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Contamination of the zirconia surface by components of mixed saliva and its control by X-ray photoelectron spectroscopy. Part 2. Influence of the functional monomer MDP-10

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Abstract

INTRODUCTION. The strength of adhesive luting of zirconia restorations largely depends on the surface state. Primers based on the functional monomer 10-methacryloyloxydecyl dihydrogen phosphate (MDP-10) provide chemical bonding of the adhesive to the oxide surface; however, during try-in the framework contacts mixed saliva, whose organic components may adsorb onto the surface. The effect of prior MDP-10 application on such contamination and its subsequent removability is insufficiently studied.

PURPOSE. To study the influence of the functional monomer MDP-10 on the elemental composition of the zirconia surface and on the resistance of the MDP-10-primed surface to salivary mucin contamination using X-ray photoelectron spectroscopy (XPS).

MATERIALS AND METHODS. Yttria-stabilized zirconia (Y-TZP) plates were examined in three states: after application of an MDP-10 solution in isopropanol (MDP-10); after subsequent application of a mucin model (MDP-10 + mucin); and after mucin application followed by distilled-water rinsing (MDP-10 + mucin + rinsing). Atomic concentrations of C, N, O, and P were determined by XPS (PREVAC EA15, Al K α , $h\nu = 1486.6$ eV) and the P/N ratio was calculated ($n = 6$ per group). Data were analysed by one-way ANOVA with Tukey post-hoc test.

RESULTS. The MDP-10-primed surface showed a pronounced phosphorus signal ($P = 7.24 \pm 0.12$ at.%) with low nitrogen content ($N = 1.10 \pm 0.05$ at.%; $P/N = 6.59 \pm 0.36$). Mucin application increased nitrogen to 2.42 ± 0.05 at.% and reduced P/N to 2.96 ± 0.08 ($p < 0.001$). After rinsing, nitrogen returned to the level of the MDP-10-primed surface (1.12 ± 0.05 at.%; no difference from the MDP-10 group, $p = 0.77$), and P/N largely recovered (5.84 ± 0.34). The phosphorus signal persisted in all groups.

CONCLUSION. MDP-10 forms a stable phosphate-containing layer on the zirconia surface. Salivary mucin adsorbs on top of the MDP-10 layer but, unlike on the untreated surface, is effectively removed by water rinsing, with nitrogen returning to baseline. MDP-10 priming increases the resistance of the surface to irreversible organic contamination, which is relevant to the protocol of preparing restorations for luting.

Keywords: zirconia, MDP-10, primer, X-ray photoelectron spectroscopy, saliva contamination, mucin, adhesion

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Контаминация поверхности диоксида циркония компонентами смешанной слюны и ее контроль методом рентгеновской фотоэлектронной спектроскопии. Часть 2. Влияние функционального мономера MDP-10

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Резюме

ВВЕДЕНИЕ. Прочность адгезивной фиксации реставраций из диоксида циркония в значительной степени определяется состоянием его поверхности. Праймеры на основе функционального мономера 10-метакрилоилоксиэцилдигидрофосфат (MDP-10) обеспечивают химическое сцепление адгезива с оксидной поверхностью, однако на этапах примерки каркас контактирует со смешанной слюной,

органические компоненты которой способны адсорбироваться на поверхности. Влияние предварительного нанесения MDP-10 на характер такой контаминации и возможность ее последующего удаления изучено недостаточно.

ЦЕЛЬ. Изучить влияние функционального мономера MDP-10 на элементный состав поверхности диоксида циркония и на устойчивость MDP-10-праймированной поверхности к контаминации муцином смешанной слюны методом рентгеновской фотоэлектронной спектроскопии (РФЭС).

МАТЕРИАЛЫ И МЕТОДЫ. Пластины стабилизированного оксидом иттрия диоксида циркония (Y-TZP) исследованы в трех состояниях: после нанесения раствора MDP-10 в изопропиловом спирте (MDP-10); после последующего нанесения модели муцина (MDP-10 + муцин); после нанесения муцина и смыва дистиллированной водой (MDP-10 + муцин + смыв). Методом РФЭС (PREVAC EA15, Al K α , $h\nu = 1486,6$ эВ) определяли атомные концентрации C, N, O, P и рассчитывали отношение P/N ($n = 6$ в каждой группе). Различия анализировали однофакторным дисперсионным анализом (ANOVA) с апостериорным критерием Тьюки.

РЕЗУЛЬТАТЫ. На MDP-10-праймированной поверхности регистрировался выраженный сигнал фосфора ($P = 7,24 \pm 0,12$ ат.%) при низком содержании азота ($N = 1,10 \pm 0,05$ ат.%; $P/N = 6,59 \pm 0,36$). Нанесение муцина повышало содержание азота до $2,42 \pm 0,05$ ат.% и снижало P/N до $2,96 \pm 0,08$ ($p < 0,001$). После смыва водой содержание азота возвращалось к уровню MDP-10-праймированной поверхности ($1,12 \pm 0,05$ ат.%; отличий от группы MDP-10 нет, $p = 0,77$), а отношение P/N в значительной степени восстанавливалось ($5,84 \pm 0,34$).

ЗАКЛЮЧЕНИЕ. Мономер MDP-10 формирует на поверхности диоксида циркония устойчивый фосфат-содержащий слой. Муцин смешанной слюны адсорбируется поверх MDP-10-слоя, однако, в отличие от немодифицированной поверхности, эффективно удаляется смывом водой. MDP-10-праймирование повышает устойчивость поверхности к необратимой органической контаминации.

Ключевые слова: диоксид циркония, MDP-10, праймер, рентгеновская фотоэлектронная спектроскопия, контаминация слюной, муцин, адгезия

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INTRODUCTION

Zirconium dioxide has firmly established itself in fixed prosthodontics owing to its combination of high mechanical strength, resistance to low-temperature degradation, biocompatibility, and satisfactory esthetics [1–5]. The clinical longevity of zirconia restorations supported by teeth and implants, including endodontically treated teeth, is confirmed by a number of studies [6–9]; however, its decisive condition remains the reliability of adhesive luting of the framework [10; 11].

The chemical inertness of zirconia and the absence of a glassy phase and silica preclude hydrofluoric acid etching and silanization. A key role in achieving a durable adhesive bond is played by the functional monomer MDP-10 (10-methacryloyloxydecyl dihydrogen phosphate): its phosphate group forms stable Zr–O–P bonds with the zirconia surface, providing the chemical component of adhesion [11; 12]. For this reason, modern primers and self-adhesive cements for zirconia are based on MDP-10 and related phosphate monomers.

A separate problem is the contamination of the framework surface by components of mixed saliva during try-in. We previously showed by XPS that on the untreated zirconia surface the organic nitrogen-containing components of saliva (mucin model) adsorb almost irreversibly and are not removed by water rinsing¹.

However, it remains open whether prior application of MDP-10 changes the character of contamination: whether the phosphate layer formed by the monomer can prevent irreversible mucin adsorption and enable its subsequent removal.

XPS, which probes a layer several nanometres thick, allows simultaneous monitoring of the phosphorus signal (P) as a marker of the MDP-10 layer and the nitrogen signal (N) as a marker of protein contamination, while their ratio (P/N) can be used as an integral characteristic of the surface state [12; 13].

PURPOSE

The aim of the study was to study the influence of the functional monomer MDP-10 on the elemental composition of the zirconia surface and on the resistance of the MDP-10-primed surface to salivary mucin contamination using X-ray photoelectron spectroscopy.

MATERIALS AND METHODS

Samples and Reagents

Plates of pre-sintered yttria-stabilized zirconia (Y-TZP; Shandong Huge Dental Material Corporation, China; ZrO₂ > 85%, Y₂O₃ < 6%, HfO₂ < 5%, Al₂O₃ < 1%) were used as the substrate. Priming was performed with a solution of the functional monomer MDP-10 in isopropanol: the solution was applied to the plate surface, after which the excess was removed and the surface was dried with a directed stream of compressed

¹ Meremkulov R.A., Khabadze Z.S. et al. A method for detecting saliva on the surface of zirconia dental devices using X-ray photoelectron spectroscopy. Patent RF (application). (In Russ.).

air. A snail secretion filtrate (SNAILINE, GALE. & COSM. S.r.l., Italy), rich in mucin-type glycoproteins, was used as a model of the mucin component of mixed saliva; the solution was applied as a thin layer and dried.

Experimental Design

Three surface states were studied:

- group 1 (MDP-10) – surface after application and drying of the MDP-10 solution;
- group 2 (MDP-10 + mucin) – MDP-10-primed surface after application and drying of the mucin model;
- group 3 (MDP-10 + mucin + rinsing) – the same surface after mucin application and subsequent rinsing with distilled water and drying.

Six samples were analysed in each group ($n = 6$).

X-ray Photoelectron Spectroscopy

XPS analysis was carried out on a PREVAC EA15 spectrometer with a high-resolution hemispherical analyser. Spectra were excited with non-monochromatized Al K α radiation ($h\nu = 1486.6$ eV, 150 W); the residual gas pressure did not exceed 5×10^{-9} mbar. The binding-energy scale was calibrated against the Au4f $_{7/2}$ (84.0 eV) and Ag3d $_{5/2}$ (368.3 eV) lines; the charging effect was corrected using the C1s line (284.8 eV). Spectra were deconvoluted in CasaXPS with Shirley background subtraction. Atomic concentrations of carbon (C1s), nitrogen (N1s), oxygen (O1s), and phosphorus (P2p) were determined; the P/N ratio was calculated from the P2p and N1s signals.

Statistical Analysis

Data are presented as $M \pm SD$. Normality of distributions was assessed using the Shapiro–Wilk test. Between-group differences were analysed by one-way

ANOVA with post-hoc pairwise comparison using the Tukey HSD test; differences were considered significant at $p < 0.05$. Calculations were performed in Python 3 (SciPy, statsmodels).

RESULTS

The summarized results of the quantitative XPS analysis are given in Table 1.

On the MDP-10-primed surface, a pronounced phosphorus signal was recorded (7.24 ± 0.12 at.%), reflecting the presence of the monomer phosphate groups, with a low nitrogen content (1.10 ± 0.05 at.%); the P/N ratio was 6.59 ± 0.36 . Application of the mucin model was accompanied by a more than twofold increase in nitrogen content – to 2.42 ± 0.05 at.% ($p < 0.001$) – and a decrease in the P/N ratio to 2.96 ± 0.08 ($p < 0.001$), reflecting the formation of a protein layer on top of the MDP-10 primer.

After rinsing with distilled water, the nitrogen content decreased to 1.12 ± 0.05 at.% and did not differ significantly from the initial MDP-10-primed surface (Tukey test, $p = 0.77$), whereas it differed significantly from the mucin group ($p < 0.001$). The P/N ratio after rinsing largely recovered (5.84 ± 0.34), approaching the level of the MDP-10-primed surface. Importantly, the phosphorus signal persisted in all groups (6.5–7.2 at.%), indicating stability of the MDP-10 layer to mucin application and water treatment. The dynamics of nitrogen content and the P/N ratio are shown in Figure 1 (ANOVA: N – $F = 1373$, $p < 0.001$; P/N – $F = 269$, $p < 0.001$).

DISCUSSION

The obtained data characterise the behaviour of the MDP-10-primed zirconia surface upon contact with organic components of mixed saliva. The stable phos-

Table 1. Surface elemental composition and P/N ratio in the study groups ($M \pm SD$; $n = 6$)

Group	C, at. %	N, at. %	O, at. %	P, at. %	P/N
MDP-10	66.80 ± 0.55	1.10 ± 0.05	24.87 ± 0.45	7.24 ± 0.12	6.59 ± 0.36
MDP-10 + mucin	61.88 ± 0.55	2.42 ± 0.05	28.53 ± 0.45	7.17 ± 0.12	2.96 ± 0.08
MDP-10 + mucin + rinsing	68.45 ± 0.55	1.12 ± 0.05	23.90 ± 0.45	6.53 ± 0.12	5.84 ± 0.34

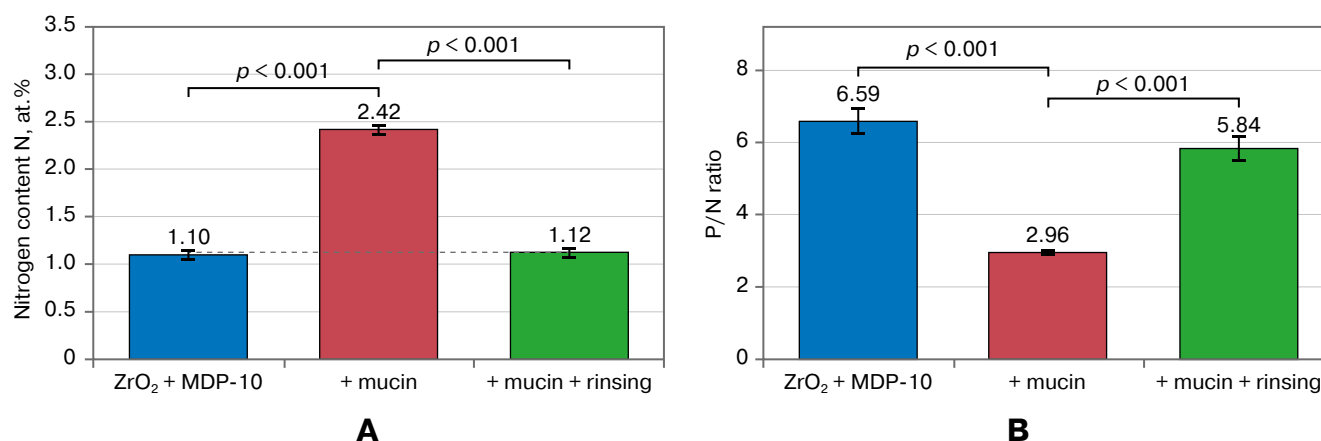


Fig. 1. Nitrogen content N (A) and P/N ratio (B) on the zirconia surface in the study groups ($M \pm SD$; $n = 6$); after rinsing, the nitrogen content returns to the level of the MDP-10-primed surface ($p = 0.77$) and the P/N ratio largely recovers

phorus signal (6.5–7.2 at.%), persisting in all groups, confirms the formation by MDP-10 of a chemically bonded phosphate layer that is stable to both mucin application and subsequent water treatment. This is consistent with the known mechanism of interaction of the MDP phosphate group with zirconia through the formation of Zr–O–P bonds [12].

Application of mucin naturally increased the nitrogen content and reduced the P/N ratio, reflecting adsorption of protein components on top of the MDP-10 layer. The behaviour of the surface after rinsing is of fundamental importance: the nitrogen content returned to the initial level of the MDP-10-primed surface ($p = 0.77$), and the P/N ratio largely recovered. This indicates that on the MDP-10-primed surface the mucin layer is retained predominantly reversibly and is effectively removed by water rinsing.

This result is in marked contrast to the behaviour of the untreated zirconia surface, for which almost irreversible mucin adsorption was previously established by XPS: water rinsing did not reduce the nitrogen content². Thus, prior application of MDP-10 not only provides the chemical component of adhesion but also increases the resistance of the surface to irreversible organic contamination, conferring on it the property of “rinsability” of the adsorbed protein film.

From a clinical standpoint, the data indicate the expediency of applying an MDP-containing primer, given that accidental saliva contamination of an MDP-10-primed surface is less irreversible and can be removed by wa-

ter treatment, whereas contamination of an unprimed surface requires more aggressive re-cleaning [11; 14]. Monitoring of residual contamination is appropriately performed instrumentally – by XPS via the nitrogen signal, which underlies the developed method for identifying saliva residues³.

Limitations of the study are associated with the use of a mucin model instead of native mixed saliva, with the surface-sensitive nature of XPS, and with the examination of a single type of zirconia material and a single monomer concentration. Assessment of the bond strength on the contaminated and cleaned MDP-10-primed surface is beyond the scope of this work and is the subject of further research.

CONCLUSION

1. The functional monomer MDP-10 forms a stable phosphate-containing layer on the zirconia surface: the phosphorus signal (6.5–7.2 at.%) persists upon mucin application and after water treatment.

2. Salivary mucin adsorbs on top of the MDP-10 layer (nitrogen content increases from 1.10 ± 0.05 to 2.42 ± 0.05 at.%; P/N decreases from 6.59 to 2.96; $p < 0.001$) but is removed by water rinsing: the nitrogen content returns to the level of the MDP-10-primed surface (1.12 ± 0.05 at.%; $p = 0.77$).

3. Unlike the untreated surface, where mucin adsorption is irreversible, MDP-10 priming increases the resistance of the zirconia surface to irreversible organic contamination, which is relevant to the protocol of preparing restorations for adhesive luting.

² Meremkulov R.A., Khabadze Z.S. et al. A method for detecting saliva on the surface of zirconia dental devices using X-ray photoelectron spectroscopy. Patent RF (application). (In Russ.).

³ Meremkulov R.A., Khabadze Z.S. et al. A method for detecting saliva on the surface of zirconia dental devices using X-ray photoelectron spectroscopy. Patent RF (application). (In Russ.).

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AUTHOR'S CONTRIBUTION

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