RESEARCH PAPER

Development and Physicochemical Characterisation of a Chlorhexidine Hexametaphosphate Coating for Stainless-Steel Orthodontic Mini-Implants

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ABSTRACT

Background: Chlorhexidine–hexametaphosphate (CHX–HMP) nanoparticles have potential applications in dental surface coatings owing to their reported antimicrobial properties and sustained chlorhexidine release.

Aim: To synthesise CHX–HMP nanoparticles, apply the formulation to stainless-steel orthodontic mini-implants, and characterise the resulting surface modifications.

Materials and Methods: CHX–HMP nanoparticles were synthesised using an aqueous precipitation method by combining chlorhexidine digluconate and sodium hexametaphosphate solutions under continuous stirring. Coated and uncoated specimens were evaluated using scanning electron microscopy (SEM), energy-dispersive X-ray spectroscopy (EDX), Fourier-transform infrared spectroscopy (FTIR), and three-dimensional optical profilometry.

Results: Optical profilometry demonstrated increases in areal surface roughness (Sa) from 2.7693 to 3.0730 µm (10.97%) and profile roughness (Ra) from 0.2222 to 0.3822 µm (72.01%) following coating. SEM revealed an irregular surface morphology with variations in texture. EDX detected chlorine and nitrogen, consistent with the presence of chlorhexidine-containing material; however, phosphorus was not clearly identified in the available quantitative results. FTIR demonstrated differences in the spectral profiles of coated and uncoated specimens, providing preliminary evidence of surface modification.

Conclusion: CHX–HMP coating was associated with changes in the surface characteristics of stainless-steel orthodontic mini-implants. Further investigation is required to confirm coating composition and evaluate antimicrobial efficacy, sustained-release behaviour, coating adhesion, and biological compatibility.

Keywords: Chlorhexidine hexametaphosphate; orthodontic mini-implants; surface coating; surface roughness; surface characterisation.

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INTRODUCTION

Chlorhexidine (CHX) is a broad-spectrum antimicrobial agent widely used in dentistry, with antimicrobial action that primarily disrupts the bacterial cell membrane, increasing permeability and leakage of intracellular constituents(1–5). However, maintaining its activity over time can be challenging when conventional formulations are applied to surfaces exposed to saliva and other oral fluids(6). This has led to the investigation of formulations that improve chlorhexidine retention while permitting its gradual release.

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Plaque buildup is often seen around orthodontic mini-implants because the portion in the oral cavity is always in contact with saliva and food(7). Bacteria collect around the implant head, and over time this can cause swelling and irritation in the surrounding tissue. Studies of mini-implants removed after clinical use have found heavy plaque deposits around the exposed portion(8–11). Stainless steel is used because it is strong and can withstand orthodontic forces. However, its surface may promote initial bacterial adhesion, thereby facilitating subsequent biofilm formation(12). Chlorhexidine–hexametaphosphate (CHX–HMP) nanoparticles are relevant here because previous studies have investigated their deposition on material surfaces and subsequent release of chlorhexidine(13–16). CHX–HMP nanoparticles, formed by combining chlorhexidine digluconate with sodium hexametaphosphate, represent one such approach(14).

Research following this initial investigation has examined the potential use of CHX–HMP nanoparticles on dental materials. Wood et al. reported successful deposition of the nanoparticles onto titanium surfaces, together with sustained chlorhexidine release and antimicrobial activity against Streptococcus gordonii(17). Their application to orthodontic materials has also been explored, including stainless-steel and nickel–titanium archwires, on which nanoparticle deposition and continued chlorhexidine release in artificial saliva have been reported. Collectively, these findings indicate the potential of CHX–HMP nanoparticles as surface-modifying agents for dental applications(16–18).

However, their performance on a substrate may depend partly on the characteristics of the formulation itself. A detailed assessment of its chemical composition, morphology, and surface properties is therefore necessary before its incorporation into a coating system or evaluation on orthodontic mini-implants(13).

Furthermore, findings from titanium surfaces or orthodontic archwires cannot be automatically extrapolated to stainless-steel mini-implants. The characteristics of the nanoparticle formulation and its behaviour when applied to stainless steel therefore warrant investigation before its suitability for this application can be established.

The present study aimed to synthesise chlorhexidine–hexametaphosphate (CHX–HMP) nanoparticles using an aqueous precipitation method, coat stainless-steel orthodontic mini-implants with the synthesised formulation, and characterise the coating using Fourier-transform infrared spectroscopy (FTIR), scanning electron microscopy (SEM), energy-dispersive X-ray spectroscopy (EDX), and three-dimensional surface topography analysis.

MATERIALS AND METHODS

Synthesis of Chlorhexidine Hexametaphosphate (CHX–HMP) Nanoparticles

Chlorhexidine hexametaphosphate (CHX–HMP) nanoparticles were synthesised using an aqueous precipitation method adapted from the protocol described in the mentioned studies(14,17). The method is based on the interaction between chlorhexidine digluconate, a cationic bisbiguanide antiseptic, and sodium hexametaphosphate (HMP), a polyanionic phosphate compound, resulting in the formation of an insoluble CHX–HMP particulate phase in an aqueous medium.

100 mL of an aqueous sodium hexametaphosphate solution (10 mmol/L) was added to 100 mL of an aqueous chlorhexidine digluconate solution (10 mmol/L) under continuous stirring at ambient temperature and pressure. The solutions were mixed under constant stirring to facilitate interaction between the two components. The immediate formation of a colloidal suspension following the addition of HMP to the chlorhexidine solution was taken as evidence of CHX–HMP particle formation. Following combination of the two solutions, the final concentrations of both chlorhexidine digluconate and sodium hexametaphosphate in the suspension were 5 mmol/L. The resulting suspension was used for subsequent characterisation.

Scanning Electron Microscopy and Energy-Dispersive X-ray Spectroscopy (SEM–EDX)

The morphology and elemental composition of the synthesised CHX–HMP nanoparticles were examined using a scanning electron microscope equipped with energy-dispersive X-ray spectroscopy (SEM–EDX; JEOL JSM-IT800 SHL). Specimens were prepared and mounted using an appropriate procedure for microscopic examination. Where required, specimens were dried and rendered conductive before imaging, following the laboratory's established preparation protocol.

SEM was used to examine the morphological characteristics of the particles, including their general appearance, surface features, and extent of aggregation. Representative micrographs were obtained for qualitative assessment of particle morphology. EDX analysis was performed to assess the elemental composition of the prepared material, with particular attention to the detection of phosphorus and chlorine, which are relevant to the phosphate component and chlorhexidine-containing material, respectively. The SEM and EDX findings were interpreted together to describe the surface morphology and elemental characteristics of the synthesised particles.

Fourier-Transform Infrared Spectroscopy (FTIR)

Fourier-transform infrared spectroscopy was performed using a PerkinElmer Spectrum 2 spectrometer to investigate the chemical characteristics of the synthesised CHX–HMP material. The analysis was intended to identify characteristic infrared absorption bands associated with the functional groups present in the constituent

compounds and to assess spectral features that may be consistent with their interaction during particle formation.

The acquired spectra were examined for characteristic absorption bands and their positions, shapes, and relative intensities. Where reference spectra of the individual starting materials were available, these could be compared with the spectrum of the synthesised CHX–HMP material to identify relevant similarities or differences. Spectral interpretation was based on the observed bands and their assignments; FTIR findings were not considered independently conclusive evidence of nanoparticle formation.

Surface Topography and Roughness Analysis

Surface topography was evaluated using a SensoFar S-Neox 5-Axis optical profilometer. Three-dimensional optical surface measurements were intended to characterise surface features and quantify roughness in specimens prepared for surface analysis. Measurements were acquired using the instrument's optical profiling system, and the resulting surface data were processed using the associated analysis software.

Surface topography was assessed from the reconstructed three-dimensional surface maps, while quantitative roughness parameters were extracted from the measured surface data. The specific roughness parameters reported, together with their units, were defined according to the analysis protocol used. Measurements were to be performed using consistent acquisition and processing conditions across the specimens to support valid comparison.

RESULTS

Surface Topography and Roughness Analysis

Three-dimensional optical profilometry demonstrated measurable differences in the surface topography of the uncoated and CHX–HMP-coated stainless-steel screws. Following coating, the arithmetic mean height (Sa) increased from 2.7693 µm to 3.0730 µm, representing a 10.97% increase. The root mean square surface height (Sq) increased from 5.0708 µm to 5.8607 µm (15.58%), while the maximum surface height parameter (Sz) increased from 90.1902 µm to 149.6376 µm (65.91%).

Profile-based analysis further demonstrated an increase in arithmetic mean roughness (Ra) from 0.2222 µm to 0.3822 µm (72.01%), accompanied by increases in root mean square profile roughness (Rq), from 0.2842 µm to 0.4700 µm (65.38%), and maximum profile height (Rz), from 1.3440 µm to 2.0011 µm (48.89%). Collectively, these findings indicate an increase in the measured surface-height variation following CHX–HMP coating, suggesting that the coating process was associated with changes in the surface topography of the analysed screw.

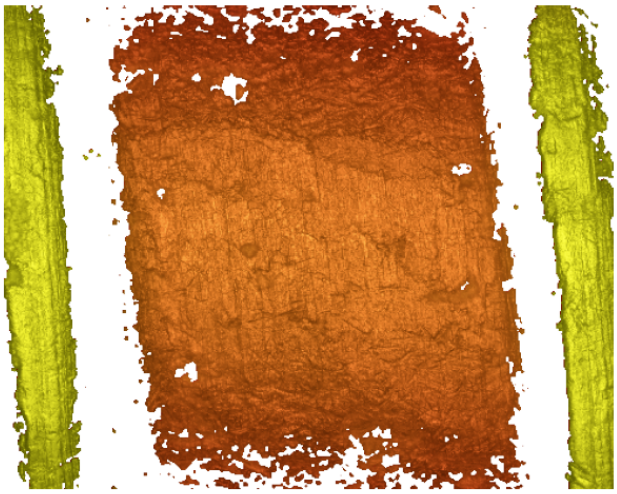


Figure 1: Surface roughness as seen under the optical profilometer

Scanning Electron Microscopy (SEM)

SEM examination of the CHX–HMP-coated screw revealed an irregular surface morphology with variations in surface texture and topography across the analysed region. The observed surface features were consistent with a modified surface following the coating procedure. Although the micrograph provided morphological evidence of surface alteration, the presence and uniformity of the deposited coating could not be established from morphology alone.

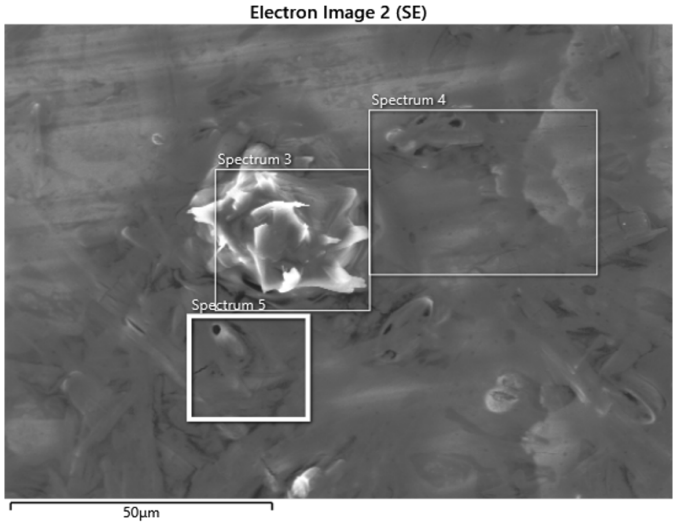


Figure 2: SEM image seen under magnification

Energy-Dispersive X-ray Spectroscopy (EDX)

EDX analysis of three selected regions of the coated screw demonstrated the presence of carbon, oxygen, nitrogen, sodium and chlorine, with variations in their relative proportions across the analysed areas. Chlorine concentrations ranged from 1.7 to 3.4 atomic %, while nitrogen ranged from 8.4 to 9.0 atomic %. Iron and chromium were also detected, and nickel was identified in one region, indicating contributions from the underlying stainless-steel substrate.

The detection of chlorine and nitrogen was compatible with the presence of chlorhexidine-containing material, while sodium and oxygen were also identified in the analysed regions. However, phosphorus was not clearly reported in the available quantitative EDX summaries. Thus, the elemental findings support the presence of chemical constituents compatible with the coating formulation, although confirmation of both components of the CHX–HMP coating requires further evidence.

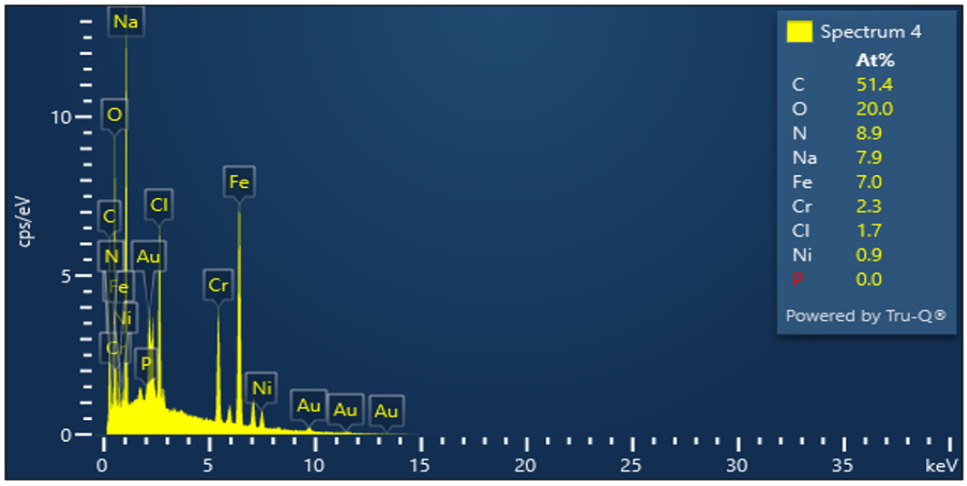


Figure 3: EDX representation of elements

Fourier-Transform Infrared Spectroscopy (FTIR)

FTIR analysis revealed differences between the spectra of the uncoated and CHX–HMP-coated screws. The uncoated specimen exhibited labelled absorption features near 2922.80, 2322.97 and 1015.96 cm^{-1}, whereas the coated specimen showed a feature near 2934 cm^{-1} and several additional labelled bands across the fingerprint region. These differences indicate changes in the recorded spectral profiles following the coating procedure.

The additional spectral features may reflect contributions from chemical constituents associated with the coating. However, because the available spectra were image-based and reference spectra of the individual constituents were unavailable, definitive functional-group assignments could not be established. The FTIR findings therefore provide preliminary evidence of spectral differences between the coated and uncoated specimens.

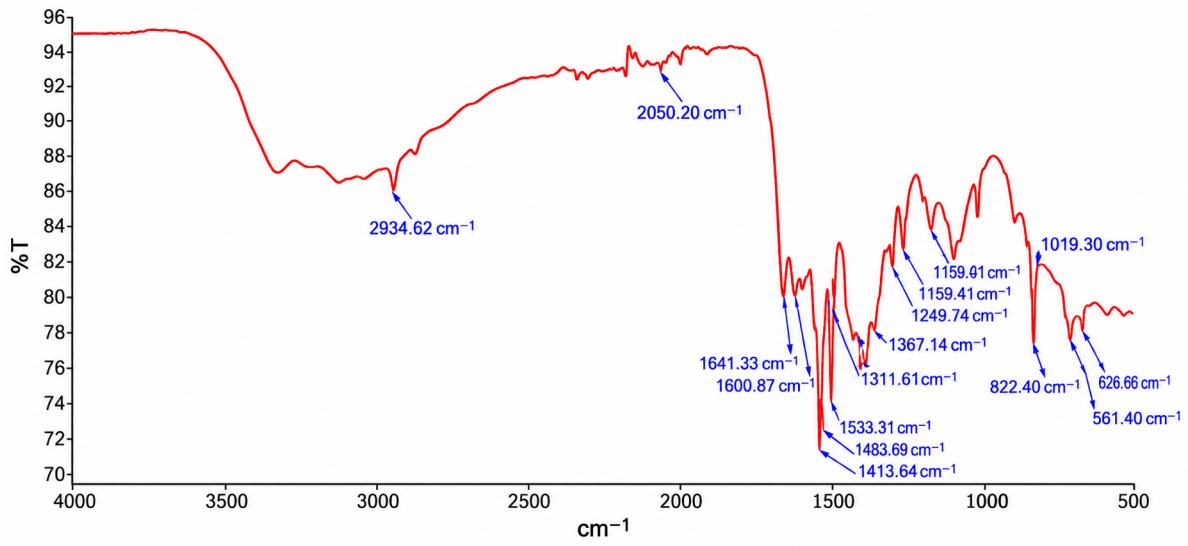


Figure 4: FTIR spectra of CHX-HMP coating

Overall Findings

The combined characterisation findings demonstrated differences in surface topography, morphology and spectral features following the CHX–HMP coating procedure. Optical profilometry showed increases in the measured areal and profile roughness parameters, while SEM revealed an irregular surface morphology. EDX identified chlorine and nitrogen among the detected elements, and FTIR demonstrated differences in the absorption features of the coated and uncoated specimens.

Overall, the findings provide preliminary evidence of surface modification following the CHX–HMP coating procedure. While the results are encouraging and support further evaluation of the coated stainless-steel mini-implant, definitive confirmation of coating composition and statistical validation of the observed roughness changes remain necessary.

DISCUSSION

The present study demonstrated an increase in surface roughness following CHX–HMP coating of stainless-steel orthodontic mini-implants. The increase in Sa and Ra suggests that the coating procedure was associated with changes in the measured surface topography. Garner et al. (13), however, reported no statistically significant difference in Ra between uncoated sandblasted, acid-etched titanium and either of their CHX–HMP coating groups, although roughness differed between the two coating concentrations. This variation may reflect differences in substrate characteristics, coating concentration, and deposition methods.

The surface modification observed in the present study may be considered alongside the findings of Barbour et al. (Barbour(14), who demonstrated that CHX–HMP nanoparticles adhered to different substrates and provided sustained chlorhexidine release for at least 50 days. Wood et al. (17) subsequently demonstrated nanoparticle deposition on titanium implant surfaces, with sustained chlorhexidine release for 99 days and antimicrobial activity against Streptococcus gordonii. These studies provide a scientific basis for investigating CHX–HMP as a potential antimicrobial coating for orthodontic mini-implants.

Evidence specific to orthodontic applications is also available. Kamarudin et al. ((16) demonstrated sustained chlorhexidine release from CHX–HMP-coated elastomeric ligatures for up to eight weeks following ethanol conditioning. Hasan and Alhuwaizi (19–21) further reported antimicrobial and antibiofilm activity of CHX–HMP-coated orthodontic mini-implants against selected oral microorganisms. Nevertheless, these findings cannot be directly extrapolated to the present specimens, as antimicrobial efficacy and sustained-release behaviour were not assessed in the current characterisation study.

Garner et al.(13) also demonstrated a reduction in multispecies biofilm formation on coated titanium surfaces, while assessing cytocompatibility using human mesenchymal stem cells. Similarly, Barbour et al. (18) reported sustained antimicrobial activity of CHX–HMP-coated wound-care materials, with lower toxicity to human placental cells than commercial chlorhexidine dressings. Carvalho et al.(22) found that incorporating CHX–HMP nanoparticles improved the antimicrobial activity of tested root canal sealers, although certain physicochemical properties were also affected. Collectively, these findings support the potential of CHX–HMP across different biomedical and dental materials, while highlighting the need to evaluate coating performance for each specific application.

Importantly, Al-Hilaly and Alhuwaizi (23), in a randomised clinical trial involving 80 orthodontic mini-implants, found no statistically significant improvement in success rate, mobility, peri-implant health, or pain perception with CHX–HMP coating over four months. This indicates that promising in vitro antimicrobial findings do not necessarily translate into improved clinical outcomes. In the present study, the detection of chlorine

and nitrogen by EDX and the differences in FTIR spectra provide preliminary evidence of surface modification; however, the absence of clearly documented phosphorus detection limits confirmation of the complete coating composition. Further compositional verification, replicate-based roughness measurements, and direct antimicrobial testing are therefore necessary to establish the reproducibility and functional significance of the observed changes.

Limitations

The present study was limited by the availability of descriptive surface characterisation data without documented replicate-level measurements, which precluded statistical comparison of surface roughness parameters. FTIR interpretation was restricted by the absence of raw spectral data and reference spectra, while phosphorus was not clearly identified in the available quantitative EDX reports. Consequently, definitive confirmation of CHX–HMP coating composition could not be established. Furthermore, antimicrobial efficacy, coating adhesion, corrosion resistance, and cytocompatibility were not evaluated..

CONCLUSION

The present study demonstrated measurable differences in surface roughness and qualitative differences in the morphological, elemental, and spectral characteristics of CHX–HMP-coated and uncoated stainless-steel orthodontic mini-implants. The findings provide preliminary support for the feasibility of surface modification using the CHX–HMP coating approach. However, further studies incorporating reproducible surface measurements, definitive compositional characterisation, and antimicrobial and biological testing are necessary to establish coating integrity, functional efficacy, and potential clinical applicability.

AI DISCLOSURE STATEMENT

Artificial intelligence (AI) tools were used to assist with language refinement and improving the clarity and readability of the manuscript.

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