Effect of sodium hypochlorite contamination on microhardness of dental core build-up materials

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This study aimed to determine the influence of sodium hypochlorite (NaOCl) contamination on the microhardness of build-up composites. Fifty-two samples, from each of three build-up materials (LuxaCore Dual, MultiCore flow and Rebilda DC) were prepared. Half of the samples from each material were stored in physiologic saline (baseline control) while the other halves were stored in NaOCl. After 1 h, the samples were rinsed with tap water, cut axially and measured for Knoop hardness at different depth levels. The results were analysed by ANOVA and unpaired *t*-tests ($p < 0.05$). Significant differences in microhardness were observed for LuxaCore Dual up to 0.2 mm, Rebilda DC up to 0.3 mm, and for MultiCore flow up to 0.4 mm under the surface level. Contact with sodium hypochlorite on build-up materials causes reduction of the microhardness. The softening is not only limited on the surface, but can also be found in deeper layers of build-up materials.

Keywords: Composite, Degradation, Microhardness

INTRODUCTION

During root canal treatment, a broad range of different rinsing solutions such as chlorhexidine (CHX)¹⁾, sodium hypochlorite (NaOCl)²⁾ or ethylenediamine tetraacetic acid (EDTA)³⁾ are used for dissolving necrotic pulp tissue, inactivating endotoxin, preventing the formation of a smear layer during instrumentation of the root canal or dissolving the smear layer if it has been formed⁴⁾. The most widely recommended rinsing solution is sodium hypochlorite (NaOCl), in concentrations ranging from 0.5%⁵⁾ to 5.25%, or even 10%⁶⁾. NaOCl is favoured because of its high efficiency in dissolving necrotic tissues and its antimicrobial effect. Beside these positive properties of sodium hypochlorite, there are also negative side effects: dentine collagen is destroyed due to fragmentation of peptide chains^{7,8)}, the elastic modulus and flexural strength of human dentine is, depending upon concentration, reduced⁹⁾ and the microhardness of root dentine is reduced after irrigation with NaOCl¹⁰⁾.

Teeth that have been degraded by caries, and in need of root canal treatment, often require a pre-endodontic build-up of the clinical crowns to preserve their functional integrity. The build-up also ensures aseptic principles during root canal treatment, such as facilitating rubber dam placement and coronal disinfection. As possible build-up materials, amalgam, glass ionomers or composites are commonly used in today's practice¹¹⁾. Due to the known disadvantages of amalgam as build-up material, (corrosion¹²⁾ and discoloration of the remaining tooth structure) or poor mechanical properties of glass ionomers as compared with composites¹³⁾, use of composites as build-up materials should be preferred. There are different studies showing a high propensity of composite materials for alteration after contact with chemical

substances such as hydrogen peroxide¹⁴⁾, organic acids¹⁵⁾ or ethanol¹⁶⁾. After completion of the endodontic treatment, the pre-endodontic build-ups are often used as a framework for the final build-up for the prosthetic restoration of the tooth. Since the strength of the build-up¹⁷⁾ is an important factor for the long-term success of the ultimate indirect restoration¹¹⁾, the aim of the present study was to evaluate the influence of sodium hypochlorite treatment on the microhardness of different composite core build-up materials.

MATERIALS AND METHODS

Samples were prepared from the core build-up materials LuxaCore Dual (DMG, Hamburg, Germany) (groups 1 and 2), MultiCore flow (ivoclar vivadent, Schaan, Lichtenstein) (groups 3 and 4) and Rebilda DC (VOCO, Cuxhaven, Germany) (groups 5 and 6). For each group, 26 samples were prepared using moulds of acrylic resin (Palavit G, Kulzer, Wehrheim, Germany) with a thickness of 4 mm and a cylindrical hole with an inner diameter of 3 mm. These moulds were placed on a glass plate and the holes were filled from the backside with the respective core build-up materials. After filling the holes, the samples were stored in physiologic saline for 7 d. After this week, any excess filling material on the backside of the moulds was ground down to the level of the acrylic resin with abrasive paper (800 grit; Water Proof Silicon Carbide Paper, Streuers, Erkrath, Germany).

The samples in groups 1, 3, and 5 were stored in physiologic saline for 1 h and served as baseline control, while the samples of groups 2, 4, and 6 were stored in 5.25% NaOCl for the same amount of time. The NaOCl, however, was renewed every 5 minutes. After 1 h, the moulds with the samples were removed from the water or the NaOCl and were rinsed with tap

water for 5 min. After rinsing, the moulds with the samples were cut axially to the cylindrical samples with a water-cooled diamond disc.

The microhardness of the samples were measured at depths of 0.1, 0.2, 0.3, 0.4, and 0.5 mm from the ground surface with a Knoop hardness-measuring device (High Quality Hardness Tester, Buehler, Düsseldorf, Germany). For Knoop hardness measurement a load of 10 g (≈ 0.1 N) and an indentation time of 20 s was applied. At each depth level, the microhardness was measured four times and the mean hardness for this depth level was calculated.

Scanning electron microscopy pictures were taken of the build-up materials to determine if there were any visible alterations of the surfaces using a Supra 50 VP Scanning Electron Microscope (Carl Zeiss NTS, Oberkochen, Germany). Samples were sputter coated with gold before examination. SEM pictures were captured at a magnification of 1000 $\times$ and an acceleration voltage of 5 kV.

With the data of a pilot study, with 8 samples in each group, a power analysis was done to determine the number of specimens required in each test group. Based on these analyses, 26 samples per group were used to gain power of 97%.

Statistical analysis was performed using ANOVA followed by unpaired t-tests to detect differences in the microhardness between the samples made from the same core build-up material but treated different (group 1 vs. 2; 3 vs. 4 and 5 vs. 6) on the respective depth level. The comparison was started at the depth level of 0.1 mm and continued to the deeper layers until the comparison revealed no significant difference between the samples stored in physiologic saline or NaOCl. The level of significance was set at $p < 0.05$.

RESULTS

The mean microhardness ($\pm$ SD) of the different build-up materials stored in physiologic saline or NaOCl is

Table 1 Table of the composition (manufacturer's information) and group allocation of the core build-up materials

groups	material	composition
1+2	LuxaCore Dual	Bis-GMA based matrix, barium glass and pyrog. silica, Filler volume: 72 wt%=49 vol% Filler size: 0.02 to 4 μ m
3+4	MultiCore flow	Bis-GMA, urethane dimethacrylate (UDMA) and triethylene glycol dimethacrylate (TEGDMA) based matrix, Barium glass, ytterbiumtrifluoride, Ba-Al-fluorosilicate glass and highly dispersed silicon dioxide Filler volume: 70 wt%=46 vol% Filler size: 0.04 to 25 μ m
5+6	Rebilda DC	Bis-GMA, urethane dimethacrylate (UDMA) and triethylene glycol dimethacrylate (TEGDMA) based matrix, benzoylperoxide, silica, bariumborosilicate glassceramic Filler volume: 71 wt%=57 vol% Filler size: mean 1.5 μ m

Table 2 Mean ($\pm$ SD) microhardness [KHN] of the different groups (core build-up materials) at different depth levels stored in different solutions. Respective groups with odd numbers were stored in physiologic saline. The p -values of the comparisons of the microhardness at the respective depth levels are also given

groups (material)	depth	stored in physiologic saline	stored in NaOCl	p -value
1+2 (LuxaCore Dual)	0.1	43.554 (0.399)	41.530 (0.399)	<0.0001
	0.2	45.772 (0.429)	44.170 (0.398)	<0.0001
	0.3	46.952 (0.461)	46.647 (0.671)	>0.05
3+4 (MultiCore flow)	0.1	44.047 (0.263)	41.590 (0.699)	<0.0001
	0.2	45.834 (0.269)	43.696 (0.401)	<0.0001
	0.3	47.093 (0.326)	45.737 (0.295)	<0.0001
	0.4	47.943 (0.257)	47.353 (0.429)	<0.0001
	0.5	48.314 (0.226)	48.462 (0.203)	>0.05
5+6 (Rebilda DC)	0.1	44.140 (0.178)	42.493 (0.432)	<0.0001
	0.2	46.424 (0.273)	44.544 (0.323)	<0.0001
	0.3	48.025 (0.236)	46.783 (0.484)	<0.0001
	0.4	48.511 (0.217)	48.510 (0.279)	>0.05

given in Table 2.

The comparison of the microhardness of groups 1 and 2 (LuxaCore Dual) at a depth level of 0.3 mm showed no significant difference between the samples stored in physiologic saline or NaOCl ($p=0.0618$). For the groups 3 and 4 (MultiCore flow) the comparisons of microhardness until the depth level of 0.4 mm revealed significant differences between storage in physiologic

saline and NaOCl. Only the comparison at level 0.5 mm showed no significant difference between the microhardness of the samples stored in the different solutions ($p=0.0538$). There was no significant difference in the microhardness of groups 5 and 6 (Rebilda DC) at a depth level exceeding 0.4 mm ($p=0.9890$).

SEM pictures of all groups are given in Figure 1.

Fig. 1 SEM pictures of build-up materials samples (1+2: LuxaCore Dual, 3+4: MultiCore flow, 5+6: Rebilda DC). Groups with odd numbers were stored in physiologic saline. The samples were fixed in the SEM, so that they could be observed from the side walls, which were also used for the determination of the microhardness.

Samples of groups 2, 4, and 6 (samples stored in NaOCl) showed a more or less pronounced porous surface. No such surface phenomenon was observed for the samples in groups 1, 3, and 5 (samples stored in physiologic saline).

DISCUSSION

In the present study, alteration of composite build-up materials due to contact with sodium hypochlorite was tested by determination of Knoop microhardness at different depth levels. Various other studies have also determined alterations of composite restorative materials by measuring changes in microhardness^{14,18,19}. Besides measuring changes in the microhardness, other mechanical properties of resin based materials could also be tested, such as: flexural strength²⁰, surface roughness²¹ and fracture toughness²². The results of the present study indicate that the alterations due to NaOCl contact of the build-up composites are a superficial phenomenon. For testing flexural strength or other material properties usually bars of the materials are fabricated and loaded. Since the outermost layer of the materials was affected only, it is assumed that only a minimal fraction of a bar would be altered by sodium hypochlorite immersion. Thus, testing the influence of sodium hypochlorite on the flexural strength, fracture toughness or compressive stress of the materials seemed to be not appropriate. Measurements of changes in microhardness were used in this study, as this is a valid method to evaluate alterations at different depth levels of the composite materials.

In contrast to some other studies concerning alterations of composite materials^{14,23}, the samples were not stored in human saliva or artificial saliva during or after treatment with NaOCl because the present study aimed to evaluate the influence of the contact of an endodontic rinsing solution on the microhardness of build-up materials. As the use of rubber dam is indicated during root canal treatment to prevent contact of saliva with the root canal or rinsing solution with the oral cavity, no incubation in saliva was chosen to simulate the clinical situation.

This study intended to simulate the clinical situation, in which a tooth requires a build-up prior to the endodontic treatment. For this build-up, self- or dual-curing materials are widely used as they can be used in bulk application technique²⁴. This procedure presents a fast, economic and less technique sensitive approach. Furthermore, due to the self-curing mechanism, also in deep areas of the cavity, a sufficient hardening of the materials is ensured. In the clinical situation, an endodontic access cavity will be drilled through the build-up to reach the pulp or the root canals. On the walls of this cavity, which is located in the build-up, the NaOCl gets in contact with the composite. Especially in deep regions of the core build-up, an additional light curing has no additional influence on the achieved microhardness^{25,26}. In these

areas of the build-up the dominating mechanism of curing is the self-curing mode. Due to this reason, no additional photo-irradiation has been performed in the present study.

The application time of the NaOCl of 1 h was chosen to simulate the clinical situation during a complete endodontic treatment starting from the trepanation up to the obturation of the root canal with multiple rinsing with fresh NaOCl as it is commonly done during the endodontic treatment. A study²⁷ reported an irrigation time with NaOCl between 15 min and 1 h being necessary to have bactericidal effects against *F. nucleatum* and *S. intermedius*. A recent study by Retamozo *et al.* (2010)²⁸ reported an irrigation time of 40 min minimum for effective removing of *E. faecalis* from infected dentine. Furthermore, an incubation time of 1 hour or even longer has been used in different other studies^{9,29,30} concerning the effect of NaOCl irrigation on dentine micromechanical properties.

Other studies have shown an influence of sodium hypochlorite treatment on the microhardness of dentine^{10,31}. In the present study, the findings were similar for composite materials. Furthermore, the influence of sodium hypochlorite on the microhardness of composite build-up materials was confirmed not only for the surface but also for deeper layers of the restorative materials. Compared with the findings of Hannig *et al.* (2007)¹⁴, where softening of light-curing composites after contact with bleaching agents was observed even in 1 mm depth, the softening observed in the present study seems to be a more superficial phenomenon.

Sodium hypochlorite, especially in 5.25% concentration, is known as a strong bleaching agent⁴. Like other bleaching agents, its reactive chlorine forms (hypochlorite and hypochlorous acid) show high oxidizing potential³². As discussed elsewhere, substrates with oxidative capacities might cleave polymer chains, which results in a decomposing of the resin based materials¹⁴. It might be assumed that this degradation leads to a softening and reduction of the microhardness of the so affected composite materials. Another possible consideration how oxidative substrates might influence the hardness of composites is that they cause a filler matrix debonding³². These explanations provide possible reasons for the different outcomes for the different build-up composites used in the present study. When comparing the information given by the manufactures concerning the composition of the resin matrix of MultiCore flow and Rebilda DC, it seems that there is no qualitative difference but there might be a quantitative one. There seems to be a high discrepancy in the filler volume of both materials. Taking into consideration, that a higher percentage filler volume results in a lower percentage matrix volume, the difference in the findings of the present study for these materials might be explained with the different amount of matrix resin to be decomposed while in contact with the NaOCl.

Vol% of filler of LuxaCore Dual and MultiCore flow is comparable, but the results of the present study for these materials are different. The lowest softening was observed for LuxaCore Dual while MultiCore flow showed softening in even double of the depth compared with LuxaCore Dual. This might be attributed to the difference in the composition of the resin matrix of both materials. LuxaCore Dual has a resin matrix solely of Bis-GMA, while the resin matrix of MultiCore flow is composed of Bis-GMA, urethane dimethacrylate (UDMA) and triethylene glycol dimethacrylate (TEGDMA). Polymerisation of Bis GMA results in the formation of hydrogen bonds between OH- and ester-groups while the polymerisation of UDMA results in bindings between NH-groups³³. As the strength of hydrogen bonds between OH- and ester-groups is stronger than that of NH groups³³, it might be concluded, that the bond between NH groups is more susceptible to decomposition during chemical attacks like it occurs during contact with NaOCl. This might explain the higher susceptibility due to softening in the present study of MultiCore flow (Bis-GMA; UDMA; TEGDMA) compared with LuxaCore Dual (Bis-GMA only). Furthermore, this might also explain the difference in the findings for LuxaCore Dual and Rebuild DC (Bis-GMA, UDMA; TEGDMA).

CONCLUSION

The results of the present study show that contact with sodium hypochlorite on composite build-up materials causes a softening of the build-up materials. This softening is limited to the outermost layer of the materials. Alteration of the composite due to the contact with NaOCl seems not to require a complete replacement of the build-up prior to the definitive prosthetic restoration.

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