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Novel graphene oxide–silane nanocoatings on sound enamel: in vitro characterization and proof-of-principle assessment evaluation against erosive tooth wear

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Article

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Abstract

Objective

This proof-of-principle study evaluated whether a novel graphene oxide (GO)–silane nanocoating applied to sound enamel can provide acid resistance under a standardized citric-acid erosion model.

Materials and Methods

Ninety bovine enamel specimens were assigned into six groups: negative control, stannous-containing positive control, and four experimental coatings combining 3-methacryloxypropyltrimethoxysilane (MPS) with GO, with or without phosphoric acid pretreatment or stannous rinse. Surface characterization was performed by scanning electron microscopy (SEM). Following a 10-day erosive cycling protocol (six daily immersions in 1% citric acid, pH 2.3, surface loss was quantified on days 0, 4, 7, and 10 using optical profilometry (FRT). Data was analyzed using repeated-measures ANOVA.

Results

The GO-based coatings did not prevent enamel erosion. The positive control group consistently showed superior protective performance (surface loss: − 19.2 ± 3.6 μm; $p < 0.05$) over the negative control group (–29.3 ± 6.8 μm). GO-coated samples showed no protective effect, with mean enamel losses of − 32.9 ± 6.2 μm (only MPS), − 33.2 ± 3.7 μm (MPS + GO), and − 28.6 ± 6.7 μm (MPS + GO + stannous), showing no significant difference from the negative control ($p > 0.05$). Phosphoric acid pretreatment (phosphoric acid + MPS + GO) caused the highest substance loss (–39.7 ± 3.8 μm; $p > 0.05$).

Conclusions

GO–silane nanocoatings can be formed on enamel but do not confer resistance to citric-acid erosion. Phosphoric acid pretreatment compromises enamel integrity and should be avoided.

Clinical Relevance:

Current GO formulation and bonding approach do not provide erosion protection. Future development must optimize coupling agents and functionalized GO systems to achieve clinically meaningful acid resistance.

Introduction

ETW has become an increasingly prevalent global oral health issue with a significant clinical concern. It is a prevalent kind of tooth wear involving the progressive and irreversible loss of enamel and dentin associated with acid exposure without bacterial involvement. In the beginning acid exposure leads to the softening of enamel, which is still reversible, whereas repeated exposure to acids leads to irreversible loss of the dental hard tissue. Unlike dental caries, which results from bacterial colonisation of the oral cavity, dental erosion stems from extrinsic and intrinsic factors. Extrinsic factors are mostly of dietary origin, including the frequent consumption of highly acidic foods and beverages [1, 2]. Whereas the intrinsic factors, such as gastro-oesophageal reflux disease (GERD) or eating disorders (anorexia, bulimia), involve the repeated exposure of the teeth to gastric acid, leading to the loss of dental hard tissue [3]. Current preventative strategies largely depend on patient compliance, such as regular use of fluoridated mouth rinses or dietary modifications to reduce acid intake. Although the usage of best performing agents, like stannous fluorides, sound promising, repeated usage is necessary, as no agent currently on the market is able to form a long lasting protective barrier.[4]

Many studies have focused on preventative methods to manage dental erosion before it progresses beyond the enamel [5–8]. These studies have found that fluoride application plays an important role in protecting the enamel from tissue loss and preventing general erosive damage. Fluorides are an established form of therapy in dentistry with a caries-protective effect and are therefore the gold standard in caries prophylaxis [5]. The fluoride intake can be administered either systemically (drinking water, table salt, fluoride tablets) or locally (toothpastes, mouth rinses, etc.) [6].Various studies have then attempted to confirm that fluorides are able to prevent erosion as well as caries [9, 10]. According to the newest meta analyses fluoride can also be used as a preventative strategy against ETW by increasing enamel resistance to acid attacks and by aiding the enamel with the remineralization process. [2] However these meta analyses showed, that the protective effects of fluorides differ significantly between the different available formulations. Although all fluorides slow down the progression of ETW, particularly stannous fluoride containing products exhibited the most promising results, compared with conventional sodium fluoride products. [8] It has been found, that monovalent fluorides (NaF/AmF) have a lower protective effect against acids than polyvalent fluorides (SnF2, TiF4) [9]. Neither high nor low concentrations of monovalent fluorides were able to protect teeth from erosive loss of hard tooth structure. In contrast, polyvalent fluorides containing stannous chloride or titanium fluoride were able to provide evident protection against erosive loss of hard tooth structure. In a comparison of different metal fluoride-containing mouth rinses, it was shown that the tin-containing mouth rinse (AmF/NaF/SnCl2) significantly reduces the loss of tooth structure due to erosion.[10] The tin chloride combined with fluoride offers a different kind of protection than the other fluoride solutions, like sodium or amine fluoride. It builds a tin layer on the surface if the enamel, protecting it from acid attacks and demineralization. Additionally, it enhances fluoride intake into the tooth, binding to its minerals and therefore increasing the resistance towards acid attacks.[11] However no permanent durable coating has been created with polyvalent fluorides, which makes the protection dependent on the compliance of the patient by frequently applying it to the enamel. Additionally, it has been reported, that there is no significant difference in the outcome of brushing teeth immediately after an ETW attack or delaying it.

[12] This shows that there are no clinical materials available on the market, that form a durable, long lasting protective layer against ETW.

Despite these advances, a clinical demand for long-lasting, user-friendly methods that require minimal application and maintenance, remains. In recent years, graphene based nanomaterials have been focused on in biomedical and dental research due to their unique physiochemical properties. In particular, graphene oxide (GO), a derivate of graphene, is an emerging nanomaterial known for its exceptional biocompatibility, barrier properties and mechanical strength. Its functional groups (OH, COOH, C = O) enable chemical bonding and have the potential for barrier formation, which may be instrumental for creating a protective surface coating. The material GO is already widely used in the dental field. In combination with Amoxicillin, it can enhance the antibacterial effects, particularly against the pathogen Enterococcus faecalis, which is typically present in periapical and periodontal inflammation [13]. In implantology, GO coatings have shown to improve osseointegration and inhibiting the adhesion of bacteria on implant surfaces [14]. Furthermore, GO has improved material properties and antibacterial effects in dental restorative materials such as cements, adhesives and silane primers [15, 16]. In orthodontic treatments, GO can also reduce the demineralization of enamel and dentin by the use of bioactive nanocomposites [15]. In the biomedical filed, GO proved to enhance to properties of epoxy powder coatings to create a protection against corrosion on a steel substrate.[17] Although GO has been used in many different areas for its promising properties, it has not been used as a material to prevent erosions on enamel yet.

Although the promising properties of GO have been established through many studies in different fields, it remains unclear, whether it can create a stable and acid resistant coating on the biologically complex enamel tissue. Even though GO coatings have been previously explored on dentin surfaces in two studies, there is no study investigating the interaction between GO and enamel. Furthermore there are no studies evaluating the potential of GO based coatings on enamel against repeated daily erosive acid attacks under clinically relevant conditions, which are necessary to assess GOs suitability for a future clinical application for erosion prevention. Therefore, the aim of the present study was to evaluate whether a novel GO-silane nanocoating applied to enamel can provide acid resistance under a standardized citric acid erosion model, and to characterize the coating morphology and interface behavior using profilometry and scanning electron microscopy.

Materials and Methods

Sample Preparation

Ninety bovine incisors were extracted in slaughterhouses from the lower jaws of cows that have reached the grown age of 18 months. These extracted teeth were stored in a 0,1% thymol solution (Sigma-Aldrich, Taufkirchen, Germany) to reduce bacterial growth. In the laboratory, the teeth were cleaned of any residual tissue. With a diamond coated saw (EXAKT 300 CL, EXAKT Advanced Technologies, Norderstedt, Deutschland), the tooth crown was separated from the root with constant water cooling. If

the crown was too big, it was halved with the diamond coated saw once more. Afterwards the tooth was glued to a microscope slide with Technovit 7230 VLC (KULZER, Wehrheim, Deutschland) and polished using P800, P 1200, P 2400 and P 4000 silicon carbide grinding paper (LECO Instrumente, Mönchengladbach, Deutschland) following the protocol of Esteves et al. [18]. to simulate an eroded tooth surface. With the assistance of a stamp, all samples could be cut into small sample blocks measuring 4 x 5 x 3 mm and stored in 0,1% thymol solution. Three to four samples were then glued to a microscope slide with Technovit 7230 VLC and 1 mm of each sample were covered with the Technovit 7230 VLC on each side to cover and simultaneously mark the reference area. This procedure has been tested before, with no loss of surface area.

Groups

Ninety samples were randomly distributed to six groups (n = 15), each receiving different surface treatments on the experimental area of the samples (Table 1). Group 1 had no pre-treatment and served as the negative control group. Group 2, the positive control group, was treated with a stannous chloride containing mouth rinse (Elmex Optischmelz, 30 seconds). Group 3 was treated with a 3 second application of phosphoric acid and was afterwards rinsed with water, followed by a 20 second application of methacryloxypropyltrimethoxysilane (MPS) and 1 minute dip-coating with GO suspension. Group 4 was only treated with MPS for 20 seconds. Group 5 was treated with MPS (20 s), followed by GO dip-coating (1 min). Group 6 combined MPS (20 s), GO dip-coating (1 min) and the stannous chloride containing mouth rinse (30 s). Between each application, the samples were air-dried.

Table 1
Group overview

Group name	Coating	Exposure time	Number
Negative control group	no coating	none	15
Positive control group	Tin containing mouth wash(Elmex Optischmelz; 500 ppm F-, 800 ppm Sn^{2+})	30 seconds on the GFL 3006 shaking apparatus (Elmex Optischmelz)	15
Phosphoric acid + MPS + GO	37% orthophosphoric acid, Monobond Plus, 3% GO suspension	20 seconds with microbrush and 1 minute dip-coating (GO)	15
MPS	Monobond Plus	20 seconds with microbrush	15
MPS + GO	Monobond Plus, 3% GO suspension	20 seconds with microbrush (MPS) and 1 minute *dip-coating* (GO)	15
MPS + GO + Elmex	Monobond Plus, 3% GO suspension + tin containing mouth wash (500 ppm F-, 800 ppm Sn^{2+})	20 seconds with microbrush and 1 minute *dip-coating* (GO) 30 seconds on the GFL 3006 shaking apparatus (Elmex Optischmelz)	15

Graphene Oxide

A 3% GO suspension via the modified Hummers and Offeman method [19], has been applied. The ratio is 30g GO to 1 liter of water. GO has the property of corrosion resistance due to its functional groups. These functional groups include hydroxyl (OH), carboxyl (COOH), and carbonyl (C = O) groups. The OH groups can form compounds with the hydroxyapatite of the enamel surface, creating a chemically resistant GO coating that protects the tooth from erosion. However, in one of the pre-trial experiments, it was found that the GO is not bonded on the enamel surface. This supports the assumption that a bonding agent is necessary to create a bond between the GO and the enamel.

Silane

Silanes can be used to create a bond between the hydroxyapatite in tooth enamel and the GO suspension. They are well-established products in dentistry (e.g. Monobond Plus, Clearfil SE, RelyX, etc.) and exhibit high biocompatibility [20]. Following prior testing of silanes, 3-methacryloxypropyltrimethoxysilane (MPS, Monobond Plus) has been selected as the bonding agent for the main test. This silane is a single-component bonding agent that has previously achieved good adhesion to artificial surfaces [21] and is used to condition a wide variety of restoration materials (including zirconia crowns and glass ceramics). It is anticipated that the adhesion of the GO to the tooth enamel is increased with silanes. In addition, MPS has already bonded successfully to GO in previous studies [20]. The bifunctionality of silanes enables to bind to surfaces via functional groups of silanols formed through hydrolysis. Silanes have both organic and inorganic groups. The inorganic groups form a bond with the hydroxyapatite of the tooth (Si-O-P or Si-O-Ca bond) and through a condensation reaction, a bond with the GO (Si-O-C bond), while the organic methacrylate group remains free and can react with other substances [20].

Erosive Cycling

The experiments were conducted using an established erosive cycle [22] that simulates the natural conditions and pH fluctuations in the oral cavity after consuming acidic foods and the regeneration of the pH in the oral cavity. For this purpose, a 1% 0.05 M citric acid solution (Carl Roth GmbH + Co. KG, Karlsruhe, Germany, **pH 2.3**) and a remineralization solution (**pH 6.7**) based on the formulation by Gerrard and Winter [23] were used. The erosive cycle lasted for 10 days. Erosive demineralization was performed six times a day for two minutes each time. After each demineralization, the samples were placed in the remineralization solution for one hour. Before switching between demineralization and remineralization solutions, the samples were rinsed for one minute with tap water. For each cycle (demineralization & remineralization), all samples were placed in a GFL-3006 shaking water bath (Gesellschaft für Labortechnik mbH, Burgwedel, Germany) with gentle horizontal movements (35 rpm) at 37°C, simulating the temperature of the oral cavity. At the end of the last cycle of the day, the samples were left in the remineralization solution overnight.

The volume calculation for the demineralization and remineralization solutions was performed according to the protocol of Esteves et al. [24], which required 2 ml of solution/mm^2 of enamel.

The 15 samples were randomly divided into groups and coated once with the solutions described above (see Groups), before the start of the erosion cycle. Only group 2, with the tin-containing mouthwash (Elmex Optischmelz), was treated twice daily, before the start of the erosion cycle and after completion of the last run in the demineralization solution, according to the protocol by Schlueter et al. 2009.

Profilometric Measurement

The surface loss during the erosive cycle was measured using an FRT profilometer (MicroProf, Fries Research & Technology GmbH, Bergisch Gladbach, Germany) on days 0, 4, 7, and 10. To do this, the Technovit was carefully removed from the left and right sides of the reference surface using a scalpel (size 15). Measurements were then taken at three points on the sample: the top, centre, and bottom. The distance between each of the three lines was 0.5 mm, and each line was 4 mm long.

The measurement lines were evaluated using software specialized for FRT (Mark III, Fries Research & Technology GmbH), which created a 2D profile. To determine the step height, three straight lines parallel to the x-axis were drawn in the profile. The vertical distance between the straight lines at the boundary between the reference and test surfaces corresponds to the height difference caused by removal or addition. The transition area is omitted from the evaluation so that only the horizontal straight lines are considered. The mean value is calculated from the three measurement lines per sample.

Further measurements of surface loss were carried out using an optical microscope (Modell Keyence VK-9710, Keyence GmbH, Neu-Isenburg, Germany), which is a high-resolution, confocal 3D laser scanning microscope for surface analysis.

For the main experiment, surface loss was determined exclusively with the FRT, as preliminary tests with FRT and Keyence showed differing results. The FRT is considered as the established method in studies. The Keyence was used to supplement the creation of 3D surface profiles at the baseline (day 0) and after the erosive cycle (day 10). One sample per group was analyzed at 10x magnification.

Since only a limited sample area can be captured with both systems (FRT/Keyence), the measurement position was selected in the middle of the transitions between the reference and test surfaces on both sides.

SEM

To determine whether the samples were completely coated, the Gemini SEM 560 scanning electron microscope (SEM) with ZEISS Gemini 3 electron optics (ZEISS, Oberkochen, Germany) was used. The SEM ensures precise examination of the sample surfaces with high resolution in the sub-nanometer range. To ensure that the results are as comparable as possible, magnifications of 27x, 5000x, and

25000x were selected. One sample before and one sample after the erosion cycle were selected and analyzed for each group. The samples are then placed in the EM ACE600 high-vacuum sputter coater from Leica (Leica Microsystems GmbH, Wetzlar, Germany) for platinum coating of the surface. Coating the samples prior to SEM analysis allows very high resolutions.

Statistical Analysis

The statistical analysis of the collected data was performed using SPSS Statistics software version 29.0.2.0 (IBM Corp., Armonk, New York). The degree of erosive tooth structure loss on the examined surfaces was described using the mean and standard deviation. The normal distribution of the examined data was tested using the Shapiro-Wilk test. Samples with gross outliers in the values were removed from the groups beforehand. An ANOVA with repeated measurements was then performed. Due to the violation of the sphericity assumption, which was confirmed by the Mauchly sphericity test and a Greenhouse-Geisser epsilon coefficient < 0.75, the Greenhouse-Geisser correction was applied. Post-hoc multiple comparisons between groups were performed using the Bonferroni correction. A p-value < 0.05 was considered statistically significant.

Results

Profilometric Measurements

All results of erosive enamel surface loss are summarized in Fig. 1.

After the 10-day erosion cycle, the various coatings were examined for enamel surface loss due to citric acid, as well as for their resistance. Enamel surface loss increased with elapsed exposure time (days 1–10), indicating the expected progression of erosive loss over time (Fig. 1).

Statistical evaluation of the treated samples after the 10-day erosion cycle showed that, as expected, the positive control group (Mean ±SD: -19.2 ± 3.6 µm) had a preventive effect and thus achieved significantly better results than all the other groups ($p < 0.001$; ANOVA). The negative control group (-29.3 ± 6.8 µm) showed no significant difference compared to the other treated groups: MPS (-32.9 ± 6.2 µm), MPS + GO (-33.21 ± 3.71 µm), and MPS + GO + Elmex (-28.6 ± 6.7 µm) ($p > 0.05$; ANOVA). Phosphoric acid + MPS + GO (-39.7 ± 3.8 µm) exhibited significantly poorer protection against erosive tooth structure loss than the negative control group ($p < 0.001$; ANOVA).

A similar pattern emerges for the individual days (0, 4, 7 and 10) (Fig. 1). Statistically significant differences ($p < 0.05$; ANOVA) were observed on various days. The positive control group performed significantly better than the other groups on all days except day 0, when there was no significantly better coating than in the MPS ($p = 1.000$) or MPS + GO ($p = 0.06$; ANOVA) groups. On day 0, baseline evaluation after treatment application and before the erosive cycle, however, there was a significant difference between the MPS group and the negative control group ($p = 0.002$). There was no significant difference between the MPS and GO containing groups ($p = 1.000$).

SEM Analysis

Prior to the erosive loss of tooth structure (pre-erosion) (Fig. 2), groups 5 and 6 exhibited a complete surface coating characterised by distinct GO-specific wrinkling, whereas the other groups displayed a uniform, unstructured surface. Following the 10-day erosion cycle (post-erosion) (Fig. 3), the surfaces of all groups were similar and the differences between the coatings had largely disappeared, resulting in comparable morphological features across all samples.

Discussion

In this in vitro study, graphene oxide (GO) was investigated as an erosion-protective surface coating for tooth enamel using an established 10-day erosion cycle. The aim was to test whether GO, after suitable preconditioning of the tooth surface, can achieve an improved protective effect compared to a reference solution containing stannous chloride [25].

The results of the erosion cycle show that the positive control group exhibited a significantly higher protective effect against erosive tooth structure loss on all measurement days than all other groups ($p < 0.05$; ANOVA). Only on day 0 was there no significant difference between the MPS and MPS + GO groups ($p > 0.05$), suggesting that the protective effect of the established coating was not yet fully developed initially. Although substance gain was observed in several groups at the beginning, this was relativized by day 4, so that erosive substance loss became clear as the test progressed.

All GO-coated groups (MPS + GO, MPS + GO + Elmex, phosphoric acid + MPS + GO) showed significantly less protective effect over time than the positive control group, with the phosphoric acid + MPS + GO group performing significantly worse ($p < 0.001$). The theoretically improved adhesion due to phosphoric acid could not be confirmed experimentally, as the pretreatment was associated with a loss of substance and no protective effect was achieved. This group performed even worse than the negative control group, so clinical application is not recommended.

Preliminary tests showed that GO does not adhere to the enamel surface without structural preconditioning. Although silanes such as MPS or OTS enabled almost complete covalent GO coating, this was sometimes accompanied by loss of tooth surface substance. APTS proved impractical due to long exposure times. This clearly shows that the adhesion and effectiveness of GO depend significantly on surface pretreatment, but at the same time there are limitations in terms of substance preservation. This observation is consistent with earlier studies in which GO was not used as a sole coating but to improve the adhesion and mechanical stability of adhesives or composites [26, 27].

The working hypothesis that GO-coated samples exhibit a comparable or better protective effect than a mouthwash containing stannous chloride after erosive cycles [25] could not be confirmed. The GO groups did not differ significantly from the negative control group and did not show any reliable erosion-protective effect. This is consistent with the limited studies available to date, which have focused

primarily on bovine root dentin (Nahorny et al. 2017; Soares et al. 2020), whose composition and microhardness differ significantly from human enamel.[28]

Limitations of the present study include the in vitro model used, the limited number of samples, and the short duration of the erosion cycle, which restricts direct transferability to clinical conditions. In addition, GO was only tested in combination with selected silanes, confining the results to these preconditioning treatments. Another major limitation is the lack of physicochemical characterization of the coating. Consequently, it remains unclear whether the observed lack of efficacy was related to insufficient adhesion, inadequate coating thickness, rapid degradation of the film, or insufficient resistance to acid penetration.

In summary, although a covalently bonded, structurally intact GO coating could be achieved after silane-based preconditioning, especially with MPS, it did not show sufficient acid resistance under erosive conditions. Under the conditions tested, GO coatings do not currently offer reliable protection against erosive tooth structure loss and are therefore not yet suitable for clinical use.

Future studies should further optimize the surface coatings, e.g., by using alternative silanes (e.g., GPTMS, TESPD) or functionalized GO surfaces [29–31]. In addition, bioactive components such as fluorides, calcium phosphate systems, or polymers could be used to improve adhesion, long-term stability, and remineralization [32, 33]. GO can be considered a clinically relevant material for the prevention of erosive tooth structure loss, after establishing a biocompatible, acid-resistant, and long-term stable GO-based coating.

Conclusion

In the present study, a newly developed GO-based coating was applied to eroded enamel samples to test its effectiveness against ETW. However, the coating did not yield the expected results under the applied experimental conditions. Although a structurally intact coating was achieved after preconditioning the surface with a silane, the GO-silane coated samples showed no significant reduction in substance loss compared to the untreated negative control group. In contrast, the tin chloride containing positive control group demonstrated consistently better protection of the samples against ETW.

In comparison to the good physiochemical properties of GO in synthetic substrates, the results show, that these properties can’t be fully transferred to the biologically complex dental hard tissue. Surface preconditioning is important to make the GO stick to the surface, although some kinds of preconditioning, like phosphoric acid, played a critical role in decreasing the protective performance of the coating.

This study highlights the limitations of the GO-based coatings, underlining the importance of optimising surface modifications to achieve a protective coating against early ETW. Further research should focus on alternative coupling agents, functionalized GO systems, and bioactive additives to improve adhesion, acid resistance, and long-term stability before clinical applicability can be considered. The tested

formulation did not provide measurable protection against erosive challenges. However, the underlying mechanisms remain unclear because coating adhesion, layer thickness stability, and acid permeability were not assessed. Future studies should include quantitative characterization of the film-forming properties and barrier function to identify the reasons for the limited efficacy.

Declarations

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Data Availability: The datasets generated during and/or analyzed during the current study are not publicly available, but are available from the corresponding author upon reasonable request.

Author contribution: D.A. and M.E.-O. contributed to the study conception and design. Experimental work, investigation, and data curation were performed by D.A., with experimental support and methodology contributed by K.S.. Formal analysis, software implementation, and validation were conducted by K.D.. D.A. prepared all figures and tables. M.E.-O. provided resources, funding, project administration, and supervision alongside K.S.. The first draft of the manuscript was written by D.A., and critical revision and editing were performed by K.S. and M.E.-O.. All authors read and approved the final manuscript.

Competing Interests: The authors have no relevant financial or non-financial interests to disclose.

Ethics Approval: This study utilized discarded biological waste (bovine teeth) obtained under Reference No. JLU_kTV_1_2024. Formal ethical approval for live animal use was not required, as no live animals were used or harmed in this research.

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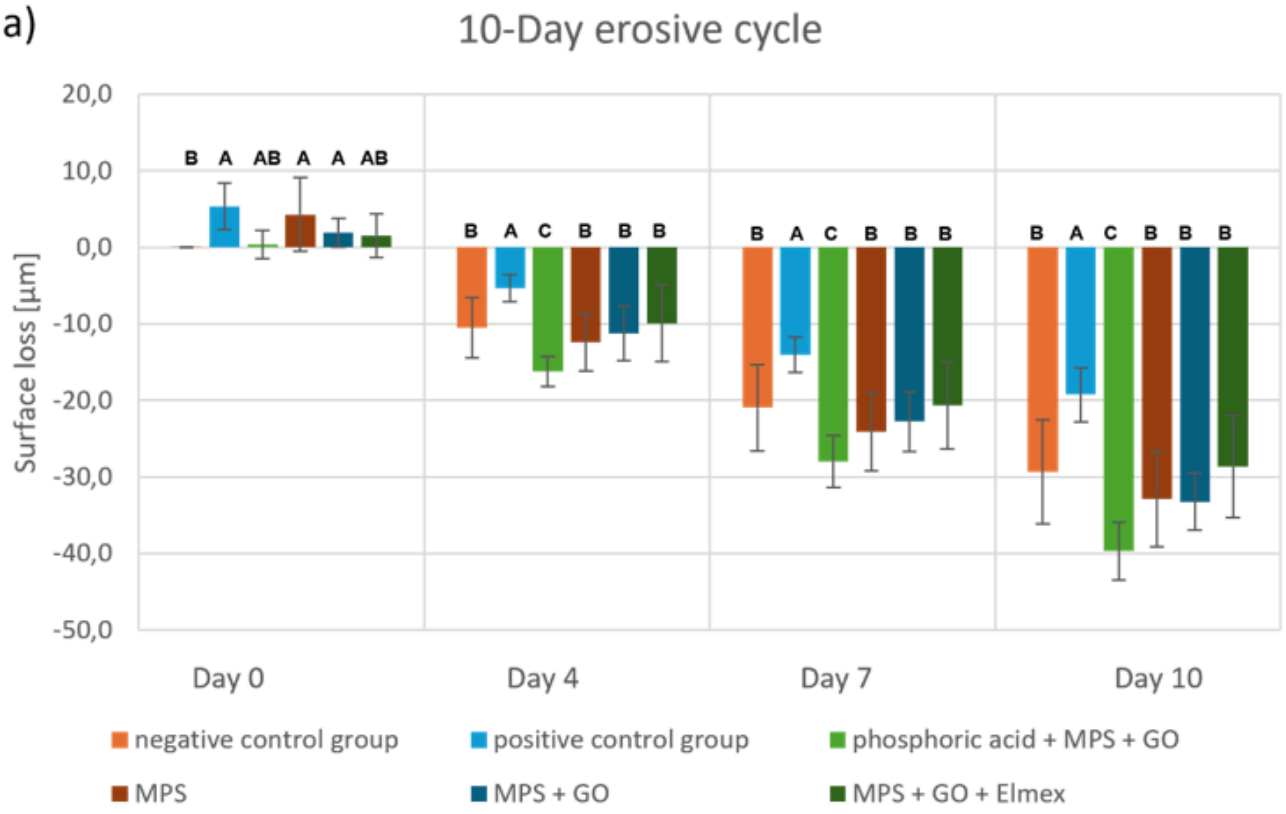
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Figures

a)



b)

	Day 0		Day 4		Day 7		Day 10	
	M	SD	M	SD	M	SD	M	SD
Negative control group	0,0	0,0	-10,5	3,9	-20,9	5,6	-29,3	6,8
Positive control group	5,4	3,0	-5,3	1,8	-14,0	2,3	-19,2	3,6
Phosphoric acid + MPS + GO	0,4	1,8	-16,2	2,0	-28,0	3,4	-39,7	3,8
MPS	4,3	4,8	-12,4	3,8	-24,1	5,2	-32,9	6,2
MPS + GO	1,9	1,9	-11,2	3,6	-22,8	3,9	-33,2	3,7
MPS + GO + Elmex	1,6	2,9	-9,9	5,0	-20,6	5,7	-28,6	6,7

Figure 1

Enamel surface loss over the 10-day experimental period; a) Mean enamel surface loss [µm] at days 0, 4, 7 and 10. Identical letters (A, B, C) above the bars indicate no significant difference between them ($p \geq 0.05$); different letters indicate significant differences ($p < 0.05$) between the groups on the respective day (ANOVA with post-hoc test) b) corresponding mean value and standard deviations for each experimental group

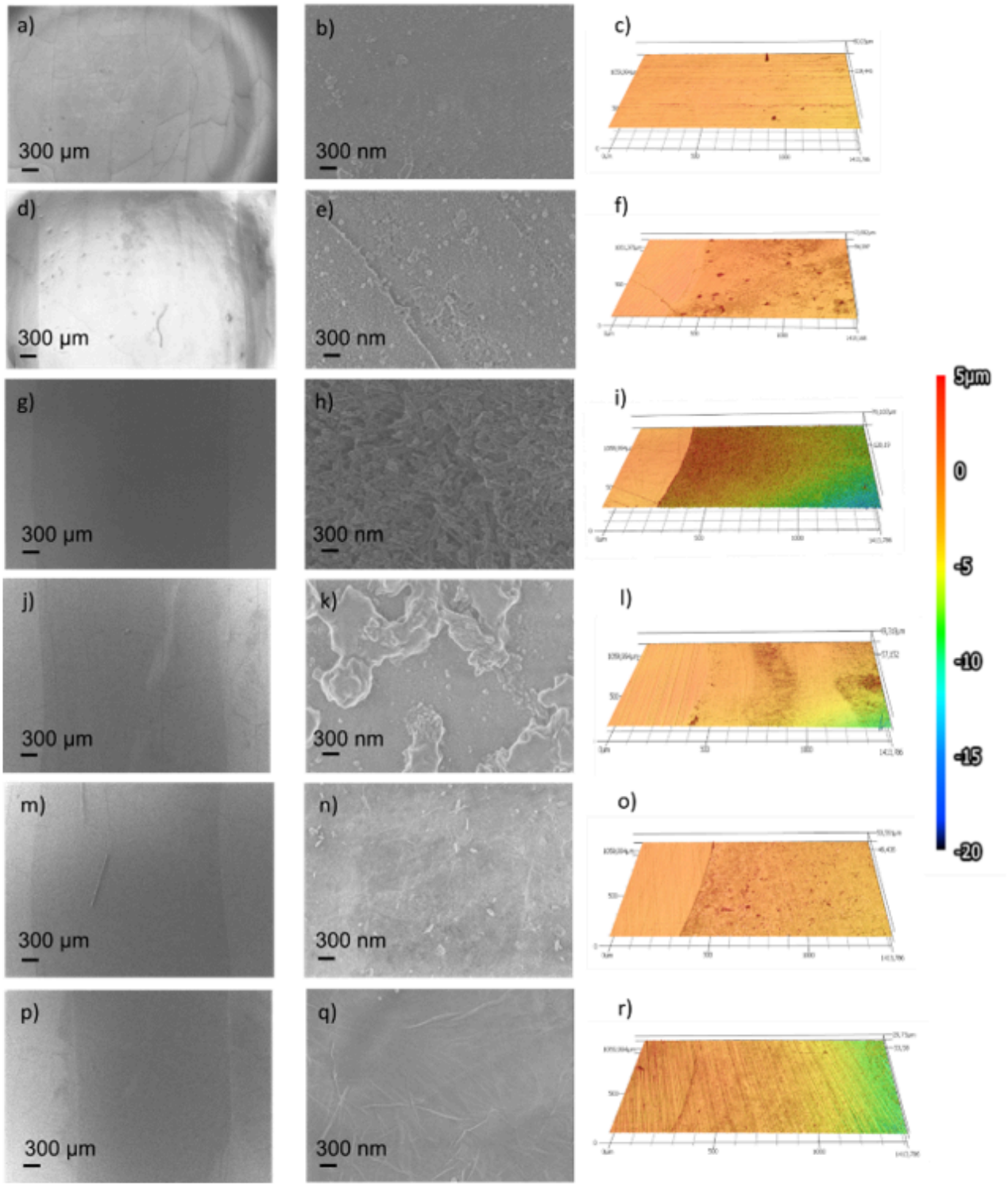


Figure 2

Pre-Erosion Columns show SEM overview (a, d, g, j, m, p), SEM detail (b, e, h, k, n, q), and Keyence profiles (c, f, i, l, o, r). Rows represent untreated negative control group (a-c), positive control group (d-f), phosphoric acid + MPS + GO (g-i), MPS (j-l), MPS + GO (m-o), MPS + GO + Elmex (p-r)

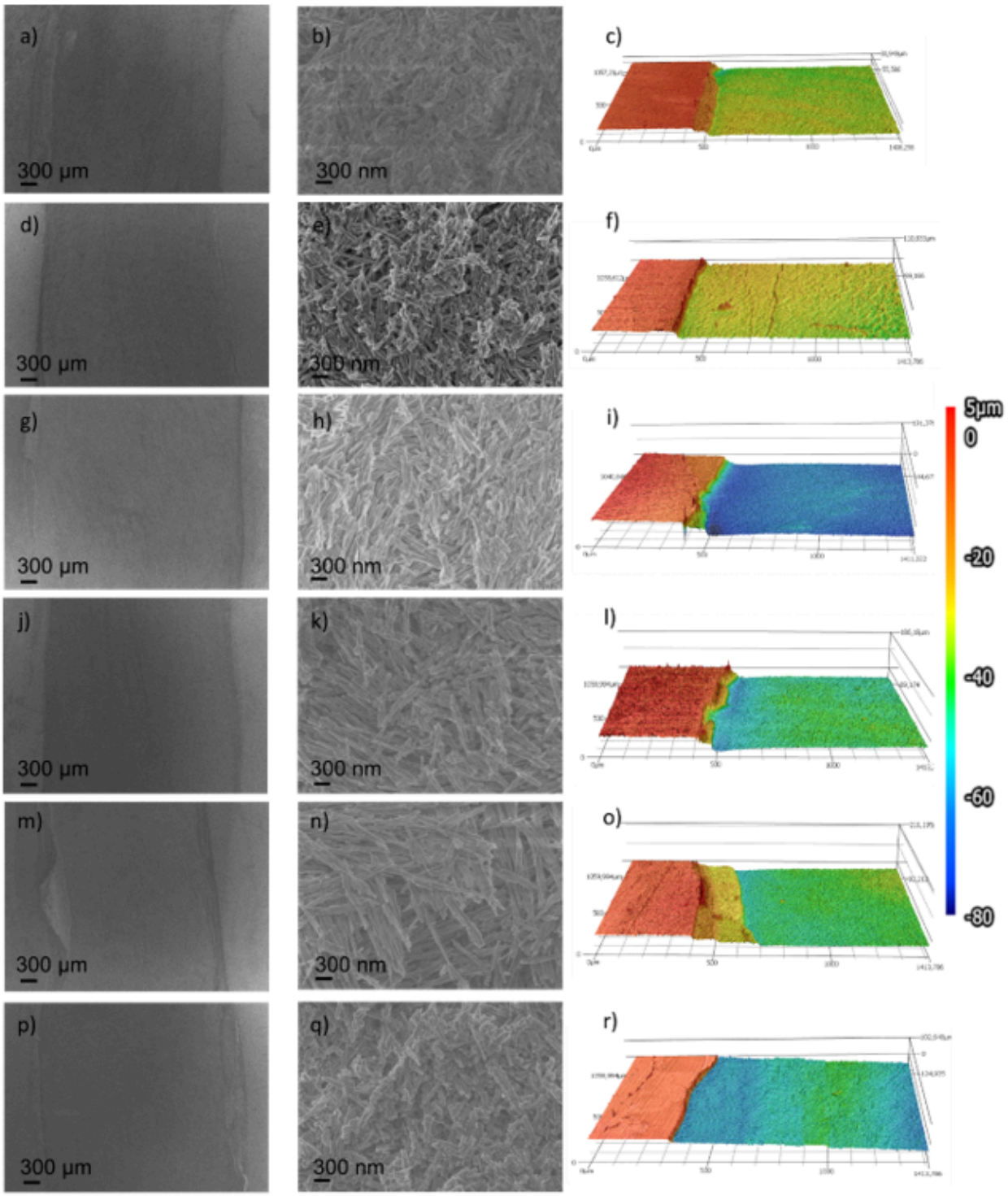


Figure 3

Post-Erosion Columns show SEM overview (a, d, g, j, m, p), SEM detail (b, e, h, k, n, q), and Keyence profiles (c, f, i, l, o, r). Rows represent untreated negative control group (a-c), positive control group (d-f), phosphoric acid + MPS + GO (g-i), MPS (j-l), MPS + GO (m-o), MPS + GO + Elmex (p-r)