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Original Article

Surface Characterization and remineralization Potential of Dental Floss Coated with Nano-Hydroxyapatite and Sodium Fluoride, An In-Vitro Study

Namrah Ayaz[1], Muhammad Sajid[2], Romana Noreen[3], Faizan Hussain[4], Amber Kiyani[5] , Uzma Hassan[6], Usman Mahmood [7], Junaid Israr Ahmed Khan [8]

[1] Demonstrator, Department of Science of Dental Materials, Islamic International Dental College, Islamabad
[2]Professor and Incharge Department of Dental Materials, Islamic International Dental College, Islamabad
[3] Assistant Professor, Department of Operative Dentistry and Endodontics, Nishtar Institute of Dentistry, Multan
[4] Demonstrator, Department of Operative Dentistry, School of Dentistry/SZABMU Islamabad
[5]Assistant Professor. Pre-Clinical Sciences, Qatar University, College of Dental Medicine
[6]Assistant professor Dental Materials Islamic international Dental college, Islamabad
[7] Professor, Department of Science of Dental Materials, Lahore Medical and Dental College, Lahore
[8] Professor, Department of Orthodontics, Lahore Medical and Dental College, Lahore

Author`s Contribution

[1,3,6]Drafting the work or revising it critically for important intellectual content, analysis, or interpretation of data for the work, [2]Final approval of the study to be published, [4,6]Substantial contributions to the conception or design of the work; or the acquisition, [5]analysis, drafting, [7,8]Active participation in active methodology,

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Correspondence
**E: Namrahayaz3@gmail.com*

ABSTRACT

Objective: To evaluate and compare the remineralization potential of Nano hydroxyapatite (HAP) and sodium fluoride (NaF)– coated dental floss on demineralized enamel surfaces of sound teeth.

Methodology: This experimental study was conducted on sound extracted teeth using untreated, Nano hydroxyapatite (Nano-HAP)-coated, and sodium fluoride (NaF)–coated dental floss, after taking approval from the ethical committee of Islamic International Dental College from Feb 2024 to August 2024. Sound teeth (Absence of caries, cracks, abrasion, restorative materials, hypoplasia, white spot lesion, staining and other enamel defects). Non-Carious teeth extracted for orthodontic cases were included in the study through convenience sampling technique. While the carious, deciduous, and fractured teeth were excluded from the study. Samples were tested at 7, 14, and 21 days with regular flossing in artificial saliva. Enamel changes were analyzed using Scanning Electron Microscopy and Energy Dispersive X-ray Spectroscopy (EDS).

Results: Scanning Electron Microscope (SEM) confirmed successful deposition of Nano-HAP and NaF coatings on dental floss, with distinct morphological characteristics. EDS verified their elemental composition, demonstrating the presence of calcium and phosphorus in Nano-HAP and sodium and fluorine in NaF. Enamel treated with Nano-HAP showed earlier and more uniform remineralization, smoother morphology, and higher Ca/P ratios across 7, 14, and 21 days, while NaF exhibited slower deposition with gradual surface improvements over time.

Conclusion: Both Nano- hydroxyapatite- coated and sodium fluoride -coated dental floss enhanced enamel remineralization, yet Nano- hydroxyapatite exhibited superior and earlier effects, highlighting its potential as a biocompatible alternative to fluoride in preventive oral care.

Keywords: Artificial saliva, Dental floss, Enamel remineralization, Fluoride, Microscopy, Nano-hydroxyapatite

Introduction

Dental caries and enamel degradation are among the most prevalent oral health problems worldwide, affecting individuals across all age groups. According to World Health Organization (WHO), untreated dental caries is the most common oral condition globally, with approximately 2.5 billion suffering from caries in permanent teeth and more than 514 million children affected in primary teeth.[1] In Pakistan, the burden is also alarming, with a pooled prevalence of 56.5%, affecting more than half of the population, including approximately 62% of children and 51% of adults.[2] Early childhood caries is observed as major dental ailment around the globe.[3] Dental patients may complaint of pain, swelling, and hypersensitivity. [4-6]

It is implicit now-a-days, that oral health is exceptionally vital for over-all physical health [7], with growing evidence linking oral ailments to systemic health conditions [8,9]. Periodontal sicknesses and caries are amongst those factors which had affected oral health-related quality of life as well.[10]

Recently, coated dental floss has gained a reputation due to their potential to deliver therapeutic agents directly to interproximal spaces by providing therapeutic compounds directly to interproximal spaces areas frequently challenging to clean successfully with traditional brushing alone coated dental floss offers a novel way to improve oral hygiene. Integrating antimicrobial agents into dental floss enhances its efficacy in controlling biofilm formation and reducing the risk of periodontal disease.[11] Remineralization of demineralized enamel is a key process for reversing early carious lesions and protecting against enamel erosion by redepositing calcium and phosphate ions.[12]

Nanohydroxyapatite (HAP) is a promising remineralizing agent in dentistry.[13] Being a naturally occurring calcium phosphate form, HAP is essential for the upkeep and restoration of dental tissues, especially when remineralizing enamel. HAP can be used in a variety of dental procedures targeted at improving oral health in general and reversing early carious lesions in particular. [14] Creating coated floss can significantly improve a patient's flossing habits by making the process more comfortable, and effective. Additionally, coatings like wax or fluoride can enhance effectiveness by promoting better plaque removal.

Coated floss with Nano-HAP and NaF have been explored as an aid for interdental plaque removal and remineralization potential. However, both materials have not been compared in the same study. Also, to our knowledge, no study compared the remineralization effect within the same conditions as oral cavity in-vitro. Therefore, the objective of this study was to evaluate and compare the remineralization potential of Nano hydroxyapatite (HAP) and sodium fluoride (NaF)– coated dental floss on demineralized enamel surfaces of sound teeth.

Methodology

This experimental in-vitro study was conducted at Islamic International Dental College, with the remaining testing conducted at the School of Chemical and Materials Engineering NUST Islamabad and Institute of Space Technology after the ethical review board approval. The duration for this study was 06 months (Feb 2024 to August 2024). Sound teeth (Absence of caries, cracks, abrasion, restorative materials, hypoplasia, white spot lesion, staining and other enamel defects). Non-Carious teeth extracted for orthodontic cases were included in the study through convenience sampling technique. While the carious, deciduous, and fractured teeth were excluded from the study.

Sound extracted teeth using untreated dental floss (group 1), Nano hydroxyapatite (Nano-HAP)-coated dental floss (group 2), and sodium fluoride (NaF)–coated dental floss (group 3) were used. Groups were followed for 7, 14 and 21 days. Chemical formula for Nano-HAP is "$Ca_{10}(PO_4)_6(OH)_2$". Oral-B waxed dental floss was used and purchased from local company in Pakistan name Dawa Healthcare. The dental floss was composed of fine nylon (20–30 μm in diameter) bound together by a binder.

Tooth samples were divided into three-time intervals: 7, 14, and 21 days. For each sample, one side served as the control, received no treatment at all. To keep anything from clinging to the control side, Vaseline was applied, and the other side was treated with a slurry of nanohydroxyapatite and sodium fluoride, which was applied using dental floss twice a day. This in-vitro investigation was carried out using a single experimental specimen. Surface characterization and re-mineralization assessment were the main strengths of this methodology, in addition to one side serving as the control, for each sample.

Slurry Preparation: The Oral-B waxed dental floss, 30 cm long stirred in 10ml of deionized water, and 0.9g of NaF and 0.1g of Nano-HAP was added. The solution was then placed on a hot plate with a magnetic stirrer and stirred for 10 minutes. A dip-coating process was employed to coat 30 cm long segments of dental floss with nano-hydroxyapatite (Nano-HAP) and sodium fluoride (NaF). The coating solution was prepared by dissolving 0.1g of Nano-HAP and 0.9g NaF in 10 mL of deionized water. The solution was placed on a magnetic stirrer and stirred continuously to obtain a homogenous suspension. Each floss segment was immersed in the prepared coating solution and incubated under constant stirring to ensure uniform deposition of the coating material. Following incubation, the floss was removed from the solution and dried at room temperature. After drying, the coated floss was applied interproximally between teeth for further evaluation

For the initiation of re-mineralization, all teeth were immersed and stored in artificial saliva for 7,14 and 21 days at normal room temperature. The materials used for composition of artificial saliva are shown in Table I

Table 1: Contents of artificial saliva.	
Material	Chemical formula
Deionized water	H2O
Sodium chloride	NaCl
Potassium chloride	KCL
Calcium chloride	CaCl2
Disodium hydrogen phosphate	Na_2 HPO_4
Sodium sulfide nano hydrate	$Na_2S \cdot 9H_2O$

Characterization Techniques: To investigate the coating uniformity and surface morphology of the coated dental floss Scanning Electron Microscope was utilized, providing a detailed overview of the surface of the dental floss and enabling a comparison between the untreated dental floss and treated floss.

Scanning Electron Microscope: (manufactured by JEOL, model #JSM-6490A) was used to provide a detailed overview of the coating uniformity of the dental floss and enabling a comparison between the untreated dental floss and treated floss. As well as examine the deposition and surface morphology of controlled (untreated side), nano-hydroxyapatite, and NaF coated teeth. This enabled us to compare the controlled side with treated side after 7, 14, and 21 days.

Elemental Analysis: Energy Dispersive X-ray Spectroscopy (EDS) was used for elemental analysis of dental floss.

Results

The tooth samples were initially divided into a control group (Group A) and treatment groups. The control group consisted of untreated samples that did not receive any intervention. The treatment groups were further categorized into Group B and Group C based on the type of coating applied. Group B samples were treated with nano-hydroxyapatite (Nano-HAP)-coated dental floss, while Group C samples received sodium fluoride (NaF)-coated dental floss. Each treatment group was further subdivided according to the duration of intervention into three subgroups: 7 days, 14 days, and 21 days. Specifically, Group B included B1 (7 days), B2 (14 days), and B3 (21 days), whereas Group C included C1 (7 days), C2 (14 days), and C3 (21 days). This grouping allowed for a comparative evaluation of the remineralization effects of Nano-HAP and NaF over different time intervals.

Scanning Electron Microscope showed that Nano-HAP powder and Sodium fluoride particles exhibits elongated shape. Particle sizes are shown in table II.

EDS verifies that carbon and oxygen are the main elements in all the dental floss spectrums suggesting its polymeric nature, while calcium and phosphorus, which are key indicators of nano-hydroxyapatite, are moderately present on the floss surface across all spectrums. Energy Dispersive X-ray Spectroscopy (EDS) spectrum confirms the elemental composition of the hydroxyapatite sample, by demonstrating the prominent peaks of calcium (Ca), phosphorus (P), and oxygen (O). While EDS spectrum of NaF confirms the presence of fluorine, sodium, and carbon in its sample. As shown in table III.

Table II: Particle sizes of study samples as revealed by scanning electron microscopy.	
Material	Particle size
Nano-HAP powder	35nm
Sodium fluoride	4 µm.

Table III: Elemental composition of study samples.	
Study sample	Composition
Nano-HAP powder	Calcium (major constituents), Phosphorus, Oxygen
Sodium fluoride	Fluorine, Sodium (major constituents) Carbon (smaller amount)

Nano-HAP Analysis: Particles observed on the enamel surface may be attributable to the nanohydroxyapatite administered through the floss. The flossing action facilitated the dispersion of these particles into microcracks and fissures, enhancing remineralization at the nanoscale after 7 days. While after 14 days, SEM images depict enhanced infiltration of Nano-HAP particles into microcracks and fissures, contributing to a smoother texture. The coating suggests active remineralization with increased surface coverage and adhesion. After 21 days follow up, the texture changes suggest the formation of a protective layer, with minimal remaining surface irregularities or fissures. Nano HAP particles successfully integrated into the enamel structure, confirming their role in enhancing surface integrity over time.

Magnification demonstrated an enhanced deposition of fluoride on the enamel surface, visible as a denser and more uniform layer after 7 days. Whereas, after 14 days, SEM images depict slow particle adherence and incomplete surface coverage. Surface irregularities remained visible, although slight reductions in texture roughness were observed. When seen after 21 days, surface coverage improved compared to 14 days, with smoother regions and reduced irregularities. The slower deposition rate of NaF suggests its potential for sustained remineralization effects over longer periods.

Discussion

Tooth decay, caused by acidogenic bacteria, remains a major global oral health issue. While fluoride effectively enhances remineralization, its long-term use can cause fluorosis and systemic effects. This has prompted research into biocompatible alternatives like Casein Phosphopeptide-Amorphous Calcium Phosphate (CPP-ACP) and Nano-Hydroxyapatite (Nano-HAP). Nano-HAP, similar in composition to enamel, can repair early lesions, seal dentinal tubules, and improve surface hardness by releasing calcium and phosphate ions that maintain mineral balance on tooth surfaces.[15,16]

The present findings demonstrated that nylon-based dental floss, composed of tightly bound fine fibers and coated with wax, provided a suitable substrate for Nano-HAP and NaF coating. The smooth and ordered morphology of untreated floss observed under SEM was consistent with earlier observations by Babayevska et al [11] and Stanojević et al [17] confirming the structural uniformity of commercial floss materials. After coating, the Nano-HAP-modified floss exhibited a noticeably rougher and more irregular texture, indicating successful deposition of Nano-HAP clusters. Similar irregular coverage patterns were reported by Babayevska et al [11], who found rod-like particles randomly distributed across the floss surface. The present EDS results confirmed the presence of calcium and phosphorus as dominant elements, reinforcing that the coating was primarily composed of hydroxyapatite. These findings align with those of Kert et al.[18]

The demineralized samples displayed noticeable surface dissolution, irregular prism structures, and loss of texture, consistent with prior findings.[19,20] Similar microstructural degradation and reduction in calcium-to-phosphorus ratios after acid exposure were also noted by Besnard [21]. Collectively, these findings confirm that phosphoric acid treatment effectively mimics early-stage carious lesions suitable for remineralization assessment.

Regarding enamel morphology, the untreated control tooth samples in this study retained smooth, uniform surfaces with mild microcracks, validating their suitability as baseline specimens. This observation parallels earlier work by another researcher, who demonstrated that mild microporosity is a natural feature of enamel after demineralization.[22].

Following 7, 14, and 21 days of application, Nano-HAP-coated floss induced progressive surface recovery, with SEM revealing agglomerated hydroxyapatite deposits gradually transforming into a smoother, more uniform enamel surface. These results strongly support the potential of Nano-HAP for gradual remineralization through crystal deposition and microcrack filling. Comparable enamel restoration has been observed in previous studies, all reporting that Nano-HAP enhances the closure of enamel defects and promotes mineralized layer formation.[22-24] The hydrophilic nature of Nano-HAP may further contribute to its ability to adhere to enamel and facilitate homogeneous remineralization.[23] EDS spectra of treated enamel confirmed the reappearance of calcium and phosphorus peaks, reinforcing the notion that Nano-HAP facilitates mineral re-deposition like natural enamel.

In comparison, NaF-coated floss produced immediate but relatively superficial remineralization. After 7 days, dense and uniform fluoride deposition was observed, leading to reduced surface porosity. However, at later intervals, the improvement in surface integrity appeared less pronounced than in the Nano-HAP group, suggesting that fluoride treatment primarily affects surface layers without inducing deep subsurface remineralization. Similar patterns have been reported by previous researchers, where fluoride enhanced the outer enamel mineral density but did not completely restore the microstructural organization. These findings indicate that although NaF effectively prevents further mineral loss, Nano-HAP may achieve more integrated and biologically stable restoration.[25,20]

When comparing both agents, the present results suggest that Nano-HAP offers advantages beyond fluoride's chemical mode of action. While NaF enhances remineralization by forming a protective fluorapatite layer resistant to acid dissolution, Nano-HAP directly integrates into the enamel matrix, physically replenishing lost hydroxyapatite crystals. This structural reinforcement may explain the superior smoothness and reduced fissures observed over prolonged application. Tschoppe also reported greater mineral deposition in Nano-HAP-treated samples compared to those treated with fluoride-based formulations. [26]

Conclusion

Both Nano- hydroxyapatite- coated and sodium fluoride -coated dental floss enhanced enamel remineralization, yet Nano- hydroxyapatite exhibited superior and earlier effects, highlighting its potential as a biocompatible alternative to fluoride in preventive oral care.

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