Year: 2010

Impact of in-vitro aging on mechanical and optical properties of veneering composites

Egli, R E

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Originally published at:
Egli, R E. Impact of in-vitro aging on mechanical and optical properties of veneering composites. 2010, University of Zurich, Faculty of Medicine.

Universität Zürich
Zentrum für Zahn-, Mund- und Kieferheilkunde
Vorsteher: Prof. Dr. med. dent. C.H.F Hämmerle

Klinik für Kronen- und Brückenprothetik,
Teilprothetik und zahnärztliche Materialkunde
Direktor: Prof. Dr. med. dent. C.H.F. Hämmerle

Arbeit unter der Leitung von Dipl. Ing. B. Stawarczyk, MSc

Impact of in-vitro aging on mechanical and optical properties of veneering composites

INAUGURAL-DISSERTATION
zur Erlangung der Doktorwürde der Zahnmedizin
der Medizinischen Fakultät der Universität Zürich

vorgelegt von
Roger Elmar Egli
von Alt St. Johann SG

Genehmigt auf Antrag von Prof. Dr. med. dent. C.H.F. Hämmerle

Zürich 2010

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1. Abstract

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negative). **Conclusion:** The veneering composite Sinfony showed the most stable tested properties.

2. Introduction

A fixed-dental prosthesis must be veneered from aesthetic point of view. Currently, two types of veneering of alloy frameworks are in use, either veneering with ceramic or with composite.

Veneering with ceramic is an established technology and has been used for more than 50 years in fixed-dental prosthesis for the anterior and posterior regions of the mouth with very good success rate (1). Depending on the manufacturer, the flexural strength of the veneering ceramic ranges between 55 and 150 MPa (2). Veneering ceramics stand out with high biocompatibility, high color stability (3), and high abrasion stability (4). However, veneering ceramic restorations have numerous undesirable characteristics, such as that the fabrication is time-consuming and technically demanding, and is abrasive for opposing natural tooth structure (5). Despite their reported superior mechanical properties of ceramic, limited number of clinical studies reported chipping of veneering ceramic, in particular in combination with zirconia restorations (6-11).

Therefore, new materials for veneering were developed, the so called laboratory composites of the second-generation. The composition of these indirect veneering composites systems is similar to that of direct composites, differing by the method of additional polymerization (12). The veneering composite is layered in thin layers and each layer is then cured with special devices using light-polymerization as well as heat curing, both in combinations with pressure/vacuum and/or nitrogen atmosphere. The mechanical and physical properties of veneering composites are based on the chemical composition: resin matrix composition (13, 14), filler particles type (13), filler size (15), filler percentage (15, 16), and filler-matrix bonding (silane coupling agent) (16, 17). The following parameters are important during the polymerization process: temperature (17, 18), durability (16), environmental conditions (19), and light intensity of polymerization unit (14). In addition, longer light exposure, secondary radiation, and post-curing by heating have been found to improve the properties of prosthetic composite materials in laboratory procedures (20-22). It has been shown that the failure probability of composite-veneered restorations was not significantly different from metal-ceramic restorations (23). However, some problems have been reported with veneering composite, such as different flexural strength reduction with aging depending on the material type (24), mutated veneer surface texture (25), and a tendency for discoloration (26).

Based on further technical development, the polymerization process could be improved with an increased conversion rate resulting in fewer unpolymerized monomers in the patient's mouth (18). The increased practice of veneering technique with indirect composites is due to improvement in the properties of composite materials in the last years (25, 27). These new composites have a volume percentage of inorganic ceramic fillers of approximately 66% which result into improved mechanical properties with a flexural strength between 120 and 160 MPa and an elastic modulus of 8.5-12 GPa (12). Most veneering composites are applied with a post-curing process that result in superior flexural strength compared to veneering ceramic, minimal polymerization shrinkage, and wear rate comparable to tooth enamel (28, 29). So far, chipping of veneering composite and long-term clinical experience have not been observed in clinical studies. It could be possible to substitute veneering ceramic with veneering composites for single crowns and multiple-unit posterior bridges, due to their similar mechanical properties. Moreover, it could be an indication of veneering composite for zirconia frameworks.

Under oral conditions, restorative materials are subjected to mechanical, chemical and thermal influences through eating, drinking and breathing. The aim of this study was to test and compare the impact of aging on three indirect veneering composites with different compositions aged in water (37°C) and with thermocycling (5°C/55°C) up to 180 days on the flexural strength, hardness, roughness and color stability. Furthermore, the abrasions stability was determined after chewing simulation up to 1.2 Mio cycles.

3. Materials and methods

Three different light curing veneering composites with different composition were tested on long-term stability: GC Gradia (GRD), VITA VM LC (VVL) and 3M ESPE Sinfony (SFN). Each veneering composite was polymerized following manufacturer's instructions in a different polymerisation unit (Table 1).

3.1 Flexural strength

3.1.1 Specimen preparation

For each tested veneering composites, 165 specimens (25x2x2 mm) were inserted in a special stainless steel mould according to DIN EN ISO 10477:2004 in bulk (30). The mould was placed over a glass plate and the composites were inserted with a spatula. A glass plate was pressed over the inserted veneering composite to extrude excess material and polymerized. Then the specimens were separated from the mould while excesses were carefully removed with silicon carbide polishing paper (SIC P400, SCAN-DIA, Hagen, Germany).

The initial flexure strength of all three veneering composites (n = 15 per group) was measured. The remaining 150 specimens of each veneering composite were randomly divided into two subgroups: (1) 75 specimens were stored in distilled water at 37 °C in the incubator (ED 240, Binder, Tuttlingen, Germany) and (2) 75 specimens were placed in the thermocycling machine (5 °C/55 °C/dwell time: 20s; Thermocycler, Willytec, Feldkirchen-Westerham, Germany). In both subgroups, at each time point 15 specimens were selected after 1, 7, 28, 90 and 180 days for flexural strength measurements.

3.1.2 Flexural strength measurement

The flexural strength was measured with three-point bending test using the universal testing machine (Z010, Zwick, Ulm, Germany). The specimens were placed on two rollers with a diameter of 2 mm and set at a distance of 20 mm. The specimens were loaded axial from above with a stamp (diameter of 2 mm). The load with a cross head speed of 1 mm/min was applied until the specimens were destroyed. The flexural strength was calculated with the following formula:

$$\sigma = \frac{3Fl}{2wh^2}$$

σ: flexural strength, F: highest applied force (N), l: distance between the rollers (mm), w: width and h: height of the specimens (mm)

3.2 Martens Hardness

3.2.1 Specimen preparation

The production of the specimens (n=10 per composite) was the same as described above. The surface of the specimens were first polished to SIC P400 and then up to P1200 with an automatic polishing device (PlanoPol-2, Struers, Ballerup, Denmark) for 60 s.

The specimens of each veneering composite were randomly divided in two subgroups: (1) 5 specimens were stored in distilled water at 37 °C in the incubator (ED 240, Binder, Tuttlingen, Germany) and (2) 5 specimens were placed in the thermocycling machine (5°C/55°C/dwell time: 20s; Thermocycler, Willytec, Feldkirchen-Westerham, Germany).

3.2.2 Martens hardness measurement

Every specimen was measured before aging and after 1, 7, 28, 90 and 180 days for Martens hardness (ZHU 2.5, Zwick, Ulm, Germany) pressed with a diamond indentor with a load of 10 N for 20 s on the surface of the specimen.

3.3 Surface roughness

3.3.1 Specimen preparation

For each veneering composite 10 round specimens (radius: 5 mm, thickness: 2 mm) were fabricated in a special silicon mould. The veneering composites were directly inserted and polymerized. The specimen surface was first polished to SIC P400 and then to P1200 with an automatic polishing device (PlanoPol-2, Struers, Ballerup, Denmark) for 60 s. The 10 specimens of each veneering composite group were divided in two subgroups: (1) 5 specimens were stored in distilled water at 37 °C in the incubator (ED 240, Binder, Tuttlingen, Germany) and (2) 5 specimens were placed in the thermocycling machine (5 °C/55 °C/dwell time: 20s; Thermocycler, Willytec, Feldkirchen-Westerham, Germany).

3.3.2 Surface roughness measurement

The surface roughness of each specimen was determined with a surface measuring unit (Perthometer S2 with feed unit GD25, Mahr GmbH, Göttingen, Germany). Each specimen was measured five times with a measuring track of 1.75 mm. The distance between the tracks was 0.25 mm. Both subgroups were measured before aging and after 1, 7, 28, 90 and 180 days.

3.4 Discoloration

3.4.1 Specimen preparation

Specimens were prepared (N=90, n=30 per veneering composite) with a diameter of 15 mm and a thickness of 1 mm, following the ISO 4049 specification (31). A plastic split ring that rested on a glass stab was used to fabricate the specimens. Veneering composite was filled into the split ring between two glass stabs and polymerized and then embedded with acrylic resin (ScandiQuick, SCAN-DIA, Hagen, Germany) in cylindrical molding cups with a diameter of 25 mm (UnoForm, Struers, Ballerup, Denmark). The surface of all specimens was uniformly polished (P400, P1200, P2400) on a polishing device (LaboPol-21, Struers) and examined under a light microscope (25x, Wild M3B, Heerbrugg, Switzerland).

3.4.2 Specimen aging

The 30 specimens of each veneering composite were randomly divided in three subgroups: (1) 10 specimens were stored in coffee (Mastro Lorenzo Classico, Kraft Foods, Glattpark, Switzerland) at 37 °C, (2) 10 specimens were stored in black tea (Lipton Yellow Label, Unilever GmbH, Thayngen, Switzerland) at 37 °C, and (3) 10 specimens were stored in red wine (Rioja, Spain) at 37 °C.

3.4.3 Discoloration measurement

The discoloration of each specimen was measured before aging and after 1, 7, 28, 90 and 180 days. The initial measurement of each group was used as reference point. The measurements were performed with a calibrated (white standard SRS-99-010-7698-a) spectrophotometer (CM-508d, Minolta, Tokyo, Japan). All specimens were scanned from 400 to 700 nm with a data interval of 1 nm at a speed of 480 nm/min. The resulting parameters (L, h, a, b) were examined by a 2° standard observer and by illuminant D65 by the software (SpectraMagic, Minolta, Tokyo, Japan) and then the ΔE-value was calculated with the following formula:

$$\Delta E = \sqrt{\Delta a^2 + \Delta b^2 + \Delta L^2}.$$

After 180 days of storage in coffee, tea and red wine, all specimens were polished for 60 s with a prophylaxis paste for cleaning (Cleanic, KerrHawe SA, Bioggio, Switzerland) and the discoloration was measured.

3.5 Abrasion stability

3.5.1 Specimens preparation

Specimens of each veneering composite were made in a special calibrated stainless steel mould (N=18, n=6 per each group) and polymerized. The specimens were polished with silicon carbide polishing paper P400, P1200, P2400 (LaboPol-21, Struers, Ballerup, Denmark).

3.5.2 Chewing simulation

All specimens were aged in a computer-controlled chewing machine (self-construction of the University of Zurich). Thermo-mechanical loading was applied during cycling loading; an occlusal loading of 50 N at 1.7 Hz and simultaneous thermal stress with temperature changes every 120 s from 5 °C and 50 °C. Palatinal cups from nearly identical upper human molars fixed in amalgam (Dispersalloy, Dentsply, Konstanz, Germany) acted as antagonists.

3.5.3 Abrasion stability measurement

The profiles of the specimens were measured with a 3D wear-measuring device (self-construction of the University Zurich) before aging and after 120 000, 240 000, 640 000 and 1 200 000 cycles. The custom-made surface analyzer consisted of a computer, connected with a surveyor's table, which was moved in 1 µm steps in x, y and z axis by three stepper motors, controlled by a touch-switch caliper-needle. As antagonistic material messiobuccal cups of maxillary 1st molars was used. An area of 9 mm² was examined in each specimen. Veneering composite loss of each specimen was calculated with the 3DS software by overlaying the profiles with congruent points and subtracting initial measurements from subsequent measurements.

3.6 Statistical analysis

Descriptive statistics [means, standard deviations (SD) and 95 % confidence intervals (95 % CI)] were computed. In order to detect differences of the flexure strength means, one-way ANOVA together with Scheffé post hoc test was applied.

For hardness, surface roughness, discoloration, and for abrasion stability measurements, linear mixed models with random intercept were applied in order to investigate the influence of the different aging levels. Akaike information criterion (AIC) and Bayesian information criterion (BIC) were used for model choice.

The data set was coded in Excel and analysed with SPSS Version 17 (SPSS INC, Chicago, IL, USA). Results of the analysis with p-values smaller than 5% were considered to be statistically significant.

In summary, a subjective assessment of all measured properties was made to give an outline of the aging stability for the three different composites.

4. Results

4.1 Flexural strength

Table 2 provides descriptive statistic of flexural strength measurement (mean, SD and 95% CI) of both aging types for each veneering composite and aging level.

4.1.1 Flexural strength of the three veneering composites at specific aging level

Water storage (37 °C)

No statistical differences between the veneering composites were observed after one day ($p=0.295$) and seven days ($p=0.085$) aging (Table 2). After 28 days ($p<0.001$), the highest flexural strength was obtained for GRD and SFN, both veneering composites showed better values than VVL. After 90 days ($p=0.001$), GRD presented significantly lower values than SFN and 180 days ($p<0.001$), SFN showed significantly higher flexural strength compared to GRD and VVL.

Thermocycling (5 °C/55 °C)

After 1 day thermocycling ($p=0.026$), GRD showed significantly higher flexural strength than VVL and after 7 days ($p<0.001$) the highest flexural strength was obtained from VVL and SFN (Table 2). Among 28 days of thermocycling ($p=0.026$), the results for SFN were significantly higher than VVL. After 90 days ($p<0.001$), GRD revealed the lowest mean flexural strength and SFN was higher than VVL. VVL had significantly higher results compared to SFN after 180 days ($p=0.040$).

4.1.2 Long-term flexural strength stability for each veneering composite

Water storage (37°C)

Among GRD (p<0.001), the highest flexural strength results were observed after 1 day and 7 days of water storage (Table 2). The lowest results were observed from initial and 180 days of aging. VVL (p<0.001) showed initially significantly higher values than after 7, 28 and 180 days. In the last aging level (180 days) flexural strength showed the lowest results. Within SFN (p<0.001), after 1 day a significantly higher flexural strength was observed compared with initial, 7 and 180 days of water aging. The specimens aged for 180 days showed significantly lower values than after 28 days and 90 days.

Thermocycling (5 °C/55 °C)

GRD (p<0.001) showed initially and after 90 and 180 days of thermocycling significantly lower flexural strength compared to aging after 1, 7 and 28 days aging (Table 2). In VVL (p<0.001), the lowest results were observed after 180 days of aging. The highest flexural strength was observed initially. Within SFN (p<0.001) the specimens showed the lowest results after 180 days of aging. Significantly higher flexural strength ware obtained after 7, 28 and 90 days aging being different from those of 1 day.

4.2 Martens hardness

Water storage (37°C)

As far as the initial mean Martens hardness is considered VVL showed higher Martens hardness than SFN (p=0.004) and GRD. All three veneering composites showed an increase of Martens hardness during water storage (p=0.016) which was not significant between the composites (between SFN and GRD: p=0.159, between SFN and VVL: p=0.126) (Table 3, 4).

Thermocycling (5°C/55°C)

The veneering composites showed no differences in the initial mean Martens hardness values level (between SFN and GRD: p=0.060, between SFN and VVL: p=0.075). All veneering composites showed an increase in Martens hardness during aging (p=0.001). VVL (p=0.045) showed a lower increase than SFN and GRD (Table 3, 4).

4.3 Surface roughness

Water storage (37°C)

With water storage, the three veneering composites showed significant difference (p<0.001). The initial mean Ra for SFN showed lower values than GRD (p=0.005) and VVL (p=0.002). An impact of aging (p=0.475) was not observed (Table 3, 4) and the three veneering composites behaved similarly and did not show an increase (between SFN and GRD: p=0.879, between SFN and VVL: p=0.596).

Thermocycling (5°C/55°C)

The lowest initial mean Ra-values was obtained for SFN being statistically different from VVL (p=0.019). GRD showed an increase in Ra-values (p=0.002) during aging (Table 3, 4). The increase of SFN and VVL was not significant (p=0.140).

4.4 Discoloration

All three veneering composites in all aging levels were significantly different than zero (ΔE-values), indicating significant colour change (Table 5abc).

Coffee storage (37°C)

A similar significant initial mean coffee discoloration (p<0.001) was found in all three veneering composites (between SFN and GRD: p=0.770; between SFN and VVL: p=0.356). The discoloration increased according to the aging level (p=0.001). GRD (p=0.002) and VVL (p<0.001) showed a higher increase than SFN (Table 6). After polishing with prophylaxis paste, the ΔE values of GRD and SFN showed similar discoloration, but VVL presented higher ΔE values after 1 day coffee storage (Table 5a).

Tea storage (37°C)

All three veneering composites discolored in the same range (between SFN and GRD: p=0.104, between SFN and VVL: p=0.720). The increase of discoloration was higher for GRD (p<0.001) and VVL (p=001) compared to SFN (Table 6). After polishing the discoloration of all three tested veneering composites faded and obtained the values range of 1 day black tea storage (Table 5b).

Red wine storage (37°C)

Over time, all composites showed a strong increase of discolorations. No difference in the increase of discoloration with storage time between the three veneering composites were found (between SFN and GRD: p=0.538; between SFN and VVL: p=0.306) (Table 6, Fig. 1). After polishing, the ΔE values found higher discoloration than after 1 day of wine storage in all veneering composites (Table 5c).

4.5 Abrasion stability

All veneering composites showed significantly mean loss of material (p<0.001). VVL showed a higher material loss than GRD and SFN (p=0.002) (Table 7, Fig. 2). SFN and GRD were similar regarding the level of material loss. The impact of the increasing number of chewing cycles resulted into a significant higher loss of material in VVL (p<0.001) compared to the loss occurred with SFN and GRD, which showed (p=0.002) only a slight increase of material loss in all remaining aging levels good abrasion stability.

4.6 Subjective assessment of obtained results

In summary, SFN showed the highest stability, followed by GRD, and VVL presented the highest number of negative points (Table 8).

5. Discussion

The three veneering composites have different composition. GC Gradia is a fine hybrid composite composed of UDMA and EDMA. VITA VM LC is a micro filled composite with EDMA, TEGDMA and DMAEMA. Sinfony belongs to the micro hybrid composite containing a resin matrix composed of HEMA and Octahydro-4,7-methano-1H-indenediyl-bis(methacrylate). Only Sinfony included a post-curing process. The composition and polymerization parameters play a major role for the needed properties of veneering composite (20). For guaranteeing longevity and success it is necessary to know the veneering composite composition and respecting polymerization cycle.

The highest flexure strength after water storage and thermocycling showed Sinfony. The flexural strength of all tested veneering composite was reduced after water storage and thermocycling. Other studies observed similar results after aging (24, 32). Clinically, veneering composites are subjected to complex mastication forces with a considerable amount of flexural stresses (33). For restorations that are subjected to large masticatory stresses, high flexural strength is desired to avoid fractures (34, 35).

The veneering composite VITA VM LC obtained a stable Martens hardness, whereby the results of GC Gradia and Sinfony showed an increase parallel to the aging level. Hence, it can be stated that throughout the entire observation period of 180 days, no negative effect of aging occurred on the surface layer. The increase in hardness during the aging time might be explained by after-polymerization. An increase of Martens hardness of direct resin composites was also reported in another study (24). Hardness can be used to evaluate the relative degree of the conversion of a composite (36). Therefore, it can be concluded that a conversion occurred during aging in the veneering composites GC Gradia und Sinfony.

Despite the uniform polishing of the specimens, differences of surface roughness have been detected. Sinfony had a smoother surface represented by significantly lower Ra-values than the other measured veneering composites. During the aging process with water storage, the surface roughness was stable for all veneering composites. Within the aging method thermocycling, the surface of Sinfony stayed stable, while the surface roughness of VITA VM LC was reduced with a smoother surface. With GC Gradia the surface roughness increased with aging and got rougher.

After 180 days, the discoloration increased from black tea over coffee to red wine, respectively. The differences were more than ΔE values of > 3.3 reflecting clinically significant visual discoloration (37). After polishing, the ΔE values for black tea, for coffee (except VITA VM LC) and red wine (except VITA VM LC) decreased below 3.3 ΔE and were clinical acceptable. These findings of acceptable color stability of these second-generation of veneering composites were confirmed in another study (38).

In our in-vitro study, Δb values representing a yellowish shimmer were predominately positive and on the higher level. An in-vivo study reported that the tested material became darker and more yellowish after 18 months (39). Another clinical study reported a statistically significant yellow discoloration after 2 years (40). The phenomenon of this yellow discoloration was attributed to the presence of residual camphorquinone, which was added to the composite materials as a photo-initiator (41). In general, the color stability is influenced by the intensity and duration of polymerization and consequently by the degree of conversion (42). Therefore, it is important to adhere to the polymerization parameters and the corresponding timing.

Veneering composite Sinfony together with GC Gradia exhibited significant less wear than VITA VM LC after 1.2 Million cycles, which is equivalent to 5 years of clinical situation (43). The included thermocycling was used to obtain an increasing wear effect (44). The loss of material results into a rougher surface, which build a predisposing factor for bacteria adhesion, plaque maturation, periodontal disease, and extrinsic staining (45). Two veneering composites (Sinfony and GC Gradia) in this study showed similar abrasion stability than human enamel (46).

The subjective assessment of the properties of the three tested veneering composite after aging resulted into highest stability for the veneering composite Sinfony.

The application of veneering composite with fixed-dental prosthesis based on metal framework is well proven. The second-generation of veneering composites yield good mechanical properties and the results of this study showed good long-term stability of the tested veneering composites. Further studies are needed to evaluate their performance on ceramics.

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7. Tables and Figures

Table 1

Composite type	Product name (short name)	Manufacturer	Composition	Used polymerisation Unit*	Shade	LOT-number
Fine hybrid	**GC Gradia (GRD)**	GC Europe, Leuven, Belgium	UDMA, EDMA, Ceramic, Prepolymer	LABOLIGHT LV-III (GC Europe, Leuven, Belgium) for 5 min	A3	0805122, 0807151
Micro filled	**VITA VM LC (VVL)**	VITA Zahnfabrik, Bad Säckingen, Germany	EDMA, TEGDMA, DMAEMA, Prepolymerized Splinters	SPEED LABOLIGHT (Hager & Werken, Duisburg, Germany) for 10 min	3M2	10030, 20281
Micro hybrid	**Sinfony (SFN)**	3M ESPE, Seefeld, Germany	HEMA, Octahydro-4,7-methano-1H-indenediyl-bis(methylene-diacrylate), Glas, Glasionomer	Pre-polymerisation: Visio Alfa (3M ESPE) for 5 s End-polymerisation: Visio Beta Vario (3M ESPE) for 16 min under vacuum	A3	335152, 351464

* all polymerisations units were chosen according to the manufacturer's specific instruction

Summary of products used.

Table 2

Aging level (days)	Water storage (37 °C) mean ± SD (95% CI)	Thermocycling (5 °C/55 °C) mean ± SD (95% CI)
GRD		
Initial	66.6 ± 12.4 (59.7,73.5)^{a,A,Z}	
1	142.7 ± 26.1 (128.2,157.1)^{a,C}	113.2 ± 10.9 (107.1,109.2)^{b,Y}
7	126.8 ± 14.3 (118.8,134.7)^{a,C}	99.1 ± 13.6 (91.6,106.7)^{a,Y}
28	122.6 ± 17.1 (113.1,132.0)^{b,BC}	95.5 ± 18.9 (85.0,106.0)^{ab,Y}
90	104.5 ± 18.9 (94.0,115.0)^{a,B}	63.3 ± 12.3 (56.5,70.1)^{a,Z}
180	73.8 ± 10.8 (67.8,79.8)^{a,A}	66.6 ± 16.0 (57.7,75.5)^{ab,Z}
VVL		
Initial	136.6 ± 23.9 (123.3,149.9)^{c,D,X}	
1	131.1 ± 15.8 (122.3,139.8)^{a,CD}	97.4 ± 14.9 (89.1,105.6)^{a,Y}
7	109.1 ± 24.3 (95.6,122.5)^{a,BC}	134.2 ± 23.0 (121.5,146.9)^{b,X}
28	104.7 ± 20.2 (93.5,115.8)^{a,B}	90.9 ± 20.9 (79.3,102.4)^{a,ZX}
90	118.9 ± 16.3 (109.9,128.0)^{ab,BCD}	106.0 ± 13.6 (98.5,113.5)^{b,Y}
180	79.4 ± 10.7 (73.4,85.3)^{a,A}	71.6 ± 11.9 (64.6,78.5)^{b,Z}
SFN		
Initial	117.2 ± 14.0 (109.4,124.9)^{b,AB,YX}	
1	141.7 ± 23.4 (128.7,154.7)^{a,C}	105.4 ± 19.1 (94.8,116.0)^{ab,Y}
7	116.3 ± 23.9 (103.0,129.5)^{a,AB}	137.4 ± 19.8 (126.4,148.4)^{b,X}
28	138.5 ± 13.8 (130.8,146.1)^{b,BC}	112.7 ± 24.3 (98.0,127.4)^{b,YX}
90	134.1 ± 25.6 (119.9,148.2)^{b,BC}	132.7 ± 24.7 (119.0,146.4)^{c,X}
180	99.7 ± 9.5 (94.4,105.0)^{b,A}	56.6 ± 16.6 (46.5,66.6)^{a,Z}

a,b,c...reflect significant difference value levels from one-way ANOVA ($p<0.05$) within the same aging levels and aging types between the three veneering composites

A,B,C… reflect significant difference value levels after water storage from one-way ANOVA ($p<0.05$) within the same veneering composites between the six aging levels

Z,Y,X… reflect significant difference value levels after thermocycling from one-way ANOVA ($p<0.05$) within the same veneering composites between the six aging levels

Mean, SD and 95% confidence interval of flexure strength (MPa) of each aging type and level and veneering composite.

Table 3

Aging level (days)	Martens hardness		Surface roughness	
	Water storage (37 °C)	**Thermocycling (5 °C/55 °C)**	**Water storage (37 °C)**	**Thermocycling (5 °C/55 °C)**
	mean + SD (95% CI)	mean + SD (95% CI)	mean + SD (95% CI)	mean + SD (95% CI)
GRD				
Initial	158.7 ± 17.9 (136.5,180.9)	169.9 ± 17.8 (147.8,192.0)	0.099 ± 0.013 (0.082,0.116)	0.112 ± 0.016 (0.092,0.132)
1	193.2 ± 39.8 (143.7,242.6)	178.4 ± 37.3 (132.0,224.7)	0.099 ± 0.013 (0.082,0.115)	0.119 ± 0.021 (0.092,0.145)
7	209.6 ± 38.0 (162.4,256.8)	239.7 ± 30.2 (202.1,277.3)	0.117 ± 0.016 (0.097,0.136)	0.128 ± 0.023 (0.099,0.157)
28	221.9 ± 34.8 (178.7,265.1)	214.5 ± 21.3 (188.0,241.0)	0.114 ± 0.026 (0.081,0.147)	0.137 ± 0.025 (0.106,0.168)
90	239.6 ± 17.5 (217.8,261.3)	257.5 ± 22.7 (229.3,285.7)	0.115 ± 0.032 (0.075,0.155)	0.172 ± 0.067 (0.088,0.255)
180	243.0 ± 17.9 (220.7,265.2)	241.1 ± 21.1 (214.9,267.2)	0.098 ± 0.020 (0.073,0.124)	0.165 ± 0.051 (0.101,0.229)
VVL				
Initial	223.2 ± 25.2 (191.9,254.5)	221.8 ± 46.5 (164.0,279.6)	0.094 ± 0.028 (0.058,0,128)	0.101 ± 0.028 (0.066,0.136)
1	218.3 ± 19.3 (194.2,242.3)	193.6 ± 20.7 (167.9,219.3)	0.113 ± 0.019 (0.089,0.137)	0.118 ± 0.026 (0.086,0.150)
7	202.6 ± 32.7 (162.0,243.2)	176.1 ± 25.7 (144.1,208.0)	0.112 ± 0.035 (0.068,0.155)	0.141 ± 0.017 (0.119,0.162)
28	194.9 ± 29.7 (157.9,231.8)	208.3 ± 15.3 (189.3,227.2)	0.129 ± 0.034 (0.086,0.172)	0.157 ± 0.024 (0.126,0.187)
90	211.8 ± 17.2 (190.4,233.2)	202.9 ± 34.9 (159.6,246.2)	0.134 ± 0.034 (0.092,0.176)	0.144 ± 0.016 (0.124,0.165)
180	217.0 ± 11.0 (203.3,230.6)	208.6 ± 32.9 (167.7,249.5)	0.089 ± 0.021 (0.062,0.115)	0.077 ± 0.009 (0.065,0.089)
SFN				
Initial	144.2 ± 26.0 (111.9,176.4)	139.8 ± 16.5 (119.2,160.3)	0.064 ± 0.008 (0.053,0.074)	0.073 ± 0.017 (0.052,0.094)
1	173.0 ± 24.8 (142.2,203.8)	161.7 ± 24.1 (131.7,191.6)	0.064 ± 0.012 (0.049,0.079)	0.082 ± 0.036 (0.036,0.126)
7	185.0 ± 11.6 (170.6,199.4)	201.7 ± 34.7 (158.5,244.8)	0.067 ± 0.013 (0.050,0.082)	0.097 ± 0.020 (0.071,0.121)
28	208.5 ± 14.3 (190.6,226.3)	207.2 ± 20.7 (181.4,232.9)	0.069 ± 0.009 (0.057,0.081)	0.113 ± 0.025 (0.082,0.144)
90	192.4 ± 29.0 (156.3,228.4)	217.3 ± 26.2 (184.8,249.8)	0.070 ± 0.006 (0.062,0.077)	0.112 ± 0.014 (0.095,0.129)
180	203.0 ± 9.2 (191.6,214.4)	218.1 ± 6.9 (209.4,226.7)	0.058 ± 0.007 (0.049,0.066)	0.070 ± 0.017 (0.048,0.092)

Means, SD and 95% confidence interval of mean Martens hardness and surface roughness Ra values of each aging type and level for all veneering composites.

Table 4

Parameter	Estimate (standard error) (95 % CI)	Significance p-values	Estimate (standard error) (95% CI)	Significance p-values
	Martens hardness		**Surface roughness**	
Water storage (37 °C)				
Constant term	174.9 (7.6) (159.0;190.8)	**< 0.001**	0.07 (0.01) (0.05;0.09)	**< 0.001**
[GRD]	19.0 (10.8) (-3.5;41.5)	0.093	0.04 (0.01) (0.01;0.07)	**0.005**
[VVL]	35.3 (10.8) (12.8;57.7)	**0.004**	0.05 (0.01) (0.02;0.08)	**0.002**
[SFN]	0.00 (0)	-	0.00 (0)	-
water storage	0.2 (0.1) (0.04;0.3)	**0.016**	<0.01 (<0.01) (<0.01;<0.01)	0.475
[GRD] * water storage	0.2 (0.1) (-0.1;0.4)	0.159	<0.01 (<0.01) (<0.01;<0.01)	0.879
[VVL] * water storage	-0.2 (0.1) (-0.4;0.1)	0.126	<0.01 (<0.01) (<0.01;<0.01)	0.596
[SFN] * water storage	0.00 (0)	-	0.00 (0)	-
Thermocycling (5°C / 55°C)				
Constant term	175.6 (9.0) (156.9;194.3)	**< 0.001**	0.09 (0.01) (0.07;0.12)	**< 0.001**
[GRD]	25.2 (12.7) (-1.2;51.7)	0.060	0.03 (0.02) (<0.01;0.06)	0.065
[VVL]	23.7 (12.7) (-2.7;50.2)	0.075	0.04 (0.02) (0.01;0.07)	**0.019**
[SFN]	0.00 (0)	-	0.00 (0)	-
thermocycling	0.3 (0.1) (0.1;0.5)	**0.001**	<0.01 (<0.01) (<0.01;<0.01)	0.532
[GRD] * thermocycling	0.01 (0.1) (-0.3;0.3)	0.919	<0.01 (<0.01) (<0.01;<0.01)	**0.002**
[VVL] * thermocycling	-0.3 (0.1) (-0.5;-0.01)	**0.045**	<0.01 (<0.01) (<0.01;<0.01)	0.140
[SFN] * thermocycling	0.00 (0)	-	0.00 (0)	-

Estimates of fixed parameters of Martens hardness and surface roughness values separately within aging types (linear mixed models analysis).

Table 5a

Aging level (days)	ΔL	Δa	Δb	ΔE
	mean ± SD (95% CI)	mean ± SD (95% CI)	mean ± SD (95% CI)	mean ± SD (95% CI)
GRD				
1	-0.70 ± 0.39 (-0.98,-0,42)	0.31 ± 0.19 (0.17,0.45)	1.25 ± 0.49 (0.89,1.59)	1.52 ± 0.49 (1.16,1.87)
7	-1.65 ± 0.46 (-1.98,-1.32)	0.70 ± 0.28 (0.49,0.89)	2.09 ± 0.59 (1.67,2.51)	2.79 ± 0.65 (2.32,3.25)
28	-2.80 ± 0.39 (-3.08,-2.52)	0.73 ± 0.20 (0.57,0.87)	1.81 ± 0.48 (1.46,2.15)	3.44 ± 0.44 (3.12,3.76)
90	-3.22 ± 0.63 (-3.67,-2.77)	0.89 ± 0.32 (0.65,1.12)	2.89 ± 0.63 (2.43,3.34)	4.44 ± 0.79 (3.87,5.01)
180	-3.02 ± 0.64 (-3.48,-2.57)	0.92 ± 0.31 (0.70,1.15)	3.27 ± 0.85 (2.65,3.88)	4.60 ± 0.80 (4.03,5.17)
after polishing	-1.20 ± 0.86 (-1.82,-0.58)	0.16 ± 0.23 (0.00,0.33)	0.96 ± 1.03 (0.23,1.70)	1.86 ± 0.80 (1.29,2.44)
VVL				
1	-0.38 ± 1.24 (-1.27,0.51)	0.10 ± 0.26 (-0.08,0.28)	1.77 ± 0.54 (1.38,2.16)	2.17 ± 0.55 (1.78,2.57)
7	-0.78 ± 1.60 (-1.93,0.36)	0.22 ± 0.24 (0.04,0.40)	3.35 ± 0.56 (2.95,3.75)	3.76 ± 0.66 (3.28,4.23)
28	-2.63 ± 1.53 (-3.72,-1.53)	0.19 ± 0.43 (-0.12,0.50)	2.85 ± 1.11 (2.05,3.64)	4.22 ± 0.81 (3.64,4.80)
90	-2.36 ± 1.63 (-3.52,-1.20)	0.19 ± 0.27 (0.00,0.38)	4.78 ± 0.95 (4.09,5.46)	5.59 ± 0.69 (5.09,6.08)
180	-2.13 ± 1.54 (-3.24,-1.03)	-0.14 ± 0.24 (-0.31,0.04)	5.69 ± 1.02 (4.96,6.42)	6.24 ± 1.09 (5.46,7.03)
after polishing	-0.97 ± 2.03 (-2.42,0.48)	-0.50 ± 0.28 (-0.70,-0.30)	3.62 ± 1.11 (2.83,4.41)	4.29 ± 0.91 (3.64,4.94)
SFN				
1	1.29 ± 3.30 (-1.07,3.65)	0.07 ± 0.25 (-0.11,0.25)	1.08 ± 0.93 (0.41,1.75)	2.52 ± 2.82 (0.50,4.54)
7	0.57 ± 3.02 (-1.59,2.73)	0.15 ± 0.31 (-0.07,0.37)	1.73 ± 0.89 (1.09,2.36)	2.71 ± 2.36 (1.02,4.40)
28	-0.38 ± 2.54 (-2.20,1.44)	0.42 ± 0.30 (0.20,0.63)	1.65 ± 0.76 (1.10,2.20)	2.87 ± 1.16 (2.04,3.71)
90	0.48 ± 3.25 (-1.85,2.81)	0.40 ± 0.25 (0.22,0.58)	2.47 ± 0.72 (1.95,2.98)	3.51 ± 2.16 (1.96,5.05)
180	1.14 ± 3.01 (-1.02,3.30)	0.48 ± 0.15 (0.36,0.59)	2.78 ± 0.84 (0.36,0.59)	3.64 ± 2.32 (1.97,5.30)
after polishing	2.46 ± 3.55 (-0.08,5.00)	0.03 ± 0.24 (-0.14,0.20)	0.19 ± 0.76 (-0.35,0.73)	2.76 ± 3.39 (0.34,5.19)

Means, SD and 95% confidence interval for coffee discoloration of each aging level for all three veneering composites.

1.

Table 5b

Aging level (days)	ΔL	Δa	Δb	ΔE
	mean ± SD (95% CI)	mean ± SD (95% CI)	mean ± SD (95% CI)	mean ± SD (95% CI)
GRD				
1	-0.50 ± 0.49 (-0.85,-0.15)	0.24 ± 0.18 (0.11,0.37)	0.66 ± 0.48 (0.31,1.00)	1.02 ± 0.42 (0.72,1.32)
7	-0.96 ± 0.59 (-1.38,-0.54)	0.31 ± 0.21 (0.15,0.46)	0.85 ± 0.55 (0.45,1.24)	1.45 ± 0.53 (1.07,1.83)
28	-1.43 ± 0.43 (-1.74,-1.12)	0.31 ± 0.27 (0.12,0.51)	0.51 ± 0.62 (0.06,0.95)	1.68 ± 0.42 (1.37,1.98)
90	-1.21 ± 0.42 (-1.51,-0.91)	0.23 ± 0.30 (0.00,0.44)	2.75 ± 1.30 (1.81,3.68)	3.12 ± 1.11 (2.32,3.91)
180	-2.04 ± 0.76 (-2.59,-1.50)	0.64 ± 0.38 (0.36,0.91)	2.86 ± 1.41 (1.85,3.87)	3.71 ± 1.26 (2.80,4.61)
after polishing	-0.29 ± 0.87 (-0.91,0.34)	0.06 ± 0.29 (-0.15,0.27)	0.82 ± 1.43 (-0.21,1.84)	1.40 ± 1.24 (0.51,2.29)
VVL				
1	-0.32 ± 1.40 (-1.33,0.68)	0.23 ± 0.15 (0.12,0.34)	0.67 ± 0.35 (0.42,0.93)	1.37 ± 0.84 (0.77,1.97)
7	-0.11 ± 1.41 (-1.11,0.90)	0.39 ± 0.16 (0.26,0.50)	1.68 ± 0.65 (1.21,2.15)	2.08 ± 0.98 (1.38,2.78)
28	-1.37 ± 1.59 (-2.51,-0.23)	0.23 ± 0.37 (-0.03,0.49)	1.33 ± 1.06 (0.56,2.09)	2.50 ± 0.98 (1.79,3.20)
90	-2.03 ± 1.71 (-3.25,-0.80)	0.59 ± 0.49 (0.24,0.94)	2.72 ± 1.17 (1.88,3.56)	3.87 ± 1.05 (3.11,4.62)
180	-1.61 ± 1.40 (-2.61,-0.61)	0.67 ± 0.37 (0.40,0.93)	3.04 ± 0.98 (2.33,3.74)	3.82 ± 0.69 (3.32,4.31)
after polishing	0.08 ± 1.55 (-1.03,1.18)	0.04 ± 0.25 (-0.14,0.21)	0.94 ± 0.93 (0.28,1.61)	1.44 ± 1.42 (0.42,2.45)
SFN				
1	0.97 ± 1.50 (-0.10,2.04)	0.14 ± 0.23 (-0.20,0.31)	0.79 ± 0.51 (0.42,1.15)	1.69 ± 1.07 (0.92,2.45)
7	0.86 ± 1.37 (-0.12,1.84)	-0.01 ± 0.33 (-0.25,0.23)	0.95 ± 0.85 (0.34,1.56)	1.79 ± 0.99 (1.07,2.50)
28	-0.15 ± 2.18 (-1.71,1.41)	0.23 ± 0.39 (-0.05,0.51)	0.24 ± 1.15 (-0.58,1.07)	2.30 ± 0.71 (1.79,2.81)
90	0.68 ± 1.44 (-0.36,1.71)	0.21 ± 0.44 (-0.11,0.52)	1.49 ± 0.91 (0.84,2.14)	2.16 ± 0.97 (1.46,2.85)
180	0.69 ± 0.91 (0.04,1.35)	0.60 ± 0.32 (0.37,0.83)	2.34 ± 0.88 (1.71,2.97)	2.71 ± 0.79 (2.13,3.27)
after polishing	1.76 ± 1.56 (0.64,2.87)	0.28 ± 0.27 (0.09,0.47)	-0.42 ± 0.59 (-0.84,0.00)	2.18 ± 1.13 (1.37,2.99)

Means, SD and 95% confidence interval for black tea discoloration of each aging level for all three veneering composites.

Table 5c

Time of aging (d)	ΔL	Δa	Δb	ΔE
	mean ± SD (95% CI)	mean ± SD (95% CI)	mean ± SD (95% CI)	mean ± SD (95% CI)
		GRD		
1	-0.90 ± 0.28 (-1.09,0.70)	0.20 ± 0.22 (0.04,0.35)	-0.13 ± 0.48 (-0.47,0.22)	1.04 ± 0.33 (0.79,1.28)
7	-2.56 ± 0.47 (-2.89,-2.22)	0.67 ± 0.28 (0.47,0.87)	-0.02 ± 0.76 (-0.57,0.52)	2.75 ± 0.47 (2.41,3.09)
28	-10.10 ± 3.13 (-12.34,-7.86)	3.59 ± 1.49 (2.52,4.66)	-0.87 ± 1.74 (-2.11,0.38)	10.93 ± 3.27 (8.59,13.28)
90	-18.12 ± 7.80 (-23.68,-12.53)	7.87 ± 4.12 (4.92,10.82)	2.16 ± 1.90 (0.79,3.52)	19.96 ± 8.77 (13.68,26.24)
180	-19.19 ± 6.22 (-23.63,-14.74)	10.50 ± 3.74 (7.82,13.18)	6.80 ± 2.58 (4.95,8.64)	23.02 ± 7.29 (17.81,28.24)
after polishing	-1.93 ± 0.92 (-2.58,-1.27)	0.53 ± 0.32 (0.30,0.76)	1.86 ± 1.16 (1.03,2.69)	2.90 ± 1.12 (2.10,3.70)
		VVL		
1	-1.66 ± 0.70 (-2.16,-1.17)	0.49 ± 0.37 (0.22,0.76)	0.07 ± 0.90 (-0.58,0.71)	1.96 ± 0.71 (1.45,2.47)
7	-2.82 ± 1.17 (-3.66,-1.98)	0.39 ± 0.55 (-0.01,0.78)	1.25 ± 1.73 (0.01,2.48)	3.61 ± 0.95 (2.93,4.29)
28	-17.33 ± 4.97 (-20.89,-13.78)	6.73 ± 1.93 (5.34,8.12)	0.56 ± 1.82 (-0.75,1.86)	18.69 ± 5.29 (14.91,22.48)
90	-24.41 ± 7.66 (-29.88,-18.93)	12.21 ± 4.27 (9.15,15.26)	4.42 ± 2.73 (2.46,6.38)	27.78 ± 8.73 (21.53,34.02)
180	-25.31 ± 7.08 (-30.37,-20.24)	14.08 ± 3.65 (11.46,16.69)	8.32 ± 2.44 (6.57,10.07)	30.30 ± 7.60 (24.86,35.74)
after polishing	-1.54 ± 0.69 (-2.04,-1.05)	0.24 ± 0.46 (-0.09,0.56)	2.79 ± 1.38 (1.81,3.77)	3.35 ± 1.20 (2.49,4.21)
		SFN		
1	0.54 ± 0.62 (0.09,0.99)	0.59 ± 0.44 (0.27,0.90)	0.11 ± 0.75 (-0.42,0.65)	1.19 ± 0.54 (0.80,1.57)
7	1.16 ± 1.66 (-0.03,2.35)	0.50 ± 0.26 (0.31,0.68)	0.14 ± 0.46 (-0.19,0.46)	1.83 ± 1.04 (1.08,2.58)
28	-8.67 ± 5.13 (-12.34,-5.00)	5.89 ± 2.53 (4.08,7.70)	2.02 ± 1.49 (0.94,3.08)	10.84 ± 5.58 (6.84,14.83)
90	-15.89 ± 6.51 (-20.54,-11.23)	10.78 ± 3.49 (8.27,13.27)	7.39 ± 2.72 (5.44,9.34)	20.69 ± 7.51 (15.32,26.06)
180	-17.52 ± 6.55 (-22.20,-12.83)	12.89 ± 3.60 (10.31,15.47)	11.47 ± 2.78 (9.47,13.46)	24.78 ± 7.27 (19.58,29.99)
after polishing	1.59 ± 0.74 (1.07,2.12)	0.37 ± 0.22 (0.21,0.52)	0.05 ± 0.63 (-0.40,0.50)	1.75 ± 0.74 (1.22,2.28)

Means, SD and 95% confidence interval for red wine discoloration of each aging level for all three veneering composites.

Table 6

Parameter	Estimate (standard error)	Significance p-values	95% CI
Coffee			
Constant term	2.67 (0.43)	**< 0.001**	(1.80;3.54)
[GRD]	-0.18 (0.60)	0.770	(-1.41;1.05)
[VVL]	0.57 (0.60)	0.356	(-0.66;1.80)
[SFN]	0.00 (0)	-	-
coffee storage	0.01 (<0.01)	**0.001**	(<0.01;0.01)
[GRD] * coffee storage	0.01 (<0.01)	**0.002**	(<0.01;0.01)
[VVL] * coffee storage	0.01 (<0.01)	**< 0.001**	(0.01;0.02)
[SFN] * coffee storage	0.00 (0)	-	-
Tea			
Constant term	1.83 (0.23)	**< 0.001**	(1.37;2.30)
[GRD]	-0.54 (0.33)	0.104	(-1.20;0.12)
[VVL]	0.12 (0.33)	0.720	(-0.54;0.78)
[SFN]	0.00 (0)	-	-
tea storage	<0.01 (<0.01)	**0.003**	(<0.01;0.01)
[GRD] * tea storage	0.01 (<0.01)	**< 0.001**	(0.01;0.01)
[VVL] * tea storage	0.01 (<0.01)	**0.001**	(<0.01;0.01)
[SFN] * tea storage	0.00 (0)	-	-
Red wine			
Constant term	3.72 (1.44)	**0.012**	(0.85;6.59)
[GRD]	0.42 (2.03)	0.838	(-3.64;4.48)
[VVL]	3.37 (2.03)	0.102	(-0.69;7.43)
[SFN]	0.00 (0)	-	-
wine storage	0.13 (0.01)	**< 0.001**	(0.11;0.16)
[GRD] * wine storage	-0.01 (0.02)	0.538	(-0.05;0.03)
[VVL] * wine storage	0.02 (0.02)	0.306	(-0.02;0.06)
[SFN] * wine storage	0.00 (0)	-	-

Estimates of fixed parameters for discoloration ΔE, separately for coffee, black tea and red wine (linear mixed models analysis).

Table 7

Chewing cycles	**GRD** mean ± SD (95% CI) (µm)	**VVL** mean ± SD (95% CI) (µm)	**SFN** mean ± SD (95% CI) (µm)
120 000	26.2 ± 7.5 (18.3,34.0)	40.8 ± 11.1 (29.2,52.5)	35.3 ± 6.9 (28.1,42.5)
240 000	29.8 ± 7.0 (22.4,37.2)	59.3 ± 21.0 (37.2,81.4)	38.5 ± 8.3 (29.8,47.2)
640 000	33.2 ± 8.5 (24.2,42.1)	77.2 ± 23.5 (52.5,101.8)	44.5 ± 10.6 (33.3,55.7)
1 200 000	38.2 ± 11.9 (25.6,50.6)	111.2 ± 31.5 (78.1,144.2)	50.3 ± 15.8 (33.7,66.9)

Estimates of fixed parameters of abrasion stability values (linear mixed models)

Parameter	Estimate (standard error) (95% CI)	Significance p-values
Constant term	42.2 (5.6) (30.1;54.2)	**< 0.001**
[GRD]	-10.3 (8.0) (-27.3;6.7)	0.214
[VVL]	30.0 (8.0) (12.9;47:0)	**0.002**
[SFN]	0.0 (0)	-
chewing simulation	0.1 (0.04) (0.0;0.3)	**0.002**
[GRD] * chewing simulation	-0.03 (0.06) (-0.1;0.1)	0.573
[VVL] * chewing simulation	0.5 (0.06) (0.3;0.6)	**< 0.001**
[SFN] * chewing simulation	0.0 (0)	-

Means, SD and 95% confidence interval of mean abrasion stability values of each aging level for all veneering composites and estimates of fixed parameters of abrasion stability values (linear mixed models analysis).

Table 8

Veneering composite properties	GRD	VVL	SFN
Flexural strength after water storage			+
Flexural strength after thermocycling			
Martens hardness after water storage	+	+	
Martens hardness after thermocycling	+	+	+
Surface roughness after water storage			+
Surface roughness after thermocycling	-		+
Coffee discoloration		-	+
Black tea discoloration			
Red Wine discoloration		-	
Abrasions stability after chewing simulation		-	

Subjective assessment of obtained results based on the statistically significant differences.

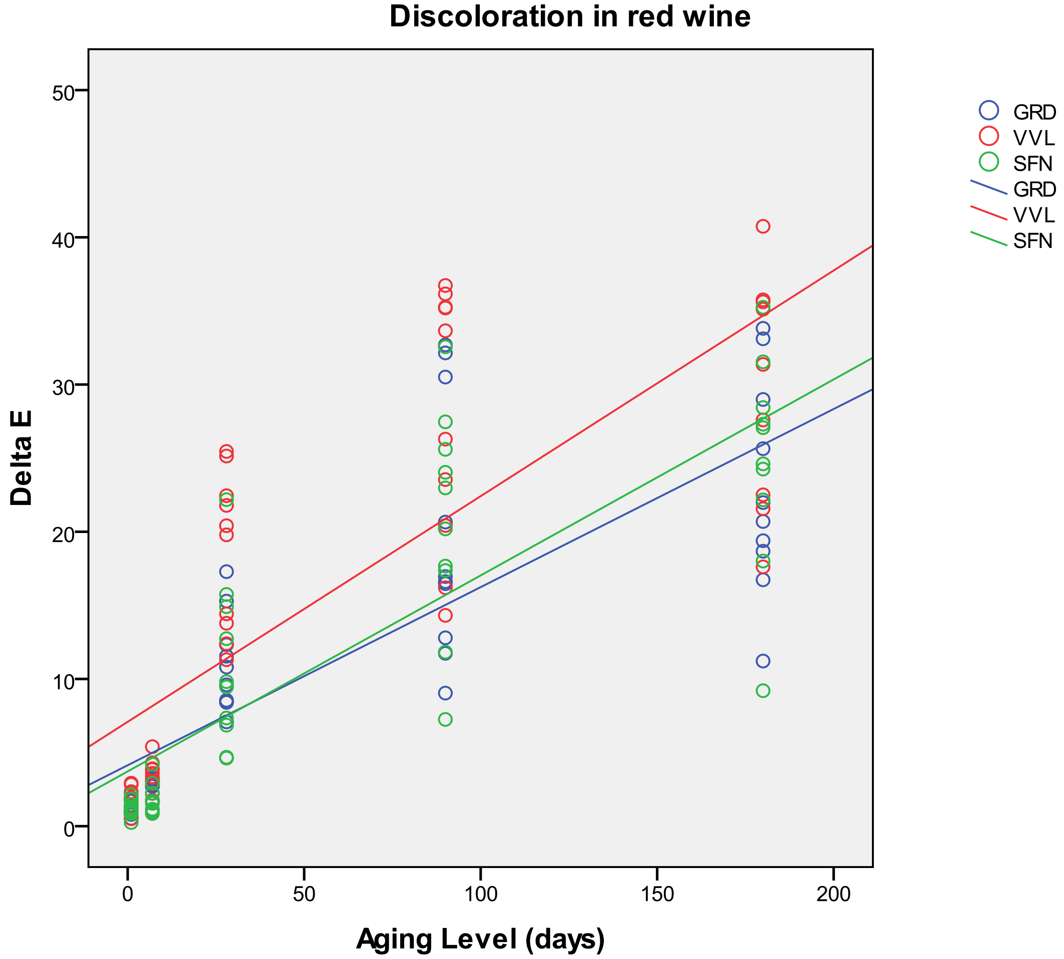

Fig. 1. Diagram of discoloration after red wine storage for each veneering composite of each aging level (GRD = GC Gradia, VVL = VITA VM LC and SFN = Sinfony).

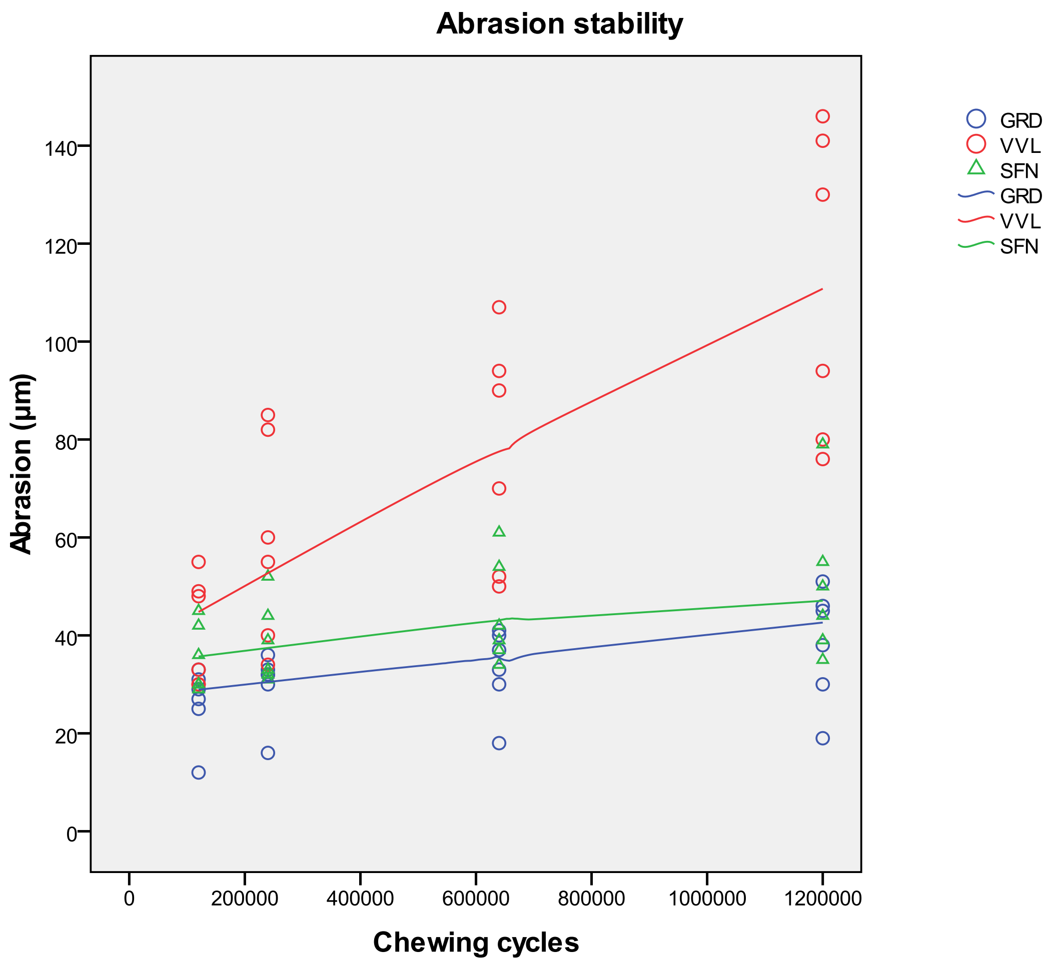

Fig. 2. Diagram of material loss of abrasion stability for each veneering composite of each aging level (GRD = GC Gradia, VVL = VITA VM LC and SFN = Sinfony).