SCIENTIFIC REPORT

Grandio blocs – Surface conditioning

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The production of indirect restorations with the help of CAD/CAM technology has established itself over decades and offers enormous advantages: it saves time, eliminates possible sources of errors and allows highly precise restorations. Widely used materials include all-ceramics (e.g., feldspar or oxide ceramics) and zirconium dioxide as well as the newer ceramic-based hybrid materials. The luting of indirect restorations is a decisive factor which can affect and dramatically shorten the longevity of restorations. Surface conditioning of the restoration is also decisive for the success of the luting. The manufacturer's specifications for the multifaceted materials vary considerably, including from material to material. Different CAD/CAM blocks, including Grandio blocs from VOCO, were pre-treated using different methods at the University of Regensburg. The resulting surface properties were evaluated and material-specific surface conditioning techniques derived.[1]

Study design

The respective blocks were cut into thin slices (14 x 14 x 2 mm) to produce the test specimens. Ceramic materials were then sintered. Following cleaning with ethanol, untreated specimens of each material were put to one side as a control. The specimens of all the materials were then pre-treated with the following procedures (see also table 2): in accordance with the manufacturer's specifications (see table 1); etching for 20 seconds with (5 %) hydrofluoric acid or (37 %) phosphoric acid, priming with Monobond etch and prime (Ivoclar Vivadent), roughening with diamond burs with simultaneous water cooling (80 and 4 µm), sandblasting with aluminium oxide (Al_2O_3) with fine (50 µm) and coarse (120 µm) particles at low (1 bar) and high (2 bar) pressure. The changes in the roughness of the surfaces and the surface energy were used to assess the specimens in comparison with the parameters of the untreated surfaces. In addition, the surfaces were also inspected for structural changes such as the formation of cracks, material loss (e.g. filler particles) and microchipping under a scanning electron microscope. Following the conditioning, the surfaces should display increased roughness (larger surface area) and increased surface energies (better coating with the luting agent) so as to guarantee an optimal bond with the luting material. At the same time, the materials should not undergo any structural changes as a result of the pre-treatment, so as to ensure that neither the strength nor the resulting longevity is affected.

Table 1: Overview of investigated materials & manufacturer's specifications for pre-treatment

Product/ manufacturer	Material*	Abbre-viation	Flexural strength [MPa]	E-Modulus [MPa]	Manufacturer's specifications for pre-treatment		
					Sandblasting	Etching	Primer
Celtra DUO/ Dentsply Sirona	Lithium silicate ceramic	CD	210 (polished)/ 370 (glazed)	70		HF (5 %), 30 s	Silane (Calibra silane bonding agent)
VITA SUPRINITY/ VITA	Zirconium dioxide-reinforced lithium silicate ceramic	VS	420	70		HF (5 %), 20 s	Silane, 60 s (Vitasil)
IPS e.max CAD/ Ivoclar Vivadent	Lithium disili-cate	EMA	360	95		HF (5 %), 20 s	Universal primer, 60 s (Monobond Plus)
IPS e.max ZirCAD/ Ivoclar Vivadent	Yttrium-stabilised zirco-nium dioxide	EMZ	>900	205	Al_2O_3, ≤ 1 bar		Universal primer, 60 s (Monobond Plus)
VITA ENAMIC/ VITA	Hybrid ceramic	VE	160	30		HF (5 %), 60 s	Silane, 60 s (Vitasil)
Cerasmart/ GC	Ceramic-based hybrid material	CS	231	12	Al_2O_3, 25-50 µm, ≤ 1 bar		Silane (Ceramic Primer II)
Lava Ultimate/ 3M ESPE	Ceramic-based hybrid material	LU	204	13	Al_2O_3, ≤ 50 µm, 2 bar		Universal adhesive, 20 s (Scotchbond Universal)
SHOFU Block HC/ SHOFU	Ceramic-based hybrid material	SH	191	10	Al_2O_3, 50 µm, 2-3 bar		Optional silane (SHOFU Porcelain Primer)
Grandio blocs**/ VOCO	Ceramic-based hybrid material	VO	333	18	Al_2O_3, 25-50 µm, 1.5-2 bar		Silane, 60 s (Ceramic Bond)
BRILLIANT Crios/ Coltene	Ceramic-based hybrid material	BC	198	10	Al_2O_3, 25-50 µm, 1.5 bar		One-bottle adhesive (One Coat 7 Universal)

*Ceramic-based hybrid materials were labelled uniformly for reasons of legibility.
** The author of the article was not aware of any manufacturer's specifications at the time of the publication.
See instructions for use for manufacturer's exact designation.

Results

Glass ceramics (CD, VS, EMA):
One widespread pre-treatment method for glass ceramics involves etching with hydrofluoric acid (HF) followed by priming with a silane. The results of this study show that etching with HF is also suitable for newer zirconium dioxide-reinforced lithium silicate ceramics. The study also shows that mechanical pre-treatment such as sandblasting or grinding with rough instruments leads to the formation of gaps and microchipping, for which reason mechanical pre-treatment methods are not recommended for glass ceramics.

Zirconium dioxide (EMZ):
Zirconium dioxide occupies a special position among the ceramics as it cannot be pre-treated in the same way as glass ceramics. In fact, the mechanical roughening by means of sandblasting with Al_2O_3 powder or silicon-modified Al_2O_3 has proven its worth for tribochemical silicatisation of the surface. In this study, it was possible to demonstrate that sandblasting at a low blasting pressure (1 bar) with coarse particles (120 µm) achieves the highest roughness and surface energy without damaging the material. As such, these conditions are recommended. In contrast, the use of fine particles (50 µm) is not recommended, as they are not capable of increasing the surface energy significantly. High pressure (2 bar) already provoked initial cracks in the material.

Hybrid ceramics (VE):
Hybrid ceramics also represent an exception from the material perspective. The material displays a hollow, spongelike ceramic structure, which is filled with a methacrylate-based resin. In accordance with the manufacturer's specifications, restorations should be etched with hydrofluoric acid for 60 seconds, and these conditions do indeed deliver good results, i.e., high roughness and surface energy. However, the results of this study show that mechanical pre-treatment can be considerably more advantageous. The roughness and surface energy values with fine particles and high pressure (50 µm/2 bar) were slightly higher than those achieved with etching with hydrofluoric acid, for which reason sandblasting (50 µm/2 bar) can be viewed as a good alterna-

tive to etching with hydrofluoric acid. In contrast, sandblasting with coarse particles at high pressure (120 µm/2 bar) led to cracks in the material.

*Ceramic-based hybrid materials (**VO**, CS, LU, SH, BC)*:

Ceramic-based hybrid materials are largely composed of ceramic filler particles and a lesser proportion of methacrylate resin. We selected a uniform label and did so for reasons of legibility.

Although the share of glass ceramics is very high, the etching with hydrofluoric acid produced relatively poor results. The hydrofluoric acid only triggers mild superficial detachment of the glass particles and the roughness is only low despite the high surface energy. Sandblasting with Al_2O_3 powder is also advantageous here, although the particle size and pressure differ from material to material. **Sandblasting with coarse particles at low pressure (120 µm/1 bar)** produced the optimal results without structural damage for the highly filled (> 80 % by weight) materials such as **Grandio blocs** and also BRILLIANT Crios. This recommendation differs slightly from our instructions for use (25-50 µm/1.5-2 bar). However, the surfaces treated with the latter parameters also displayed good results. Lower-filled (< 80 % by weight) materials gave better values when sandblasted with fine particles at low pressure (50 µm/1 bar). Higher pressures resulted in more and more structural damage on the surfaces. This damage not only weakens the material but can also result in lower adhesion values.

Intraoral repairs:

This study also shows that roughening with rotary instruments is not suitable for the majority of ceramics, as it often results in structural changes and weakening of the materials. This is not the case for ceramic-based hybrid materials. This method produced good but not the best surface properties, which were achieved with sandblasting. For this reason, roughening with burs is not recommended for the pre-treatment prior to luting of restorations. However, roughening with rotary instruments does represent a very interesting option for intraoral repairs. This is a considerable advantage of composites compared with ceramics.

Table 2: Overview of investigated materials & manufacturer's specifications for pre-treatment.
Fields marked green: recommended conditioning; fields marked orange: structural damage, conditioning not recommended.

Method	Materials				
	Glass ceramic	Zirconium dioxide	Hybrid ceramic	Ceramic-based hybrid materials (ceramic > 80 % by weight)	Ceramic-based hybrid materials (ceramic < 80 % by weight)
				e.g., Grandio blocs	e.g., Lava Ultimate
20 s, hydrofluoric acid (5 %)	++	o	o	o	+
20 s, phosphoric acid (37 %)	o	o	o	o	o
Monobond etch and prime (Ivoclar Vivadent)	o	+	o	o	-
Water-cooled diamond bur (80 µm)	+	++	++	++	++
Water-cooled diamond bur (4 µm)	-	o	o	-	o
Sandblasting, Al_2O_3 (50 µm, 1 bar)	+	+	+	+	++
Sandblasting, Al_2O_3 (50 µm, 2 bar)	++	+	++	+	+
Sandblasting, Al_2O_3 (120 µm, 1 bar)	++	++	+	++	+
Sandblasting, Al_2O_3 (120 µm, 2 bar)	++	+	++	++	+
Manufacturer's specifications	++	+	+	+	+

Legend

No change from untreated surface	o
Improvement in roughness and surface energy	+
Deterioration of roughness and surface energy	-
Structural changes, method not recommended	
Conditioning recommended in this study	

Conclusion: The pre-treatment of workpieces for indirect restorations has a considerable influence on the adhesive bond to the luting agent and thus also the tooth. Conversely, the pre-treatment affects the mechanical properties of the restoratives and can, in the worst case scenario, result in their damage and, consequently, reduced durability. It is therefore necessary to find methods which deliver the maximal roughness and surface energies without damaging the material. As a general rule, the pre-treatments in accordance with the manufacturer's specifications produce optimal results. However, this study has shown that the parameters can nevertheless be improved in some cases. The pre-treatment of our Grandio blocs/discs as per the instructions for use produces good results. However, the study showed that blasting pressures as of 2 bar can already be too high. It is thus recommended that lower blasting pressures be used.

[1] Strasser T, Preis V, Behr M, Rosentritt M, *Clin Oral Invest* (2018) *22*: 2787.