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Effect of tetracalcium phosphate/monetite toothpaste on dentin remineralization and tubule occlusion in vitro

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ABSTRACT

Objectives. To investigate the tubule occlusion and remineralization potential of a novel toothpaste with active tetracalcium phosphate/monetite mixtures under de/remineralization cycling.

Methods. Dentin de/remineralization cycling protocol consisted of demineralization in 1% citric acid at pH 4.6 with following remineralization with toothpastes and soaking in artificial saliva. Effectiveness of toothpastes to promote remineralization was evaluated by measurement of microhardness recovery, analysis of surface roughness, thickness of coating and scanning electron microscopy.

Results. The novel tetracalcium phosphate/monetite dentifrice had comparable remineralization potential as commercial calcium silicate/phosphate (SENSODYNE®) and magnesium aluminum silicate (Colgate®) toothpastes and significantly higher than control saliva ($p < 0.02$). Surface roughness was significantly lower after treatment with prepared and SENSODYNE® dentifirice ($p < 0.05$). The coatings on dentin surfaces was significantly thicker after applying toothpastes as compared to negative control ($p < 0.001$).

Conclusions. The new fluoride toothpaste formulation with bioactive tetracalcium phosphate/monetite calcium phosphate mixture effectively occluded dentin tubules and showed good dentin remineralization potential under de/remineralization cycling. It could replace professional powder preparation based on this mixture. It was demonstrated that prepared dentifrice had comparable properties with commercial fluoride calcium silicate/phosphate or magnesium aluminum silicate dentifrices.

1. Introduction

The dentin hypersensitivity (DH) represents an increasing problem in dentistry and it is one of the common clinical problems concerning human teeth. The incidence of hypersensitivity ranges from 10% to 30% of the general population and various procedures for the DH reduction based on simple home desensitizing or professional therapies such as surgery, pulpectomy or laser treatments are utilized [1,2]. It

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was found that the prevalence of DH ranged from 60% to 98% in patients with periodontal conditions. The most widely accepted theory which explains the biological mechanism of DH is the hydrodynamic theory based on pain-inducing stimuli due to increase in fluid flow within the dentinal tubules and the stimulation of mechanoreceptor nerves in the central end of tubules [3,4]. Considerable research effort has been expended on understanding the processes and reasons leading to DH so as on developing effective treatments for its prevention, which could be divided into two main types — nerve desensitization or occlusion of dentinal tubules. It is known that potassium ions (in the form of nitrate, chloride and citrate salts) are active depolarizing agents, which are believed to act by reducing the excitability of intra-dental nerves and removing the discomfort associated with DH [5]. The reduction in dentin permeability involves the use of dentin tubule blocking agents such as potassium oxalate, calcium phosphates, arginine–calcium carbonate or dental adhesive materials [6]. Strontium salts may act as desensitizers by biomechanical blocking of neural transmission through intradental nerves or may occlude the dentin tubules by precipitation of strontium apatite [7–9]. Arginine combined with calcium carbonate formed insoluble deposites including calcium phosphates and covered dentin tubules [10,11]. The calcium carbonate was identified in occluded surface layer of desensitizing toothpastes (Sr, potassium compounds and sodium silicate) containing this component as abrasive or by the direct precipitation of carbonates due to dentifrice composition after brushing and soaking in the artificial saliva [12]. Stannous fluoride produced precipitates onto dentin, which were water and acid-resistant [13]. Calcium sodium silicate phosphates represent another active system solving DH characterized by the decomposition and precipitation of calcium phosphate and silicate acid resistant layer on surface of dentin, which occludes dentin tubules [14,15]. The study of the influence of experimental calcium silicate cement slurries with an addition of monetite or oxalate on dentinal permeability showed significant reduction in dentinal permeability immediately after application. The permeability reduction was more stable during soaking in artificial saliva than that of professional bioxalate or polymethacrylate cements (D-Sense Crystal and Clearfil S3-Bond) [16]. The single dentin treatment with Portland cement (calcium silicate) experimental dentifrice and commercial oxalate-based toothpastes significantly decreased the dentin permeability as the result of created microcrystals and precipitates on the dentin surfaces with reduction in the diameter of dentinal tubules and enhancing the acid resistance [17].

The phosphate and fluoride compounds currently applied in toothpaste formulations represent sodium phosphate, calcium phosphates (brushite, amorphous calcium phosphates, casein phosphopeptide-stabilized amorphous calcium phosphate (CPP-ACP), functionalized β-tricalcium phosphate (fTCP), nanohydroxyapatite), sodium monofluorphosphate, calcium glycerol phosphate, NaF, amine fluoride and compounds of Zn, Sr or Sn [18–22]. Fluorides enhanced the mineralization of hydroxyapatite and reduced hydroxyapatite solubility by the formation of fluorohydroxyapatite or CaF_2 precipitates, which can block fluid movement through tubules [23,24]. On the other hand, casein phosphopeptide–amorphous calcium phosphate toothpaste only partially reduced the dentin permeability with precipitates dissolving in saliva and citric acid. This is an evidence that such a type of calcium phosphate dentifrice originally developed to reduce the formation of enamel caries lesions was not sufficiently active in single application for solving dentin hypersensitivity [18]. Strontium acetate and arginine-based dentifrices (8 wt% each) with the same content of fluorides (1100 ppm) showed the statistically significant dentin tubular occlusion compared to controls and following the strong dietary acidic challenge (ex vivo) had enhanced impact on the occlusion provided by the arginine contrary to strontium acetate toothpaste [25]. From the point of view of remineralization potential of dentifrices, the higher remineralization effect on dentin lesions was achieved with n-HAp or $ZnCO_3$/n-Hap toothpastes compared to the amine fluoride dentifrice probably as the result of a higher pH value of slurry favored remineralization by the incorporation of n-HAp particles into the dentin lesions [26]. The highly effective dentifrices with anti-caries protection and remineralization potential contain active calcium silicates, calcium silicate/phosphates (NovaMin™) or bioactive glasses (e.g.Bioglass 45S5®), which are characteristic with ability to deposition of the uniform nanohydroxyapatite coating on enamel or dentin surfaces [27,28]. Nanohydroxyapatite was used mainly in the form of slurries in aqueous solutions directly applied on eroded enamels or added into acid drinks [29,30]. Remineralizing potential and dentin tubule occlusion were evaluated using toothpastes containing the small additions of hydroxyapatite (1–3 wt%) and carboxymethyl cellulose hydrogels with up to 30 wt% content of hydroxyapatite [31–33]. Note that the dissolution and bioactivity of stable calcium phosphates (like e.g. hydroxyapatite or tricalcium phosphate) are limited by the physico-chemical properties of surrounding (e.g. pH, presence of organic species), their inner crystalline structure (ordering) and morphology of particles. [34–36]. It is known that enhanced bioactivity of biocements composed of tetracalcium phoshate/monetite mixtures in bone defect healing [37,38] was utilized for the preparation of active commercial dentin desensitizer based on fluoride-free powder tetracalcium phosphate/monetite mixture (TEETHMATE® desensitizer, Kuraray Noritake Dental Inc.) directly applied on dentin as slurry in phosphate solution [39,23].

The aim of this paper was to develop biocompatible toothpaste (chemical, structural biocompatibility of final hydroxyapatite product, no adverse reaction to surrounding tissue, no cytotoxicity) containing an active tetracalcium phosphate/monetite mixture (TTCPM) and to compare its potential for both the occlusion of dentin tubules and dentin remineralization under de/remineralization cycling with actual commercial products SENSODYNE® (SEN, GlaxoSmithKline, UK) and Colgate® (COL, Colgate-Palmolive, China). The prepared fluoride toothpaste (TTCPMF) contained TTCPM and the same fluoride amount as commercial dentifrices (1450 ppm F- as NaF). The surface hardness recovery and microstructure of dentin surface were studied. The primary hypothesis is that prepared TTCPMF paste significantly reduced number of open dentin tubules, surface roughness and promoted surface microhardness recovery under cycling than only artificial saliva treated dentin samples (AS) (neg-