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Effect of different mechanical cleansing protocols of dentin for recementation procedures on micro-shear bond strength of conventional and self-adhesive resin cements

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ABSTRACT

Service life of debonded indirect dental restorations could be prolonged by recementation. This process requires removal of cement remnants from dentin. This study evaluated the effect of different mechanical cleansing protocols of dentin for recementation procedures on micro-shear bond strength (μSBS) of conventional and self-adhesive resin cements. The labial surfaces mandibular incisors (N=200) were ground with a low speed saw to expose the coronal dentin. The teeth were randomly divided into two subgroups (n: 100 per group) and received either (a) conventional (Panavia F 2.0, Kuraray, PAN) or (b) self-adhesive (Clearfil SA, Kuraray, CSA) resin cement. Resin cements were condensed into polyethylene molds incrementally and photo polymerized using an LED polymerization unit. Specimens were stored in distilled water at 37 °C for 24 h and subjected to μSBS (0.5 mm/min). Resin cement remnants on bonded dentin surfaces were removed using by (a) composite finishing bur (cb), (b) tungsten carbide bur (ob), (c) ultrasonic scaler tip (sc) or (d) pumice-water slurry (pw). Non-cleaned teeth acted as the control group (cn) (n: 20 per subgroup). After cleaning, the same cement type was rebonded simulating clinical recementation. Failure types were analyzed using optical microscope and Scanning Electron Microscope (SEM). Data (MPa) were analyzed using Wilcoxon Signed Ranks, Mann-Whitney U and Bonferroni tests (α=0.05). Overall, CSA (6.42 ± 2.96) showed significantly lower results than that of PAN cement (7.88 ± 3.49) ($p < 0.05$). All cleansing protocols (4.29 ± 2.17 to 5.82 ± 2.5) showed significantly lower results than that of the control group (9.84 ± 4.88) for PAN cement. For CSA cement, all cleansing protocols presented non-significant results (4.25 ± 2.74 to 6.44 ± 2.4 MPa) compared to control group ($p > 0.05$) expect cb method (3.42 ± 1.47) ($p < 0.05$). Remnants of cements were detected on dentin surfaces in all groups at varying degrees. SEM showed that while using pumice-water slurry was the least effective for PAN, tungsten carbide bur was the most effective for both cements. All other methods showed similar cleansing efficacy. None of the cleansing protocols yielded to complete removal of resin cement rest on dentin upon recementation for both cements tested.

1. Introduction

Long-term durability of indirect dental restorations is dictated by the tooth preparation geometry, internal roughness, fit of the restoration and type of the luting cement [1]. Although the debonding is one of the undesired technical complications in fixed prosthodontics, the retention loss especially for single or multiple unit resin-bonded fixed dental prosthesis (FDP) are encountered as a clinical failure between 2 and 21% around 5 years [2,3]. The abutment teeth of cantilever FDPs are more likely to face retention loss compared to conventional full-coverage ones [4]. Debonding of a restoration is initiated either from the intaglio surface of the restoration or from the prepared tooth structure [5]. When this technical failure is not related with the tooth geometry after preparation or the prosthesis itself, the reason may be due to the luting material itself [6]. In that regard, residual provisional cement on dentin surface may influence the interlocking or adhesion of the definitive cement on the dentin [1]. In a debonding situation, recementation of the debonded restoration may resume its survival without replacing the restoration or changing the preparation geometry. Although the recementation procedures seem to duplicate the initial cementation process, practitioners have to consider the adhesive properties of the cement on dentin.

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Table 1
The types, brands, main chemical compositions, manufacturers and batch numbers of the resin cements used for the experiments.

Type of resin cement	Brand	Chemical composition	Manufacturer	Batch number
Self adhesive resin cement (CSA)	Clearfil SA cement	Paste A: 10-MDP, bis-GMA, TEGMA, hydrophobic aromatic dimethacrylate, silanated barium glass filler, silanated colloidal silica, di-camphorquinone, benzoyl peroxide, Initiator Paste B: bis-GMA, hydrophobic aromatic dimethacrylate, Hydrophobic aliphatic dimethacrylate, silanated barium glass filler, silanated colloidal silica, surface treated sodium fluoride, accelerators, pigments	Kuraray, Osaka, Japan	009ADA
Conventional resin cement (PAN)	Panavia F2.0	ED Primer 2.0 A: HEMA, 10-MDP, 5-NMSA, water, accelerator ED Primer 2.0 B: 5-NMSA, water, accelerator, sodium benzene sulfinate Paste A: 10-MDP, hydrophobic aromatic dimethacrylate, hydropholic aliphatic dimethacrylate, hyrdophilic dimetacrilate, silanated silica, photoinitiator, benzoyl peroxide Paste B: Hydrophobic aromatic dimethacrylate, hydropholic aliphatic dimethacrylate, hyrdophilic dimetacrylate, sodium aromatic sulfinate, accelerator, sodium fluoride, silanated barium glass	Kuraray	041320 Paste A: 00095A Paste B: 00498A ED Primer A:00301A ED Primer B: 00175

bis-GMA,bis-phenol-A-glycidylmethacrylate; HEMA,2-hydroxyethyl methacrylate; MPD,methacryloyloxydecyl-dihydrogen phosphate; TEGMA,Triethyleneglycol methacrylate; 5-NMSA,N-Methacryloyl-5-aminosalicylic acid.

Luting cements can be classified as water-based and polymerizing cements [7]. Recently, the so-called resin cements are commonly preferred for luting FDPs as they offer several advantages such as high compressive and tensile strengths, low solubility, superior optical properties and high adhesive performance [3]. Resin cements require the use of adhesion promoters for conditioning dentin prior to their application. The adhesives used in conjunction with resin cements may be based on etch-and-rinse, two-step self-etch or one-step self-etch systems where the latter two decreases the chairside time [8]. Further developments in resin technologies introduced the self-adhesive resin cements that do not necessitate any conditioning of dentin or intaglio surfaces of the restoration [9]. While the simplicity of such cements extended their popularity, some authors observed obscure success of the retention of FDPs luted with such resin cements [10,11]. Yet, debonding problem is not completely solved and therefore recementation of a restoration may still be needed in situations where no macro-mechanical retention is available. In such occasions, residual cement may still be observed on the tooth surface, on the inner surface of the restoration or both. Thus, such residual cement needs to be removed to maintain the proper initial seat of the FDP.

Previous studies mainly concentrated on the use of various cleansing procedures for removal of provisional cement from cut dentin before cementation [12,13] or focused on the behavior of enamel to rebonding attempts [14,15]. Although the debonding of FDP was found to be a common cause of technical failures [4,16], only one study had suggested a strategy for removing the residual cement from dentin surface prior to recementation [5]. Other studies reported that residual provisional cements on prepared abutment impair the bond strength of permanent cements [17,18]. To the authors‘ best knowledge, an ideal cleansing protocol to remove the resin cement remnants from cut dentin surface has not been established to date and their effect on recementation is not known.

The objectives of this study therefore were to (a) assess the effect of different mechanical cleansing protocols of dentin for recementation procedures on micro-shear bond strength (μSBS) of conventional and self-adhesive resin cements and (b) classify the failure types after debonding.

2. Experimental

2.1. Materials and methods

Mandibular sound incisor teeth ($N=200$), extracted due to periodontal reasons, were used in this study. After calculus and tissue remnants were removed with a scaler (H6/H7; Hu-Friedy, Chicago, IL), teeth were stored in distilled water for 2 weeks. The roots were removed from the coronal parts using a diamond disc (IsoMet 1000, Buehler Ltd, USA) under water-cooling. The coronal part of teeth were embedded in a polyvinyl chloride (PVC) mold with their labial surfaces exposed using auto-polymerizing acrylic resin (Akribel, İzmir, Turkey). Dentin surfaces were exposed with a low speed disc under water-cooling and ground finished using 600, 800 and 1000-grit, silicon carbide abrasive papers under water in sequence.

The teeth were randomly divided into two subgroups (n: 100, per group) and received either (a) conventional adhesive (Panavia F 2.0, Kuraray, Osaka, Japan, PAN) or (b) self-adhesive (Clearfil SA, Kuraray, CSA) resin cement. The cements were applied according to the manufacturers' recommendations. The types, brands, main chemical compositions, manufacturers and batch numbers of the materials used for the experiments are listed in Table 1. Schematic description of the experimental design is presented in Fig. 1.

In PAN group, one drop of ED Primer A and ED Primer B were mixed and applied to the dentin surface. After waiting for 30 s, the surface was gently air-dried. Equal amount of Paste A and Paste B of the cement were mixed and inserted into the transparent polyethylene tube (inner diameter: 1 mm), positioned in the middle third of each specimen. The CSA cement did not require any conditioning of dentin. Resin cements were condensed into polyethylene molds incrementally. Each layer was photo polymerized for 40 s using an LED polymerization unit (Elipar S10, 3M ESPE, St. Paul, MN, USA) from a constant distance of 2 mm from the surface. The light output was 800 mW/cm^2 verified by a radiometer (Demetron LC, SDS Kerr, Orange, CA, USA). The specimens were then stored in distilled water for 24 h at 37 °C until the testing procedures.

2.2. Microshear (μSBS) test

Specimens were mounted in the jig of the Universal Testing Machine (Bisco, Schaumburg, IL, USA) and shear force was applied to the adhesive interface until failure occurred (Fig. 2a). The load was applied to the adhesive interface, as close as possible to the surface of the substrate at a crosshead speed of 0.5 mm/min. μSBS (MPa) was calculated by dividing the maximum load (N) by the bonding surface area of the resin cement.

2.3. Cleansing protocols and recementation

After debonding, bonding area was pinpointed to position the tubes in the same location for recementation (Fig. 2b). Resin cement remnants on the dentin surfaces were removed either by (a) composite finishing bur (cb) for 5 s at 30,000 rpm, (b) tungsten carbide bur (ob) for 10 s at 2000 rpm, (c) ultrasonic scaler tip (sc) for 10 s at 20,000 Hz or (d) pumice-water slurry (pw) for 15 s at 2000 rpm. Non-cleaned teeth acted as the control group (cn) (*n*: 20 per subgroup) (Table 2). The rotation of cleansing procedures was clockwise. A light pressure was applied under finger pressure. No loops were used during cleansing.

Dentin was conditioned for PAN as described above and for CSA no conditioning was performed. Polyethylene cylindrical tubes were positioned on previously bonded areas and resin cements were polymerized accordingly. With the indicated borders of the initial bonding areas, the recemented area at the debonded surface was overlapped (Fig. 2b). One operator performed all cleansing and bonding procedures. The specimens were then stored in distilled water for 24 h at 37 °C and μSBS was performed as described above.

2.4. Failure type evaluation

Failure sites were initially observed using an optical microscope (Stemi 2000-C, Carl Zeiss, Gottingen, Germany). Since varying degrees of resin cement were still present on all specimens, two random specimens from each group were first sputter-coated with a 3 nm thick layer of gold (80%)/ palladium (20%) and analyzed using cold field emission Scanning Electron Microscope (SEM) (LEO 440, Electron Microscopy Ltd, Cambridge, UK). Images were made at 25 kV at a magnification of x2000. The debonded enamel/dentin surfaces were first sputter-coated with a 3 nm thick layer of gold (80%)/ palladium (20%) prior to examination.

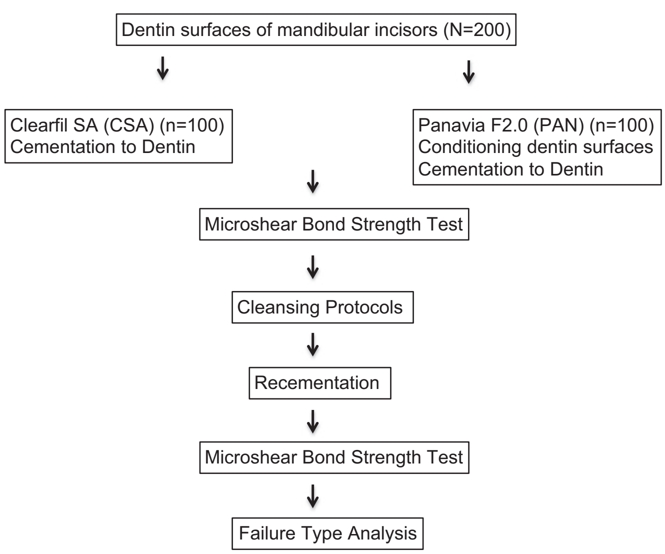

Fig. 1. Schematic description of the experimental design.

2.5. Statistical analysis

Statistical analysis was performed using SPSS 15.0 software for Windows (SPSS Inc., Chicago, IL, USA). The data were not normally distributed with inequal variance (Kolmogorov–Smirnov and Shapiro–Wilk, $\alpha=0.05$). Accordingly, Wilcoxon Signed Ranks and Mann Whitney U non-parametric analysis were carried out to find out the significant differences between groups. Multiple comparisons were made using Bonferroni adjustment. *P* values less than 0.05 were considered to be statistically significant in all tests.

3. Results

Overall, CSA (6.42 ± 2.96 MPa) showed significantly lower results than that of PAN cement (7.88 ± 3.49 MPa) ($p < 0.05$) (Table 3).

All cleansing protocols (4.29 ± 2.17 to -5.82 ± 2.5 MPa) showed significantly lower results than that of the control group (9.84 ± 4.88 MPa) for PAN cement. For CSA cement, all cleansing protocols presented non-significant results (4.25 ± 2.74 to -6.44 ± 2.4 MPa) compared to control group ($p > 0.05$) expect cb method (3.42 ± 1.47) ($p < 0.05$). (Table 4).

Remnants of cements were detected on dentin surfaces in all groups at varying degrees (Figs. 3a–d and 4a–d). Among all cleansing methods, ob provided the cleanest surface for both cements.

4. Discussion

An effective cementation protocol is crucial to increase service life of debonded FDPs where cement remnants need to be removed. For this reason, this study was undertaken to evaluate the effect of different mechanical protocols, for cleaning cement remnants from dentin surfaces, on bond strength to dentin.

In this study, bond strength was tested using μSBS test. With this method, inherent problems associated with shear test and microtensile tests could be eliminated. While the macroshear test results in cohesive failure of the substrate, not revealing the true bond strength, pretest failures or misalignment of the specimens are the other problems associated with microtensile test [19]. In μSBS test, bonded cylindrical resin cement surface is small enough to be negatively affected from such factors yielding to more reliable results. One translucent polyethylene mold filled with the cement was bonded on each dentin specimen surface. In fact, more number of molds could have been bonded on one specimen [20] but then the variation in dentin tubule orientation could have affected the results. Since the bonding area was fairly

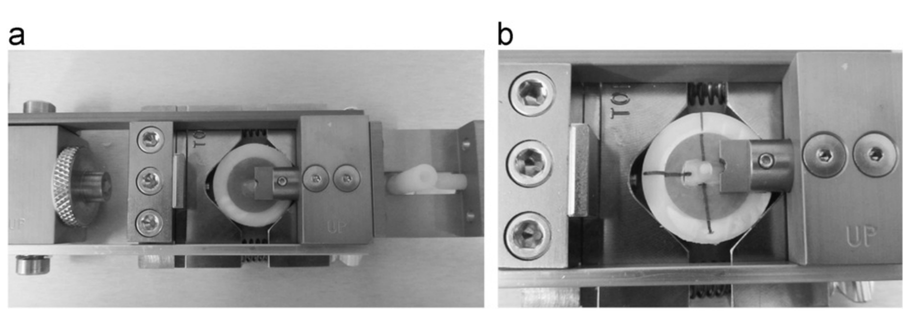

Fig. 2. (a) Position of the bonded specimen fixed in the jig of the Universal Testing Machine for μSBS testing after initial cementation, (b) the borders of the debonded area marked to overlap with the same area after recementation.

Table 2
Mechanical cleansing protocols applied on dentin prior to recementation, manufacturers of cleansing agents, application modes, abbreviations of subgroups.

Mechanical dentin cleansing protocols	Manufacturers	Application mode	Abbreviations of the subgroups (n=20)
Composite finishing bur (cb) (extra fine diamond grid)	Diatech Dental Ac, Heerbrugg, Switzerland	5 s at 30,000 rpm	CSA-cb PAN-cb
Tungsten carbide bur (ob)	Karmed Gebr. Brasseler GmbH, Lemgo, Germany	10 s at 2000 rpm	CSA-ob PAN-ob
Ultrasonic scaler tip (sc)	G3, EMS Corp., Nyon, Switzerland	10 s at 20,000 Hz with tactile pressure	CSA-sc PAN-ob
Pumice-water slurry (pw)	Imicryl, Konya, Turkey	15 s at 2000 rpm	CSA-pw PAN-pw
No cleansing (cn)	–	–	CSA-cn PAN-cn

Table 3
The mean µSBS (MPa), standard deviations, minimum and maximum values for the two cements tested. Different superscript letters in the same column show statistically significant differences ($p<0.05$).

Cement type	Mean (MPa)	SD	Min	Max
Clearfil SA	6.42^{a}	2.96	3	19.25
Panavia F2.0	7.88^{b}	3.49	2.35	16.9

Table 4
The mean µSBS (MPa), standard deviations, minimum and maximum values for the subgroups tested after recementation. Different superscript letters in the same column show statistically significant differences ($p<0.05$).

Subgroups	Mean (MPa)	SD	Min	Max
CSA-cb	3.42^{a}	1.47	1.5	6.65
CSA-sc	$4.25^{a,b}$	2.74	1.95	7.73
CSA-ob	$4.96^{a,b}$	3.28	1.08	13.09
CSA-pw	$6.44^{b,c}$	2.4	2.35	10.05
CSA-cn	$5.83^{b,c}$	2.87	2.53	12.4
PAN-cb	4.82^{d}	2.54	1.15	8.75
PAN-sc	4.54^{d}	2.58	1.3	10.12
PAN-ob	4.29^{d}	2.17	1.9	8.25
PAN-pw	5.82^{d}	2.5	2.8	10.7
PAN-cn	9.84^{e}	4.88	3.2	17.9

small, and in order to rebond the cement on the same area as the surface prior to cementation, the borders of the bonded area were indicated. Also, slightly darkish, shiny surface even after cleansing protocols enabled to realize the bond on the same surface area. The absence of dentin tubuli in the SEM images verifies that the same surface area was used prior and after recementation.

Water-based luting agents such as zinc-phosphate cements are considered to have weak bond strength to dentin [21,22]. Therefore, polymerizing resin cements were used in this study. Both resin cements tested in this study are mainly based on 10-MDP which is an acidic monomer. This monomer forms chemical bonds with calcium (Ca) around the collagen fibrils of hybrid layer [23,24]. A stable MDP-Ca bond enhances the strength of the adhesive layer [25]. Considering the two cements consist the same monomer, similar adhesive behavior could be expected. However, based on the results this could not be stated since self-adhesive resin cement, CSA, presented significantly less adhesive strength than that of conventional resin cement, PAN. The adhesive resin used in conjunction with PAN is a two-step self etch adhesive that contains hydrophilic and acidic monomers, acidic molecules, diluent monomers, photo-initiators and solvents usually at low pH [26]. Self-adhesive resin cements in turn require no adhesive, simultaneously etch the dentin and infiltrate the adhesive monomers into the dentin [27]. Cantoro et al. found no statistically significant difference in bond strengths of self-adhesive and conventional resin cements [28] but others pointed out better adhesion to dentin with conventional resin cements used in combination with self-etch adhesives compared to the self-adhesive resin cements [8]. Less favorable adhesion and more adhesive failures with CSA cement were attributed to the limited penetration and infiltration of the cement into demineralized dentin substrate [29,30]. Similarly, in this study, bond strength was less with CSA but complete adhesive failures between the dentin and resin cement were not observed with both of the cements. As the specimens were tested only after 24 h water storage, this study simulates and early recementation scenario. Maximum polymerization with these cements may take up to 24 h [31] and during this time the patients need to function and consequently early debondings may occur. The extended storage time in water or challenging the interfaces in thermocycling after initial cementation could be taken into account in future studies.

A generalized protocol about the elimination of resin cement remnants from dentin surface in repetitive cementation procedures is lacking. Cleansing protocols for removal of provisional cements have been studied [12,13,17,18,32] and categorized into mechanical and chemical methods [13]. Mechanical cleansing protocols included the use of pumice aided with rotary instrumentation, air polishing and air-abrasion with abrasive particles, the use of excavator, air-scaler and sonic toothbrush. On the other hand, chlorhexidine digluconate, sodium hypochloride, ethanol containing agents, hydrogen peroxide and polyacrylic acid applications constituted chemical cleansing agents. Excavator, air-scaler and sonic toothbrush were used for cleaning provisional cements [12]. Residual resin cements from enamel surfaces after debonding orthodontic brackets were suggested to be removed using burs, stones, discs, polishing cusps and laser applications [33,34]. One of the most common and cheapest medium is pumice-water slurry to remove cement remnants from tooth surface [35]. Considering these studies, multiple mechanical cleansing protocols such as composite finishing burs, tungsten carbide burs, ultrasonic scaler tip and pumice-water slurry were tested for removal of resin cements.

According to the SEM images of all protocols, pumice-water slurry application with rotary instruments showed the worst efficiency in PAN removing but displayed similar removal of cement remnants in CSA. In a previous study, toothbrush abrasion of CSA was studied and wear resistance of this cement was found comparable to conventional resin cements [29]. However the filler size of resin cement can affect the wear resistance of the material and thereby its removal using a mechanical medium [36]. The average filler size of CSA resin cement is about 2.5 µm [8,30] and the particle size of the fillers of PAN ranges from 0.4 to 19 µm according to the manufacturers' information. One reason for inefficacy of pumice-water slurry might be due to larger filler size of PAN where nearly 10 µm fillers were evident in the SEM

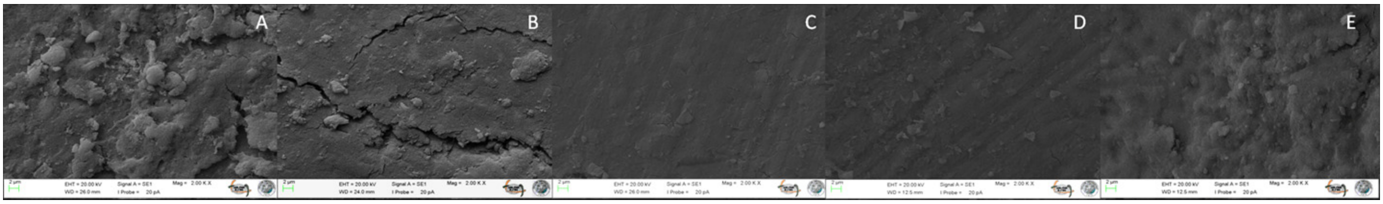

Fig. 3. SEM micrographs (× 2000) belonging to CSA group after cleansing protocols; (a) cb, (b) sc, (c) ob, (d) pw, (e) cn. Note the cleanest surface after ob protocol. See Table 2 for group abbreviations.

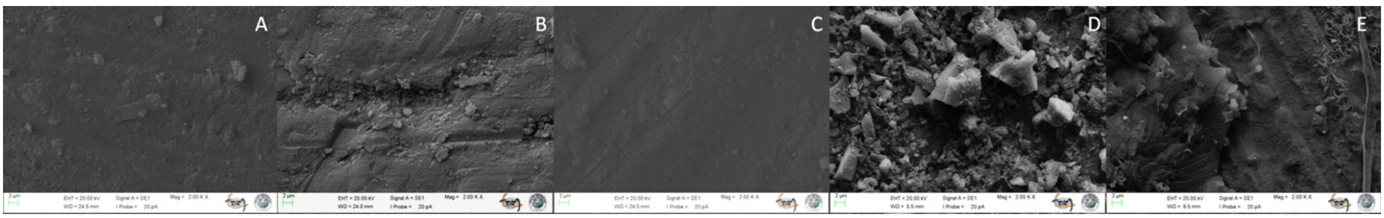

Fig. 4. SEM micrographs (× 2000) belonging to PAN group after cleansing protocols; (a) cb, (b) sc, (c) ob, (d) pw, (e) cn. Note the substantial resin cement remnants after pw cleansing method and the cleanest surface after ob protocol. See Table 2 for group abbreviations.

images. Santos et al. have reported negative effect of the use of pumice on the bond strength of the resin to fresh dentin due to obscuration of the dentinal tubule openings by pumice residues [37]. In the present study, pumice-water slurry was applied on the cement surface where the dentinal tubules were impregnated by the resin that has a different implication. Yet, this protocol did not significantly affect the μSBS compared to the control group and not totally eliminated the micromechanical interlock between dentin-resin interface in CSA. Since there were some dominant remnants of the PAN on the surface, the second cementation was possibly performed on the cement layer that altered the substrate properties. The obtained results were therefore a kind of resin–resin bond. This can be stated for all groups based on the SEM findings. In that respect, recementation procedures may be compared with the resin coating techniques or immediate dentin sealing where a bonding agent and low-viscosity resin is applied on freshly prepared tooth structure in order to hybridize the dentin surface [38]. With this route, the bond strength of the final polymerized resin is enhanced. Shafiei et al. have demonstrated the resin coating efficiency for PAN cements [39]. According to their findings, resin coating of ED primer reduced the marginal microleakage of the restorations after 6 months. On the other hand, surface roughness of the existing composites and available free monomers, activated by a bonding agent increased the bond strength in composite repair studies [40,41]. In this study, no bonding agent was applied after cleansing for recementation protocols. This could have increased the results but clinically this attempt would interfere with the fit of the restorations.

For both cements tungsten carbide bur at 2000 rpm was more effective optically but not necessarily for bond strength. Prolonged duration of mechanical cleaning could result in dentin exposure but this may then decrease the fit of the restoration. For this reason, short application duration was employed. The rpm chosen represents the rpm of rotating instruments in clinical dentistry. From the design point of view, while the composite finishing bur fits to the aerator, tungsten carbide bur in the micromotor. Eventually, the rpm chosen was different for these two burs and the ultrasonic scaler depending on the working mechanism of the rotating instruments. Application of burs was restricted to 5 s for high rpm in order to avoid heat transmission to dentin due to prolonged friction. Future studies should look at the effect of time-dependent cement removal in order to optimize the cleansing protocols and investigate the reactivation of the surface with an adhesive resin bearing in mind that the cement remnants were not completely removed.

5. Conclusions

From this in vitro study, the following conclusions were drawn

1. In control group, recementation with self-etch cement presented less favorable adhesion than that of conventional resin cement used in association with a two-step self-etch adhesive.
2. In terms of bond strength, while for self-etch cement only the effect of using composite finishing bur for 5 s at 30,000 rpm was less efficient than those of other methods tested, for conventional resin cement all cleansing protocols tested showed similar cleansing efficacy.
3. None of the cleansing protocols could remove cement remnants completely from dentin surfaces for both cements tested.

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