surface was pretreated with the SeCure Primer and an additional application of Porcelain Liner M was done onto the LAVA-crowns luting surface.

No additional disinfection was carried out to avoid a potential interaction with the cements. All stumps were cleaned by means of a prophylaxis paste (Zirkate-Prophy-Paste, Dentsply). All teeth had to be brought to 37°C prior to cementation!

All systems were used only in their dark-curing mode, which means without any kind of additional light exposure. This was to simulate the clinical situation where light is not able to pass through the LAVA-caps.

The cementation of the investigated materials followed strictly manufacturers' recommendations. Particular attention had to be paid on the in vitro cementation, which in some cases causes more trouble and misunderstanding than a clinical cementation process. Each manufacturer provided information on the setting time the material needed prior it will have contact to water.

Cementation was performed by finger pressure (3 minutes).

No glycerine gel was applied on:

- Ketac Cem (3M ESPE)
- Zinc phosphate cement
- Meron Plus (VOCO)

A glycerine gel (Airblock/DENTSPLY) was applied on those materials:

- Maxcem Elite (Kerr Hawe)
- RelyX Unicem Clicker (3M ESPE)
- RelyX Unicem Aplicap (3M ESPE)
- SFCem 44 (3M ESPE)
- iCEM (Heraeus)
- QuickCem (Ivoclar)
- SECure

With Panavia 21, the Oxyguard II was used instead.

The remainders of luting material were removed prior to setting of the cement with a foam-pet.

After complete setting of the cements (30 min) at 37°C temperature and 100 % humidity (Tupper ware with wet towel), the teeth were checked for further excess of cement. If this was detected, it was removed by hand instruments at this stage.

Afterwards, the teeth were stored in phosphate buffered tap water at 37 °C for one week, followed by thermocycling (5.000 ×, 5-55 °C, dwelling time 30 s in each bath).

After thermocycling, the 20 teeth per group were randomly divided into two sub-groups: One group underwent the debonding procedure immediately; the other group was stored for one year in tap water (weekly change of the water) and was pulled off then.

Debonding procedure:

Prior to the debonding procedure, the distraction unit with an inserted screw base had to be fixed axially on the cemented LAVA-cap. To ensure the binding to the specially designed low shrinkage

