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# The control of phosphate ion release from ion permeable microcapsules formulated in to rosin varnish and resin glaze

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## ABSTRACT

**Objectives.** The occurrence of recurrent caries at the interface of dental materials and the enamel surface is an important performance issue. The objective of this study was to investigate the most effective way to control the release rate of bioavailable phosphate ions contained in aqueous solutions within ion permeable microcapsules formulated in to rosin based varnishes and resin based sealants, in order to promote remineralization.

**Methods.** Microcapsules that contained aqueous solutions of  $K_2HPO_4$  with concentrations from 0.8 to 7.4 M were prepared. 3–50 w/w% of microcapsules were loaded into both rosin and resin based dental formulations.

**Results.** The effect of initial salt solution concentration inside the microcapsules and weight percent loading of the microcapsules on release rate were contrasted. The effect of microcapsule loading was found to be highly dependent on the continuous phase. In rosin, 3–15 w/w% loading resulted in rapid release of ions. Higher weight percent loadings were initially slower but resulted in sustained release of ions. In resin, 3–15 w/w% formulations slowly released ions for at least 300 days, while higher loading formulations released an initial burst of ions. Initial salt solution concentration contained inside the microcapsule affected ion release rate. Initial rate of ion release was greatest at a concentration that was less than the maximum concentration studied in both continuous phases.

**Significance.** Phosphate ion release can be controlled from resin or rosin based dental material by adjusting initial salt solution concentration in microcapsules or percent loading of microcapsules. The potential for burst release from a varnish or slow, sustained release from a sealant has been demonstrated.

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## 1. Introduction

From a historical perspective, the field of dentistry is beginning to broaden its direction of patient care from surgical

procedures (e.g. restorations) to preventative approaches that work to interrupt the caries process [1]. Tooth decay continues to affect adults and children, and can be influenced by factors such as consumption, hygiene, and fluoride exposure [2,3]. As this epidemic lingers among society the methods of

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prevention have been forced to evolve. This is partly because when successful, preventative methods can provide a cost-effective treatment plan to those susceptible to decay and to those with limited options for treatment. Oral hygiene is the easiest means of preventing the initiation and spread of bacterial decay. Current products such as varnish, toothpaste and sealants have all targeted this process. One approach to treat this disease is through a course known as tooth remineralization. Remineralization is accomplished when ions such as calcium, fluoride, and phosphate contribute to the rebuilding of enamel. Although there are plenty of products which claim to inhibit demineralization and rebuild tooth structure, the dental field continues to search for new methods that challenge the progression of caries.

The cycle of remineralization and demineralization is a continuous one that takes place naturally in the oral environment, with and without dental therapies. Typically, it starts at the enamel surface. Dental enamel is composed primarily of inorganic mineral (approximately 95–98%), and is often described as hydroxyapatite (HAP) with a molecular formula of  $\text{Ca}_{10}(\text{PO}_4)_6(\text{OH})_2$ . However, actual biological apatite has other impurities (such as carbonate) which can influence the solubility of the tooth structure [4]. It is more accurate to describe enamel as carbonated hydroxyapatite, which has slightly different properties from pure hydroxyapatite [5].

Caries is a disease that affects the tooth by disrupting its inorganic structure. It begins with the ingestion of sugars, often in the form of carbohydrates that are metabolized by bacteria (*Streptococcus mutans*) into organic acids. If the acidic environment is persistent, the tooth structure becomes highly susceptible to decay [6]. *S. mutans* is often affiliated with the caries process because it is both acidogenic and aciduric. When bacterial metabolism occurs, there is a decrease in pH in the fluids surrounding the teeth and gums. HAP dissolves if the pH drops below the critical pH and if the saliva is undersaturated with respect to the tooth [4]. The concentration of ions (e.g. calcium, phosphate) present in the saliva help to determine whether HAP will precipitate or dissociate in the oral environment. The longer the tooth is exposed to a lower pH, the faster the rate of dissolution. Often times tooth decay appears as a white spot, and lesions with this appearance are described as areas of demineralization below the surface of the tooth [2–4]. The outer surface is believed to form as calcium and phosphate ions diffuse from the enamel and into the plaque fluid [3].

With the help of saliva, remineralization is a process that can naturally take place in the oral environment. Remineralization is stimulated by the supersaturation of ions in the saliva, especially when the pH is greater than the critical pH and the mineral can precipitate [4]. When the pH drops below the critical pH, the saliva is no longer saturated and the tooth will demineralize [4,7]. The cycle of remineralization and demineralization in the mouth is a continuous one, but if ion loss continues unchecked, a permanent carious lesion will result [7]. Mineral loss and repair is determined by the degree of saturation of the fluid; typically, ions needed for tooth repair are provided by saliva in the oral environment [7]. Thermodynamic supersaturation of enamel is determined by the ions which contribute to the structure and the solubility product of the mineral [8]. The progression of mineral loss has been

replicated in numerous experiments as an attempt to understand how the process occurs. While enamel loss in vitro can occur rapidly under certain conditions, caries development in vivo is commonly understood to occur over a longer period of time [9].

The role of fluoride in the process of remineralization has recently been investigated. Aside from its incorporation into drinking water, fluoride has been utilized in products such as cavity varnish, toothpaste, and mouthwash. The greatest challenge facing products aimed at rebuilding tooth structure is the bioavailability of ions able to perform this task. In terms of fluoride (as provided in a clinical setting), bioavailability has been described by White as, “the amount of fluoride taken up by enamel/dentin, the retention of fluoride within enamel and/or dentin, or the ability of the treated surfaces to provide low levels of fluoride in saliva.” [8] It has been claimed that fluoride may be capable of replacing hydroxyl ions in the enamel structure, creating fluoridated hydroxyapatite (FAP) [10]. However, Ten Cate et al. also claimed that the precipitation of fluoridated hydroxyapatite onto the enamel (or rather, unincorporated in the tooth) may also be beneficial [7,10]. The use of fluoride provides a unique benefit to the rebuilding of tooth structure, because when FAP is formed, it provides better protection against acidic environments. This could be explained by the lower critical pH and a lower solubility product of FAP relative to tooth structure [10–12].

In addition to the importance of fluoride ions for remineralization, calcium and phosphate ions must be present for the process of remineralization to occur [13]. This justifies the incentive to create new products which utilize calcium and phosphate for enamel repair. For instance, formulations have included casein phosphopeptide to create calcium phosphate derivatives such as casein phosphopeptide amorphous calcium phosphate (CPP-ACP) [14]. This was devised as an effort to stabilize calcium and phosphate and localize ion release near the tooth surface [14]. The role of CPP as a binding agent has been claimed to be successful because of its ability to localize calcium and phosphate for enamel repair, and has been utilized in products such as Recaldent™ [15]. Amorphous calcium phosphate (ACP) products have also been marketed without the CPP component. Even as an independent structure, ACP is believed to precipitate as HAP [16]. ACP has been utilized in toothpaste and dental composites in an effort to remineralize [15,17]. Another approach uses a calcium sodium phosphate silicate bioactive glass that releases ions when combined with saliva [15,18]. Ideally, when placed in the oral cavity, calcium and phosphate dissociate from the structure and precipitate near the surface of enamel [18].

Despite current efforts, the challenge still remains to create a ubiquitous platform that can deliver bioavailable ions from a variety of dental materials that are capable of promoting remineralization and preventing demineralization. Our research group has recently reported the synthesis of ion permeable microcapsules containing aqueous salt solutions useful for remineralization [19]. Salts that contained either a calcium, phosphate, or fluoride ion were solvated in an aqueous solution within the microcapsule. Key variables that affected the rate of ion release from the microcapsule into nanopure water were identified in the previous study. This approach confirmed the potential of solvated salts to exogenously

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