A novel alumina-ceria polishing paste demonstrated superior polishing of lithium disilicate compared with diamond paste

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The aim of this study was to develop a polishing paste containing ceria to polish lithium disilicate. The samples were prepared, polished with sandpaper using a polishing-machine, the surface roughness (Ra) was measured using a profilometer and randomly divided into 7 groups (n=10). The control group was polished with diamond paste (D). The 6 remaining groups were polished with alumina-ceria paste with different ratios of deionized water:alumina:ceria by weight: 1:0.5:0.5 (AC0.5), 1:0.5:1 (AC1), 1:0.5:1.5 (AC1.5), 1:0.5:2 (AC2), 1:0.5:2.5 (AC2.5) and 1:0.5:3 (AC3). The specimens were polished for 30 s and their Ra values were determined. The surface roughness measurement was repeated after an additional 30 s of polishing until 120 s of polishing had been performed. The Ra values decreased as the ratio of ceria increased. The surface morphology of the samples analyzed using scanning electron microscopy corresponded with their Ra values.

Keywords: Cerium oxide, Chemical mechanical polishing, Diamond paste, Lithium disilicate, Polishing paste

INTRODUCTION

The use of all-ceramic restorations has become popular, especially at the aesthetic zone. One of these materials is lithium disilicate-reinforced ceramic. IPS e.max CAD is a chair-side monolithic restorative material with high strength and aesthetics produced to eliminate applying veneering porcelain, which is the weak point of layered ceramic restorations, and to finish the treatment in one visit. However, lithium disilicate-reinforced ceramic requires a final adjustment before permanent fixation of the restoration, resulting in a rough surface. A rough surface can lead to partial chipping or fracture and affect the strength of the ceramic. A previous study reported that the fracture strength of IPS e.max Press decreased with increasing surface roughness. Moreover, an inadequately polished surface increases the wear of the adjacent and opposing tooth and increases biofilm accumulation, which leads to periodontal tissue inflammation.

Polishing or glazing are used to restore the smooth and shiny surface of the restoration. Reglazing involves repeated firing in the dental laboratory, which requires more patient visits. Polishing is an easy way to establish a smooth surface in a single visit. There are many polishing modalities used to polish restorations. Previous studies reported that polishing produces as smooth or smoother surface, and gave a higher gloss and higher translucency compare with glazing.

The polishing methods mentioned above are purely mechanical process. However, there is a polishing method that combines mechanical and chemical processes together, known as chemical mechanical polishing (CMP). CMP is a process where mechanical and chemical actions complement each other to improve the material removal rate; therefore, the abrasive used in CMP must chemically react with the surface of the polished material. The aim of CMP is to eliminate direct material removal by mechanical abrasion or chemical etching. The scratching and corrosion caused by abrasion and etching, respectively, damage the polished surface.

Cerium oxide (CeO$_2$, ceria) has been used as a polishing compound from the last decades. It is considered the most effective abrasive used for polishing precision optical components because cerium oxide chemically reacts with silica on the glass surface. The chemical and mechanical process that occurs between cerium oxide and silica has been investigated. Cook hypothesized that the surface chemical process plays an important role in the material removal process during CMP. He proposed that metal oxides in the abrasive, such as cerium oxide, form surface functional groups in an aqueous environment. The material removal comes from the temporary attachment of silica on the glass surface during polishing to the ceria (cerium oxide) particles, forming a layer. This layer is removed by abrasives plowing the slurry particles and exposes an unreacted surface on which a new layer is formed and removed. Suphantharida and Osseo-Asare reported that silicate ions are chemically bonded or chemisorbed to the cerium oxide surface.

Silica (SiO$_2$) is the main component in glass and lithium disilicate ceramic, and cerium oxide (CeO$_2$) is the best material with chemical mechanical process in glass polishing. However, the use of a cerium oxide-incorporated paste in polishing lithium disilicate has not been reported.

A previous study reported that polishing feldspathic porcelain with a polishing paste composed of alumina...
and zirconia as abrasives was more effective than diamond paste. We performed a pilot study with lithium disilicate polished with diamond paste, alumina paste, cerium oxide or alumina ceria paste. The Ra values of the post-polishing groups were significantly lower ($p<0.05$) compared with the baseline roughness. The Ra values were significantly different between the groups ($p<0.05$) (cerium oxide paste>alumina paste>alumina ceria paste>diamond paste). These results indicated that cerium oxide paste alone can polish lithium disilicate. Moreover, using cerium oxide combined with alumina generated a significantly smoother surface, however, not as effectively as diamond paste ($p<0.05$). Thus, the aim of the present study was to develop a new CMP paste containing cerium oxide with the optimum alumina ceria paste formulation that would be as or more effective than diamond paste. The null hypothesis was that the surface roughness of the lithium disilicate polished with alumina ceria polishing paste would not be significantly different from that polished with diamond paste.

MATERIALS AND METHODS

Specimen preparation

Seventy-two samples were used in this study. The sample size calculation was performed using the data from our pilot study. Pre-sintered lithium disilicate blocks (HT A1, IPS e.max CAD, Ivoclar Vivadent, Schaan, Liechtenstein) were cut into 7 mm long, 6 mm wide, and 5 mm thick samples using a low speed precision cutting machine (IsoMet, 1000 No. 11-2180, Buehler, Lake Bluff, IL, USA) and ultrasonically cleaned with deionized water for 5 min (CP360 Powersonic, Crest Ultrasomics, Ewing Township, NJ, USA), rinsed with deionized water and dried. The pre-sintered samples were fired in a furnace (Programat P300, Ivoclar Vivadent) per the manufacturer’s directions. After firing, the samples were cooled in the furnace. Each sample was embedded in a polyvinyl chloride pipe with epoxy resin using a plastic template to set the position of the sample at the center on the pipe’s surface. After the epoxy resin had completely hardened, a registration mark was made at the bottom of the pipe (4 mm wide and 6 mm deep) to allow the specimen to be aligned at the same position during multiple roughness measurements (Fig. 1).

The specimens (6 specimens/round) were polished for 5 min with 180 grit silicon carbide sandpaper (3M Wetordry abrasive sheet, 3M, St. Paul, MN, USA) by a polishing machine with an automatic head (NANO 2000 grinder-polisher with FEMTO-1000 polishing head, Pace Technologies, Tucson, AZ, USA). During polishing, the sandpaper was rotated 200 rpm clockwise and the samples were rotated 200 rpm counterclockwise. The force applied on the specimens was set at 1 kg/cm². New sandpaper was used each round. The polished specimens were ultrasonically cleaned in deionized water for 5 min, rinsed with deionized water and dried.

The surface roughness ($Ra$) of the specimen surface was measured using a profilometer (Talyscan 150,
Taylor Hobson, Leicester, UK) to determine the baseline roughness. Five 2 mm measurements were taken at the center of the specimen sample (cut-off value of 0.25 mm and stylus speed of 0.5 mm/s). The vertical distance between each transverse measurement was 0.4 mm. The sample was then rotated 90° and re-measured using the same procedure. The Ra values were averaged to generate a mean Ra value per sample.

After the baseline roughness evaluation, the samples were randomly divided into 7 groups (n=10) based on the different polishing pastes. The polishing paste compositions used to polish each group are shown in Table 1.

Polishing paste preparation
Deionized water was used as a lubricant in this study. Two types of abrasive particle were used in the present study: alumina particles (Alumina powder, 1 µm diameter particles, LECO, St. Joseph, MI, USA) and cerium oxide particles (Cerium oxide powder, <5 µm diameter particles, 99% trace metals basis, Sigma-Aldrich®, Merck, Darmstadt, Germany). Diamond paste (Diamond polishing paste, 1 µm diameter particles, Jota, Ruthi, Switzerland) was used as the abrasive in the D group. The remaining groups were prepared using different ratios of deionized water:alumina:ceria by weight: 1:0.5:0.5 (AC0.5), 1:0.5:1 (AC1), 1:0.5:1.5 (AC1.5), 1:0.5:2 (AC2), 1:0.5:2.5 (AC2.5) and 1:0.5:3 (AC3). The polishing pastes were prepared by weighing the components to within 0.0001 g using an analytical balance (GR 200, A&D, Tokyo, Japan) based on each group's composition and mixed using a spatula for 5 min. The mixtures were loaded into a syringe (0.1–1 mL scale) (Slip-tip disposable tuberculin syringe, Medline Industries, Northfield, IL, USA). The polishing pastes were used within 12 h.

Polishing method
Each polishing paste (0.05 mL) was injected on the center of the specimen and then polished using a felt wheel (2.2 mm diameter, Felt wheel, Jota) on a micromotor (Micromotor and handpiece, Saeyang microtech, Daegu, Korea) for 30 s and ultrasonically cleaned in deionized water for 5 min, rinsed with deionized water, and dried. A new felt wheel was used for each group. The revolution speed was set at 6,000 rpm, calibrated using a tachometer (Digital tachometer, RS components, Corby, UK). The polishing pressure was 40 g force. The operator was calibrated using a precision scale before and during the procedure. The calibration was repeated every 10 specimens. All polishing procedures were performed by one operator. After polishing, the specimens were ultrasonically cleaned in deionized water for 5 min, rinsed with deionized water, and dried.

After polishing, the surface roughness of the specimen was measured using a profilometer as described for the baseline roughness measurement. After the measurement, the samples were ultrasonically cleaned in deionized water for 5 min, rinsed with deionized water and dried.

The surface roughness measurement was repeated after an additional 30 s of polishing until 120 s, i.e. measured after 30, 60, 90 and 120 s of polishing, of polishing had been performed.

Surface roughness measurement
The surface roughness after polishing was measured using a profilometer as described above with the samples placed at the same position as at the baseline roughness measurement.

Scanning electron microscopy (SEM) analysis
Two samples from each post-polishing (120 s) group and two unpolished samples with the baseline roughness were removed from the epoxy resin and ultrasonically cleaned in deionized water for 5 min, rinsed with deionized water and dried. The specimens were mounted onto adhesive-coated aluminium stubs (1 sample/stub) and gold sputter-coated (100 s, 50 mA) using a sputtering device (JFC-1200 Fine Coater, JEOL, Tokyo, Japan). The surface images were taken using an electron microscope (QuantaTM 250 FEG scanning electron microscope, FEI, Hillsboro, OR, USA) with 20 kV accelerating voltage and 350× magnification to evaluate the surface morphology.

Statistical analysis
The data were statistically analyzed using two-way repeated ANOVA followed by Bonferroni correction to compare the differences in mean Ra values between groups at the 95% confidence level (SPSS version 22.0 for windows, SPSS, Chicago, IL, USA). p-Values<0.05 were considered significant.

RESULTS
We determined the mean Ra value, standard deviation and significant differences between the groups (Table 2). The mean Ra values were not significantly different between the groups at baseline (p>0.05). Within each group, the mean Ra values significantly decreased as the polishing time increased (p<0.05, Fig. 2). After polishing for 120 s, the AC2.5 group demonstrated the lowest mean Ra value and was significantly lower than that of the D group (p<0.05) however, there were no significant differences in mean Ra value between the D, AC2 and AC3 groups.

Figure 3 illustrates the relationship of the Ra values between the sample groups polished with different ratios of alumina ceria pastes at the different polishing times. Increasing the cerium oxide in the polishing paste resulted in decreased Ra values, except in the AC3 group. There are no significant difference between the AC3 and AC2.5 groups after polishing for 30 s and the AC3 and AC2 groups after polishing for 120 s.

The surface roughness of the samples observed by SEM (Fig. 4) correlated with their Ra values. The surface of the sample at baseline was the roughest (Fig. 4). After polishing, the AC2.5 group had a smoother surface compared with the other groups (Fig. 4).
DISCUSSION

The present study evaluated whether a polishing paste containing cerium oxide as an abrasive was as effective as diamond paste for polishing lithium disilicate. The results indicated that the alumina ceria polishing paste created a smoother surface with a significantly lower ($p<0.05$) Ra value compared with the diamond paste. Based on these results, the null hypothesis was rejected.

Within each group, the mean Ra values significantly decreased in a polishing time-dependent manner ($p<0.05$). These results corresponded to those of a previous study that found that longer polishing time created a superior surface finish\textsuperscript{23}. Furthermore, between the groups, the Ra value decreased as the cerium oxide ratio in the polishing paste increased, except for the AC3 group. The AC2.5 group had the lowest mean Ra value. These findings were similar to those of a previous study that demonstrated that increasing the polishing slurry concentration resulting in decreased roughness\textsuperscript{24}.

A prior study suggested that more than one
paramater should be used to evaluate the surface characteristics of dental restorations\(^{25}\). Because the Ra value is a quantitative parameter, SEM was chosen to be a qualitative parameter in the present study. The SEM images of the post-polishing groups after polishing for 120 s correlated with the Ra values. The AC2.5 group, which had the lowest Ra value, demonstrated the smoothest surface and AC0.5 group, which had the highest Ra value, demonstrated the roughest surface. Moreover, every post-polishing group exhibited a smoother surface compared with baseline.

Cerium oxide is considered the most effective abrasive used in glass polishing because it chemically reacts with silica on the glass surface. Silica is also the main component in lithium disilicate. Lithium disilicate (Li\(_{2}\)O\(_{5}\)Si\(_{2}\) or Li\(_{2}\)O\(\cdot\)2SiO\(_{2}\)) is composed of ~70% by volume of lithium disilicate crystals that are embedded in a glassy matrix. The composition of lithium disilicate-reinforced ceramic as described by Ivoclar contains 57.0–80.0% SiO\(_{2}\), 11.0–9.0% Li\(_{2}\)O, 0.0–13.0% K\(_{2}\)O, 0.0–11.0% P\(_{2}\)O\(_{5}\), 0.0–8.0% ZrO\(_{2}\), 0.0–8.0% ZnO, 0.0–5.0% Al\(_{2}\)O\(_{3}\), 0.0–5.0% MgO and 0.0–8.0% colouring oxides\(^{26,27}\). The chemical reactions during glass polishing have been investigated. The reaction between silica and water is that the siloxane bond is hydrolyzed to form hydrated silica on the glass surface (1).

\[
\equiv\text{Si–O–Si} \leftrightarrow 2\equiv\text{Si–OH} \quad (1)
\]

Furthermore, in an aqueous environment, cerium oxide forms functional groups on the surface of its particles (2).

\[
\text{CeO}_2 + 2\text{H}_2\text{O} \rightarrow \text{Ce(OH)}_4 \quad (2)
\]

The hydroxyl groups from (2) then attach to the hydrated silica from (1) and form a chemical bond with the glass surface (3).

\[
\equiv\text{Si–OH}+\text{HO–Ce} \rightarrow \equiv\text{Si–O–Ce}+\text{H}_2\text{O} \quad (3)
\]

During polishing, the mechanical force interacts with the cerium oxide particles and increases the strain on the \(\equiv\text{Si–O–Ce}\) bond. Because the bond strength in the \(\equiv\text{Si–O–Ce}\) complex is higher than the \(\equiv\text{Si–O–Si}\) complex, when the strain is high enough, the bond between \(\equiv\text{Si–O–Si}\) and \(\equiv\text{Si–O–Ce}\) is broken. The \(\equiv\text{Si–O–Ce}\) complex is removed from the glass surface (4).

\[
\equiv\text{Si–O–Si–O–Ce–O–Ce}+\text{H}_2\text{O} \rightarrow \\
\equiv\text{Si–OH}+\text{HO–Si–O–Ce–O–Ce} \quad (4)
\]

This results in a new unreacted surface being exposed and the CMP process cycle repeats\(^{19,20,28,29}\). A previous study reported that the polishing compounds used in glass polishing depended on the presence of water. CMP rarely occurred when using hydrocarbon liquids or liquids without hydroxyl groups as a lubricant\(^{19}\). The polishing rates increased directly with increased hydroxyl reactivity. Thus, water, which has the greatest hydroxyl reactivity, was used as the lubricant in this study.

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**Fig. 4** Scanning electron microscope photographs (350×) of the sample surfaces polished with different polishing pastes, A: surface before polishing, B: AC0.5, C: AC1, D: AC1.5, E: AC2, F: AC2.5, G: AC3 and H: D.
There are 3 models of chemical mechanical synergy during the CMP process. The first approach is based on chemistry-assisted mechanical abrasion. The second is an approach based on mechanism-assisted chemical effects. The third approach is the chemically-formed layer is low ratio is based on chemistry-assisted mechanical abrasion\(^{24,30}\). Stein et al. demonstrated that the polishing rate is a function of the polishing slurry (a suspension of abrasives in water) concentration\(^{31}\). Increasing the concentration results in an increased removal rate. The reaction rate between silica and cerium oxide is slow and limits the overall polishing rate. Increasing the oxidizer (cerium oxide) concentration leads to increased chemical reactions that are involved in the polishing rate. At high concentration, the chemical reaction is relatively quick; however, the polishing process is limited by the mechanical process, because the fast chemical reaction results in a large amount of chemical complexes that need to be removed by the abrasive plowing of the slurry particles, which is the mechanical process, then the new unreacted surface site is exposed\(^{30}\). Furthermore, increasing the cerium oxide ratio to 3 by weight increases the polishing paste viscosity. This high viscosity results in a negative pressure that hinders the particles from contacting the polishing surface. Moreover, a high ratio of the cerium oxide particles decreased the load per particle. These factors explain the results of the present study where increasing the cerium oxide ratio to 3 by weight did not decrease the surface roughness\(^{24,30,31}\).

The factors that have an effect on CMP are abrasive type, size and shape, load during polishing and polishing speed (number of revolutions per time)\(^{30,32-36}\). However, in this study, only 2 abrasives were used and the other factors were fixed as the controlled variables. Moreover, the previous study reported that pH, point of zero charge and temperature also had an effect on the CMP\(^{30}\). To improve the efficiency of the polishing paste, these factors require further investigation.

Our results have practical implications for clinicians. We found that the mean Ra value of the D group after polishing for 120 s and the AC2.5 group after polishing for 90 s were approximately equal. Thus, if the dentist wants to acquire the same roughness as diamond paste, using an alumina ceria polishing paste with a ratio of deionized water:alumina:ceria of 1:0.5:2.5 by weight could save chair time. Moreover, alumina and ceria abrasives are more cost effective than diamond paste. Clinician can make the alumina ceria polishing paste by mixing the compositions according to the ratio mentioned above. Furthermore, the consistency of the mixture is not too viscous so it is easy to load into a syringe before using. For chairside use, loading the paste into a syringe makes it easier to carry the paste onto the restoration. Because the present polishing paste is a loose abrasive type, a soft foam or felt wheel is advised to be used as a polishing paste applicator\(^{37}\). However, the mixture needs to be stored in closed container to prevent the deionized water from evaporating. The syringe tip also needs to be capped after being used.

The limitation of this study is that it was performed in vitro. The efficiency of a polishing paste might be different under clinical conditions. Moreover, different results might be expected with different polishing protocols and types of material. Further study is needed to evaluate the surface roughness of other ceramic materials and other polishing protocols.

**CONCLUSION**

Using an alumina ceria polishing paste with a ratio of deionized water:alumina:ceria of 1:0.5:2.5 by weight to polish lithium disilicate results in a smooth surface. This polishing paste generates a significantly lower Ra value and a smoother surface compared with diamond paste. Thus, an alumina ceria paste could be an alternative to diamond paste in polishing lithium disilicate.

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