Effect of a cordless retraction paste material on implant surfaces: an *in vitro* study

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Abstract: Cordless retraction paste material for gingival retraction in implant dentistry has recently become of interest to the clinician. However, few studies have been conducted on the use of retraction pastes and their possible interaction with implant surfaces. This *in vitro* study evaluated the effect of a cordless retraction paste material, Expasyl® (Acteon), on TiUnite® (Nobel Biocare) implant surfaces. Three areas of the fixtures were evaluated before and after contact with the retraction paste using scanning electron microscopy to evaluate changes in surface topography and energy-dispersive spectroscopy to identify any surface chemistry modifications. Alteration of the initial surface after exposure to Expasyl® was identified, with the implant collar showing the most changes.

Descriptors: Dental Impression Materials; Dental Impression Technique; Dental Implants.

Introduction

Gingival retraction is commonly used in fixed prosthodontics to expose margins of abutments before impression-taking. Proper exposure of the margins without irreversible damage to the gingival tissues is essential to provide a long-term successful restoration.[1]

Conventional gingival retraction cord techniques are often used for the cement-retained implant restoration,[1] even though there is little research available on the use of retraction techniques around implants. In implant dentistry the retraction process should not only expose the abutments' margins atraumatically but should also not alter the implant surface. Surfaces of the implant could be altered either mechanically with cord packing or chemically with chemical adjuncts.

The surface of dental implants is known to play a key role in the success of osseointegration. During implantation, oxidation of the implant surfaces results in the production of free radicals and oxygenated derivatives on the titanium surface, which lead to the thickening of a titanium oxide (TiO_2) layer. Calcium and phosphorus ions from the bone matrix are then incorporated within the porous titanium oxide layer, making the bone/implant interface highly dynamic. This oxide layer is critical to successful ossseointegration.[2] Conversely, the contamination or destruction of the oxide layer can lead to the pathological loss of osseointegration that could be considered a risk factor for peri-implantitis. Alterations of the implant surface during the retraction process could trigger an

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inflammatory response.[3]

Since the architecture of the gingival crevice surrounding implants is more fragile than the structure around natural teeth, using a cordless retraction paste for gingival retraction in implant dentistry appears to have considerable potential to afford a less traumatic procedure, compared to the other available techniques.[4]

This *in vitro* study evaluated the effect of a cordless retraction paste material, Expasyl® (Acteon), on TiUnite® (Nobel Biocare) implant surfaces. The surface morphology was investigated using scanning electron microscopy (SEM), and the elemental analysis was performed using energy-dispersive X-ray spectroscopy (EDS).

Methodology

Implant samples & retraction paste

Five identical sterile NobelReplace TiUnite® implants (4.3 × 13 mm, Nobel Biocare, Goteborg, Sweden) were used. All implants were equipped with a sterile abutment to facilitate their handling. A gingival retraction paste, Expasyl® (Acteon, Bordeaux, France), was applied (0.125 cm³) to three different areas on each implant: the collar (C), the junction of collar and microthread (JC) and the microthread (MT) itself, using a handgun applicator (Figure 1). Each abutment had a distinct notch carved for precise positioning of the retraction paste and consistent observation of the surfaces exposed to Expasyl®. Two exposure times were selected: one minute (Group 1) as per manufacturer instructions and ten minutes (Group 2) to simulate possible incomplete removal of the paste after rinsing. Each of these groups included two rinsing protocols: 1 and 2 rinses with distilled water and air spray set at 50 PSI for two minutes to remove Expasyl®. EDS was also performed on the retraction paste alone to confirm its elemental components (Table 1).

To reduce environmental contamination, implants were opened only at the start of the experiment and handled with sterile gloves and sterile titanium pliers contacting the abutment only and stored in air-tight containers to limit any further contamination. The implant surfaces were in contact only with Expasyl® at any given time.

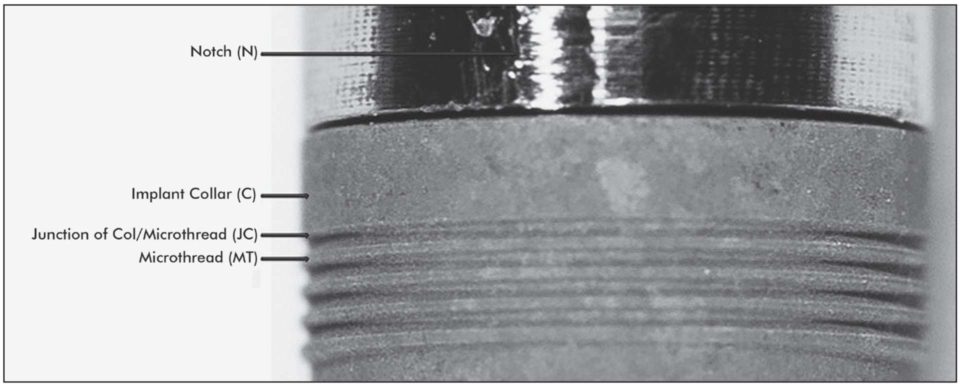

Figure 1 - Implant surfaces on which the paste was applied.

Table 1 - Study outline.

Implant Distribution		Contact with Expasyl®	Rinse cycles	Before contact with Expasyl®	Immediately after contact	After 1, 2 and 4 weeks storage
Control	Implant 1*	N/A	N/A	SEM/EDS	N/A	SEM/EDS
Group 1	Implant 2*	1 minute	1	SEM/EDS	SEM/EDS	SEM/EDS
	Implant 3*	1 minute	2	SEM/EDS	SEM/EDS	SEM/EDS
Group 2	Implant 4*	10 minutes	1	SEM/EDS	SEM/EDS	SEM/EDS
	Implant 5*	10 minutes	2	SEM/EDS	SEM/EDS	SEM/EDS

* The collar (C), collar/microthread junction (JC) and microthread (MT) areas were investigated for each implant.

Surface topography and chemistry

A scanning electron microscope (SEM) (JEOL 6700F Field Emission SEM, Tokyo, Japan) was used to reflect surface morphology of the implant sites before and after Expasyl® application. An accelerating voltage of 5kV was selected for SEM analysis and the vacuum pressure was maintained below 9.63×10^{-5} Pa. Local compositional differences across the surface were observed in the backscattered electron mode (BSE). The same magnification and working distance were selected for all implants for direct comparison of surface morphology.

EDS was used to analyze the surface chemistry composition. Survey EDS data was acquired over an accelerating voltage of 15kV. X-rays were irradiated on the implant directly beneath the notch made on the abutment to ensure consistent observation of the areas previously exposed to Expasyl®.

The implant surfaces were examined before and immediately after contact with the retraction paste. The surfaces were then re-examined one-week, two weeks and four weeks after initial contact with Expasyl®.

Description of the surface topography was carried out with semi-quantitative EDS analysis for each sample. Field emission scanning electron microscope (FESEM) and EDS operating conditions were kept constant for each sample and as close to 1500 counts per second as possible. A calibration standard was used before each session to monitor the calibration of the system. The JEOL 2300F EDS software system (JEOL Ltd, Tokyo, Japan) was used to apply a ZAF correction factor to the data. This takes into account the atomic number effect (Z), the X-ray absorption effect (A), and the X-ray fluorescence effect (F) of a sample. This correction allows a more accurate amount of each element present in the sample to be calculated.

Statistical analysis

The data obtained from the surface chemical analysis of each implant was analysed by three-way analysis of variance (ANOVA) using the StataV11 statistical program (StataCorp, College Station, USA). The number of rinses, the exposure times and the three areas (C, JC and MT) were the selected factors. Separate analysis was performed for each sample for the different periods. $P < 0.05$ was considered statistically significant.

Results

Expasyl® composition

Chemical evaluations of Expasyl® showed high elements of aluminium (Al), silicon (Si), oxygen (O) and chloride (Cl), with traces of iron (Fe) present. The main constituents are Al and Si.

TiUnite® surface

Analysis of the TiUnite® surface on a nanometer scale revealed a porous structure with numerous craters created during the breakdown phenomenon of anodic oxidation produced by the manufacturer. Elements of Ti, O, C and a small amount of phosphorous (P) were present on the TiUnite® implant surfaces.

Surface topography

Under SEM low magnification (×500), no difference could be seen on the surface before and after contact with the retraction paste. However, in BSE mode, composition contrast was evident with "white particles" observed on the implant surfaces. Local analysis via EDS showed these "residual deposits" to be composed of Al and Si. SEM images at higher magnification (×5000) showed more surface alterations in the form of similar deposits on the implant surfaces (Figure 2).

Surface chemistry

EDS showed traces of Al and Si on the implant surfaces after exposure to the retraction paste (Figure 3). Traces of Al and Si were identified on the implant surfaces following both the one- and ten-minute exposure times. After the one-minute exposure time to Expasyl® and the one rinse cycle, surface alterations were seen in all areas. Implant surfaces with two rinses showed surface modifications only in the C and JC areas (Table 2). For the ten-minute exposure time, elemental surface alterations were observed in two areas (C, JC) after one rinse cycle, but only on the implant collar after two rinse cycles (Table 3). Within group 2, traces of Al remained at

1, 2 and 4 weeks on the implant collar after two rinses (Figure 3).

The Si component was significantly different between the two groups for both the number of rinses and the time of exposure of the implant surface immediately after Expasyl® application ($P < 0.05$). The difference remained significant at the 1-week and 4-week observation periods. Similarly, significant differences were observed for Al between 1 and 2 rinses immediately after application for both groups ($P = 0.05$). The different areas showed significant differences at 1 week ($P = 0.006$) and at 2 weeks ($P = 0.01$) after exposure to Expasyl®, with both groups displaying a considerable change immediately after contact with Expasyl® ($P = 0.02$).

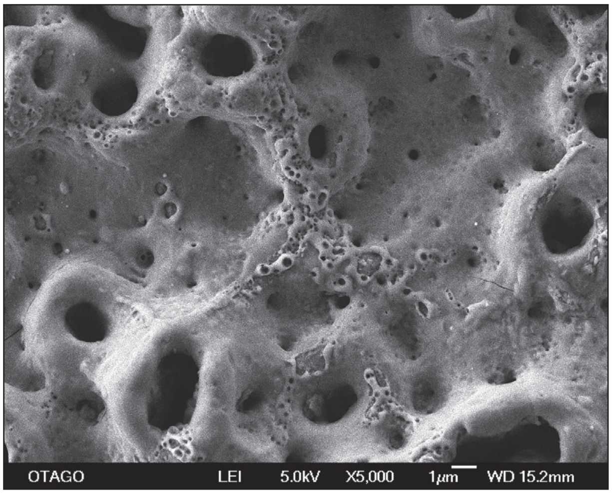

Figure 2 - SEM image at ×5000 magnification shows deposits of the retraction paste on the surface.

Both groups showed significant changes in the P element for the different areas immediately after application ($P = 0.01$), and at 2 and 4 weeks ($P = 0.05$). A similar trend was found for the Ti element. Significant changes for the different areas were observed at 1 week ($P = 0.04$), 2 weeks and 4 weeks after Expasyl® application ($P = 0.01$). Overall, the C area showed more surface alterations compared to the JC and MT areas of the implant, with the MT area being the least affected area in both groups.

Table 2 - EDS of Group 1 with 1 rinse for the C, JC and MT implant areas.

Group 1 – 1 Rinse															
	Before contact			After contact			1 week			2 weeks			4 weeks		
Area	C	JC	MT	C	JC	MT	C	JC	MT	C	JC	MT	C	JC	MT
C*	1	1	1	1	1	2	1	1	1	1.5	1	1	1	1	1
P_2O_5*	14	13	15	16	13	14	14	14	14	16	14	14	13	12	13
TiO_2*	85	87	86	83	85	83	84	85	85	82	85	85	86	86	86
Al**	0	0	0	**0.3**	**0.6**	**0.5**	**0.3**	0	0	**0.3**	0	0	**0.1**	0	0
Si**	0	0	0	**0.2**	**0.4**	**0.5**	**0.2**	0	**0.2**	**0.35**	0	0	**0.1**	0	0

* Elements originating from Expasyl®; ** Elements originating from NobelReplace TiUnite®.

Table 3 - EDS of Group 1 with 2 rinses for the C, JC and MT implant areas.

Group 1 – 2 Rinses															
	Before contact			After contact			1 week			2 weeks			4 weeks		
Area	C	JC	MT	C	JC	MT	C	JC	MT	C	JC	MT	C	JC	MT
C*	1	1	1	1	1	2	1	1	1	1	1	1	1	1	1
P_2O_5*	16	14	14	14	13	14	16	14	15	14	14	14	13	13	12
TiO_2*	84	85	85	85	86	85	83	85	84	84	85	85	85	86	87
Al**	0	0	0	**0.3**	**0.25**	0	**0.3**	**0.3**	0	**0.4**	**0.1**	0	**0.15**	**0.1**	0
Si**	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0

* Elements originating from Expasyl®; ** Elements originating from NobelReplace TiUnite®.

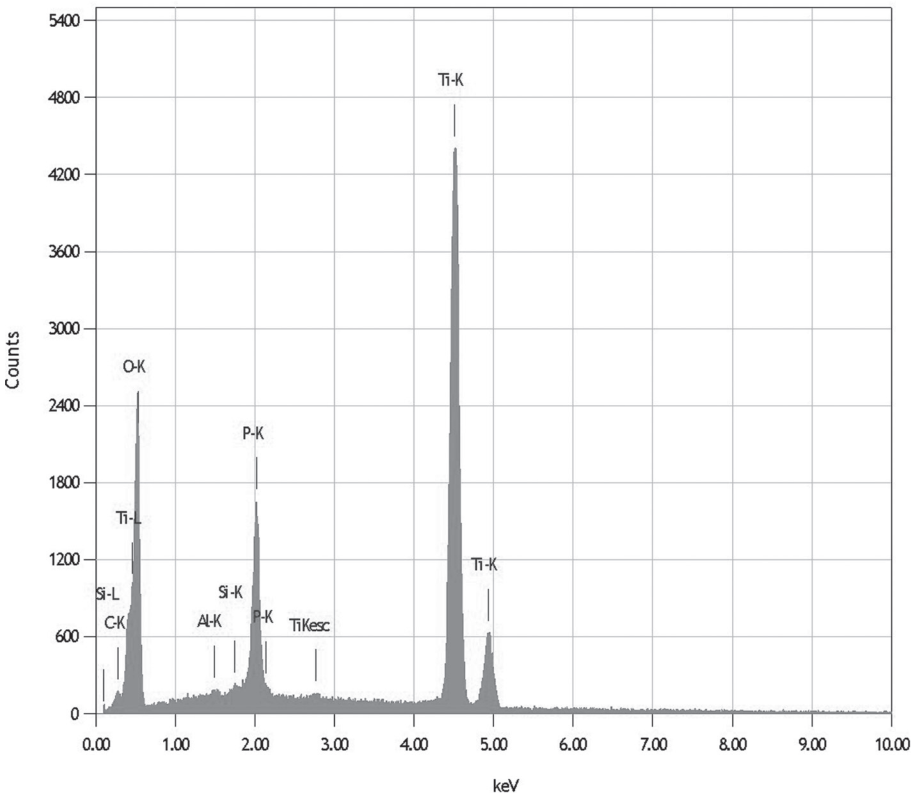

Figure 3 - EDS showing implant surfaces after 1 rinse.

Discussion

Achieving an accurate replication of the finishing margins for fixed prosthodontics requires adequate gingival retraction. Expasyl® is a retraction paste that provides this function. It is comprised of three components: kaolin, water and aluminum chloride. Retraction is provided via the kaolin, which is a clay-like, dense substance, checking problematic sulcus injuries and bleeding that could occur during placement and/or removal of a retraction cord. On natural abutment teeth there is little risk of damage to the epithelium attachment using Expasyl®.[5] Phatale *et al.* (2010) showed a higher percentage of intact junctional epithelium histologically with Expasyl® in comparison to the use of a retraction cord.[6] Junctional epithelium that surrounds implants is at greater risk of experiencing penetration damage than the more robust sulcus of natural teeth, when exposed to trauma such as gingival cord retraction procedures. Placement of a cordless retraction paste eliminates the risk of laceration of the sulcular groove.[4] The injectable matrix is hydrophilic and can be flushed away relatively easily from the gingival crevice.[7] Nevertheless, small traces of residue may persist and remain in the gingival crevice. In this study, all the samples that were exposed to Expasyl® retained some residual elemental components of the retraction paste.

Surface topography

Morphological images of the TiUnite® surface before exposure to Expasyl® showed a porous structure with numerous craters created during the surface modification phenomenon of anodic oxidation, produced by the manufacturer. Elements of Ti, O, C and a small amount of P were present on the TiUnite® implant surfaces. Previous studies have shown this to be a characteristic feature of these implants, insofar as P is incorporated into an anodic titanium oxide layer[8,9] to stimulate bone regeneration and increase the biochemical interlocking between bone matrix proteins and surface materials, thus improving osseointegration.[2]

EDS analysis of the paste, before usage, showed predominantly high elements of Al and Si, and was used to compare the paste composition to that of the implants after exposure to Expasyl® for elemental identification. The Si identified by EDS is in fact silicate coming from the kaolin, which is an aluminum silicate hydrate. The aluminum originates from the aluminum chloride and the kaolin.

The results showed minimal alterations of surface morphology and chemical composition on the implant surfaces after contact with Expasyl® in the three areas studied. Examination at ×500 magnification of the implant surfaces showed no differences on the SEM images before and after exposure to Expasyl®. However, BSE images displayed the alterations as "white particles" of a higher density on the surface after contact with Expasyl®. The density of these "particles" was highest at the collar of the implant. This may have occurred due to the easy accessibility of this area in contact with Expasyl® in comparison to the microthread, which is irregularly shaped and situated farther down the implant. SEM at ×5000 magnification showed residual remnants of Expasyl® and surface alterations of the implants. The paste appeared to accumulate on the implant surface, filling the crack propagations visible on the surface and forming continuous depositions along the ridges of the cracks. Upon drying for SEM preparation, the paste was compressed, causing air bubbles to burst in the cracks creating "voids" visible on SEM images. Implant surfaces with longer exposure to Expasyl™ showed more residual deposition and bubble formation. The surface alterations were still present after 4 weeks of storage.

Surface chemistry

Traces of Si and Al were found on implant surfaces after one rinse for Group 1 and Group 2. The remnants were all below 1%. Surprisingly, Group 1 presented more Al remnants than Group 2. After 2 rinses, only Al remnants were observed for both groups. Data obtained from EDS should be used as an indication rather than a definite value since it has a set minimum reading range of 1%.

The collar area consistently showed more Si and Al elements than the other exposed areas. Since it is the most coronal part of the implant, with initial and ongoing bone remodeling of the crestal peri-implant bone, this area is most likely to be in contact with Expasyl® during gingival retraction in clinical settings.

Phosphorous and titanium are components of the TiUnite® surface. Their concentrations were not affected by either the exposure time or by the number of rinses during the experimentation.

Changes in the surface chemical composition demonstrated that two rinses were more effective in removing the paste than one. Following the same exposure time, implant surfaces with one rinse had Al and Si elements remaining on the surfaces whereas Al was the only remaining element after 2 rinses.

For the elements present on the implants and in Expasyl®, the predominantly covalent bonds between Si and O are the strongest followed by the bonds between Al and O and then by the bonds between Ti and O. This is based on the difference in electronegativities of Si, Al and Ti relative to oxygen. In this case, Si would most likely bond to free oxygen present in the environment rather than to the small amount of oxygen available on the implant surface (grade IV titanium has only 0.4% oxygen). The lack of bonding with the implant surface might explain why Si is not present on the implant surface after two rinses. Al is very similar in size to Ti, and it is reasonable to consider that Al could replace some of the Ti in the structure via chemical substitution. This would explain why some Al persists on the implant surface even after two rinses. To validate this hypothesis, a transmission electron microscopy (TEM) analysis of the implant surface would be needed.

Surfaces with one rinse showed alterations in all three areas, and, with two rinses, changes to the C and JC areas were seen. Regardless of the number of rinses, C and JC showed consistent elemental alterations. The JC and MT areas are generally covered by surrounding tissues and, therefore, would not be in contact with Expasyl® during the retraction process. However, due to different implant systems and their connectors, in addition to variations in the degree of bone remodeling, these areas of the implant may become exposed, requiring retraction of the surrounding soft tissues to obtain an accurate impression.[10]

According to the manufacturer, Expasyl® has a pH value of 3 that may cause modifications on the implant surface. Whether this pH level may cause surface deterioration is questionable, since variables such as the length of application and subgingival environment cannot be reproduced. The relatively small sample size was a limitation to this study and further research with a larger sample size should be conducted to validate the present findings.

Implant surface contamination triggers a chain reaction leading to the dissolution of the TiO_2 layer, and it has been suggested that the presence of inorganic contaminants could reduce the clinical success of osseointegration.[11] Al ions are suspected to impair bone formation by a possible competitive action with calcium that could affect success in osseointegration.[12] However, Piattelli *et al*. showed results that did not support this hypothesis.[13] SEM images at ×5000 magnification did not show any sign of corrosion, and the surface alterations observed were not consistent with corrosion. The effect on the TiO_2 layer was not analyzed in this study.

Passivation of the implant surface following any contamination is believed to be essential for both corrosion resistance and the biocompatibility aspects of titanium implants.[14] TiO_2 can pacify tissue-destroying agents immediately after surgical trauma inherent to implantation.[3] In this study implants were left undisturbed in titanium sterile airtight containers for 4 weeks to evaluate if passivation of the surface could mask the remnants of Al and Si. No changes were observed upon re-examination.

Conclusion

Even though minimal changes to the implant surface morphology and composition were observed after Expasyl® contact, a definitive conclusion cannot be drawn due to the small sample size. Further research with test cultures using osteoblasts and fibroblasts should be conducted to assess the biocompatibility of Expasyl®-exposed implant surfaces.

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