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The impact of dental varnishes on the immediate surface microhardness and roughness of restorative dental materials: an *in vitro* study

Uticaj dentalnih lakova na mikrotvrdoću i hrapavost površine restaurativnih materijala: *in vitro* studija

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Abstract

Background/Aim. Dental caries is a multifactorial disease that ultimately results in the demineralization of dental tissues and is recognized by the World Health Organization as the most prevalent disease among the global population. Dental varnishes are effective in preventing caries in children. The aim of this study was to investigate the way in which dental varnishes, one of which contained fluoride and the other casein phosphopeptide-amorphous calcium phosphate (CPP-ACP), affect the microhardness and roughness of the three most commonly used restorative materials in pediatric dental practice [resin-modified (RM) glass ionomer cement (GIC), high-viscosity (HV) GIC, and micro-hybrid composite (MHCOMP)]. **Methods.** The study included 60 discs and 60 bars, of which 20 discs and 20 bars each were made from one of the three commonly used dental restorative materials. After preparation, incubation, and subsequent basic measurement, the samples were divided into two subgroups (each subgroup containing 30 discs and 30 bars), and each of them was treated with fluoride or CPP-ACP varnish according to a precisely established protocol. After treatment with varnishes, all samples were retested for microhardness and roughness. **Results.** The application of fluoride or CPP-ACP varnish increased the microhardness of the MHCOMP and RM GIC but reduced the microhardness of HV GIC. The roughness was more pronounced with the composite material, as well as with the RM GIC. **Conclusion.** Prophylactic varnishes containing fluoride and calcium have the potential to alter the microhardness and roughness of dental restorations; that is why carefully selecting the appropriate varnish is important.

Key words:
child; dental cements; fluorides; preventive dentistry.

Apstrakt

Uvod/Cilj. Dentalni karies je multifaktorska bolest koja za krajnji rezultat ima demineralizaciju zubnog tkiva, a Svetska zdravstvena organizacija ga je označila kao najrasprostranjeniju bolest u svetskoj populaciji. Dentalni lakovi su se pokazali efikasnim u prevenciji karijesa kod dece. Cilj rada bio je da se istraži način na koji zubni lakovi, od kojih je jedan sadržao fluor a drugi kazein fosfopeptid-amorfni kalcijum fosfat (KFP-AKF), utiču na mikrotvrdoću i hrapavost tri najčešće korišćena restaurativna materijala u pedijatrijskoj stomatološkoj praksi [smolom modifikovani stakleno-jonomerni cement (SJC), visokoviskozni SJC i mikrohibridni kompozit (MHKOMP)]. **Metode.** U studiju je bilo uključeno 60 diskova i 60 pločica, od kojih je po 20 diskova i 20 pločica bilo napravljeno od jednog od ukupno tri restaurativna materijala. Uzorci su nakon pripreme, inkubacije i sledstvenog bazičnog merenja, podeljeni u dve podgrupe (sa po 30 diskova i 30 pločica), od kojih je svaka tretirana fluoridnim ili KFP-AKF lakom, prema tačno utvrđenom protokolu. Nakon tretiranja lakovima, na svim uzorcima je ponovo testirana mikrotvrdoća i hrapavost. **Rezultati.** Primenom fluoridnog ili KFP-AKF laka povećala se mikrotvrdoća MHKOMP i smolom modifikovanog SJC, ali se smanjila mikrot

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vrdoća visokoviskoznog SJC. Hrapavost je bila izraženija kod kompozitnog materijala, kao i kod smolom modifikovanog SJC. **Zaključak**. Profilaktički lakovi koji sadrže fluor i kalcijum imaju potencijal da promene mikrotvrdoću i hrapavost zubnih nadoknada i zbog toga je veoma važno pažljivo izabrati odgovarajući lak.

Ključne reči:
deca; zub, cement; fluoridi; stomatologija, preventivna.

Introduction

Dental caries, affecting both deciduous and permanent teeth, is recognized as the most prevalent disease among the global population, as reported by the World Health Organization [1]. This multifactorial disease ultimately results in the demineralization of dental tissues. Glass ionomer cement (GIC) is commonly employed as a filling material in dentistry to address both early-stage and cavitated carious lesions in deciduous and permanent dentition [2]. Moreover, their fluoride content and subsequent release enable them to exhibit a cariostatic effect, aiding in the prevention and control of dental caries [3]. Additionally, composites are widely utilized as restorative materials for the treatment of both deciduous and permanent dentition rehabilitation. The wear of light-curing composite resin is influenced by multiple factors, including the material's physical properties, microstructure, resistance to abrasion, and the impact of masticatory forces [4]. Conversely, the wear of GIC represents one of the drawbacks associated with this material [5].

Prophylactic varnishes containing fluoride and calcium are widely employed for the prevention of initial carious lesions following timely diagnosis [6]. Several studies have demonstrated the effectiveness of topical fluoride application in reducing caries incidence [7]. A crucial role of fluoride varnishes lies in the formation of fluorapatite, a compound that exhibits resistance to acidic environments and demineralization. These varnishes can effectively adhere to dental surfaces, ensuring long-term and controlled fluoride release [8].

Amorphous calcium phosphate (ACP) serves as a precursor for both hydroxyapatite and casein phosphopeptide (CPP) [9]. It is noteworthy that ACP represents the first commercially available artificial hydroxyapatite product [10].

The literature suggests that the application of topical varnishes enriched with fluorides and CPP-ACP particles, followed by their incorporation into GIC, can influence the mechanical properties of the material. Specifically, it can affect the surface microhardness and roughness. Some studies have reported a decrease in microhardness, while others have observed an increase when using different restorative materials [11–13].

The primary aim of this study was to determine the microhardness of three restorative materials that we use in our daily practice, and the secondary aim was to compare the effect of varnishes containing fluoride, calcium, and phosphate on the microhardness and roughness of these dental materials.

Methods

The study adheres to the principles outlined in the Helsinki Declaration and has obtained approval from the Ethics Committee of the Faculty of Medicine, University of Banja Luka, Republic of Srpska, Bosnia and Herzegovina, reference Number 18/4.17/23.

For the measurement of microhardness, samples were prepared using pre-made molds (Figure 1).

The molds were used to create discs with a diameter of 15 mm and a thickness of 2 mm. The roughness measurement samples were created using a bar-shaped mold with dimensions of 30 mm × 2 mm × 2 mm (Figure 2).

A total of 120 samples were prepared, comprising 20 discs and 20 bars for each of the three dental materials used: resin-modified (RM) glass ionomer cement (GIC) (Fuji II LC®, GC Corporation Tokyo Japan), high-viscosity (HV) GIC (FUJI IX GP®, GC Corporation Tokyo Japan), and micro-hybrid composite (MHCOMP) Te-Econom Plus® (Ivoclar Vivadent, Schaan, Liechtenstein). To prevent the materials from sticking and ensure easy removal from the mold, the walls of the molds were coated with a layer of soft paraffin.

Fig. 1 – Sample molds.

Fig. 2 – Material samples, in the shape of discs and bars.

RM GIC and HV GIC samples were precisely prepared according to the manufacturer's instructions. After mixing, RM GIC and HV GIC were introduced into the molds with a spatula so that a glass plate was pressed from the bottom side of the mold, not allowing the material to leak, and after filling the mold, the glass plate was also pressed from the top by its weight. The HV GIC was cured at room temperature for 15 min, according to the manufacturer's instructions, and RM GIC was polymerized by the light-curing lamp (Woodpecker LED.B, 1,200 mW/cm^2, wavelength: 385 nm – 515 nm, Guilin Medical Instrument Co., Ltd.) in direct contact with the material surface. Similarly, the MHCOMP was introduced into the mold, and the mold was secured with glass plates to maintain proper positioning. The composite material was then polymerized by direct contact with a light-curing lamp for 20 sec. That ensured thorough and adequate polymerization of the composite [14]. All samples underwent polishing and fine instrument processing (3M Espe, Sof-lex XT, Pop-On Polishing Discs 12.7 mm) following the standard protocol for restorative fillings on teeth. This step ensured that the samples were appropriately finished and prepared for further analysis.

Following their fabrication, all the discs were placed in plastic containers containing 2 mL of distilled water [12]. The containers were then incubated at a temperature of 37 °C for two days. This step aimed to simulate the conditions found in the oral cavity, allowing for potential interactions and changes that may occur over time. On the other hand, the lack of thermocycling of restorative materials is also another caveat of the study.

After the incubation period, the samples were carefully dried and transported to the laboratory. The microhardness was determined using the Vickers method (Emco Test GmbH, Dura Scan 20 G5, Figure 3).

During the microhardness testing process, a diamond pyramid with a square base and a tip angle of 136° was used as an indenter, and a load of 100 g was applied for 15 sec. Measurements were taken at three different points on each sample within a 15 mm diameter area. The average value of these measurements was then considered as the baseline microhardness value for each sample. Roughness was measured by contact profilometry using a Mitutoyo SJ-310 (Mitutoyo Corp., Kawasaki, Japan) profilometer (Figure 4).

The measuring sensor that slides along the examined profile had a measuring probe with a diameter of 2 µm under the action of force F = 0.7 mN.

Each material was divided into two subgroups (10 samples each), of which the first subgroup was treated with fluoride varnish [Fluor (F) protector S, 7,700 ppm, 1.5%], and the second was treated with MI Varnish™ (5% sodium-fluoride, 22,600 ppm, CPP-ACP).

The same procedure for determining the microhardness and roughness of the samples after applying the varnish was repeated in the laboratory. The obtained values are presented in the form of statistical data.

Statistical processing and analysis were done in the SPSS version 24. The alpha value was established at $p < 0.05$.

Frequencies and percentages were used to describe important parameters, while the median (Me) with interquartile range (IQR) was used to describe numerical variables. Differences in two time intervals were tested by Wilcoxon Test.

Results

The microhardness of HV GIC material was statistically significantly different after MI Varnish™ application (Me = 44.55; IQR = 2.85) compared to baseline values (Me = 72.10; IQR = 5.03), $p < 0.001$, but also after F protector S application (Me = 36.15; IQR = 2.85), $p < 0.001$. A statistically significant difference compared to the baseline measurement was also recorded when it comes to the MHCOMP material after the MI Varnish™ application ($p < 0.001$) and after the F protector S application ($p < 0.001$). Differences were noted in the microhardness of the RM GIC material after the MI Varnish™ application ($p < 0.001$), as well as after the F protector S application ($p < 0.001$).

The microhardness of HV GIC, MHCOMP, and RM GIC was statistically significantly different in baseline measurement (Me = 24.75; IQR = 0.82), in comparison to

Fig. 3 – Vickers microhardness device.

Fig. 4 – Mitutoyo SJ-310 profilometer.

the measurement after MI Varnish™ (Me = 43.00; IQR = 3.35) or F protector S application (Me = 52.5; IQR = 1.93), $p < 0.001$. The results are shown in Figure 5.

The roughness of HV GIC material is statistically significantly different when comparing baseline measurement (Me = 0.26; IQR = 0.03) to the measurement performed after MI Varnish™ (Me = 1.13; IQR = 0.11) or F protector S application (Me = 0.91; IQR = 0.16), $p < 0.001$. A statistically significant difference was also noted concerning the MHCOMP material ($p < 0.001$). The roughness of this material was lowest in the baseline measurement (Me = 0.80; IQR = 0.10), statistically significantly higher after MI Varnish™ (Me = 1.23; IQR = 0.12) or F protector S application (Me = 1.22; IQR = 0.13), $p < 0.001$. Differences were noted in the roughness of the RM GIC material after MI Varnish™ (Me = 0.33; IQR = 0.08) and F protector S application (Me = 1.07; IQR = 0.16), $p < 0.001$.

The roughness of all materials was statistically significantly different in baseline measurement (Me = 0.33; IQR = 0.08) compared to measurement after MI Varnish™ application (Me = 1.24; IQR = 0.12), and after F protector S application (Me = 1.07; IQR = 0.16), $p < 0.001$. The results are shown in Figure 6.

Discussion

In this study, we observed that the addition of MI Varnish™ and fluoride varnish leads to differences in the microhardness and roughness of the restoration materials. Similar findings are found in previous studies, examining different

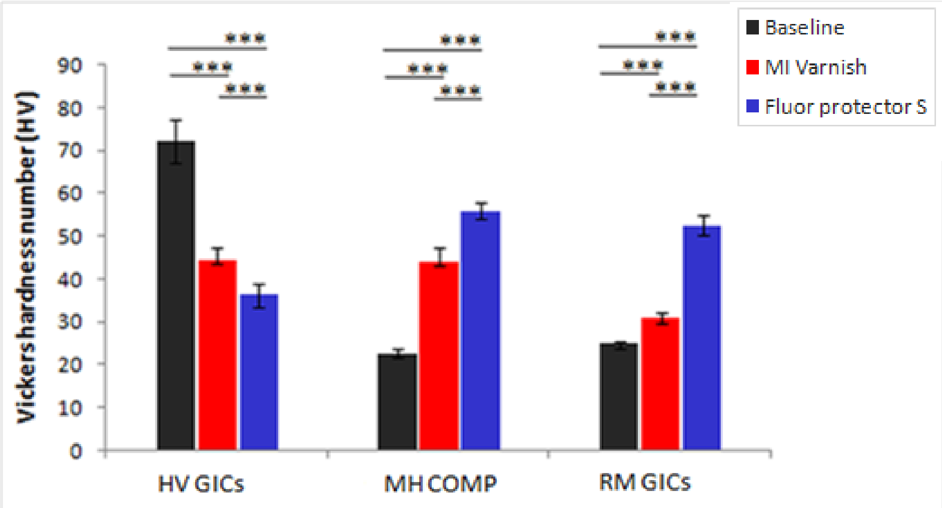

Fig. 5 – Microhardness of the high-viscosity (HV) glass ionomeric cement (GIC) Fuji IX GP®, micro-hybrid composite (MHCOMP) Te-Econom Plus® and resin-modified (RM) GIC Fuji II LC®. Baseline measurement compared to measurement after application of MI Varnish™ or Fluor (F) protector S (**p* < 0.001). Results are shown as median and interquartile range. Wilcoxon test was applied.**

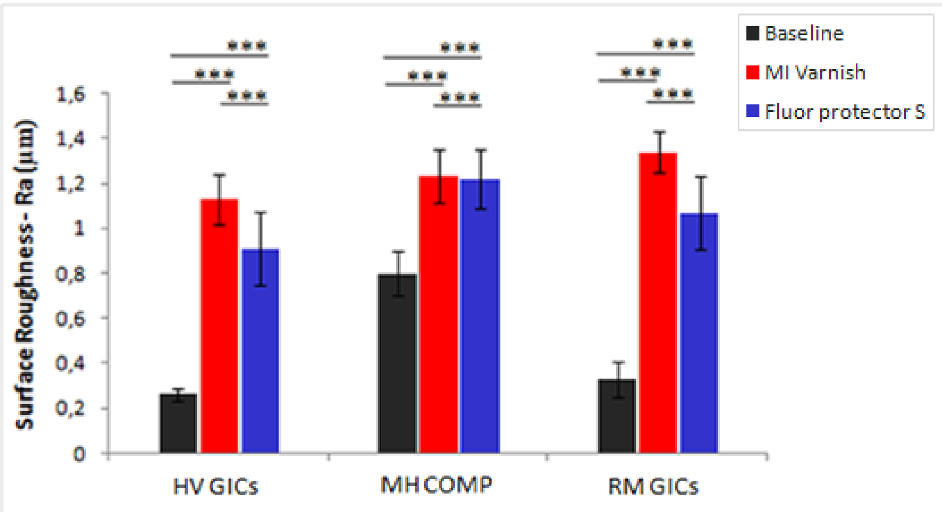

Fig. 6 – The roughness of the HV GIC Fuji IX GP®, MHCOMP Te-Econom Plus®, and RM GIC Fuji II LC®. Baseline measurement compared to measurement after application of MI Varnish™ or F protector S (**p* < 0.001). Results are shown as median and interquartile range. Wilcoxon test was applied. For abbreviations, see Figure 5.**

dental varnishes [15]. HV GICs are materials composed of fluor aluminosilicate glass and polyacrylic acid. These types of cement have a higher powder-to-liquid ratio, specifically a higher proportion of polyacrylic acid to the powder. As a result, they exhibit greater resistance compared to standard glass ionomers [16]. Both HV and RM types of GIC have a disadvantage when it comes to their dissolution in an acid-base environment [17]. This means that these types of cement may experience degradation or erosion when exposed to acidic or alkaline conditions, which can affect their longevity and effectiveness as dental restorative materials. GIC restorations are biocompatible with dental structures and act as a fluoride ion reservoir, aiding in the remineralization process. The addition of CPP-ACP varnish over glass ionomer restorations enhances their effectiveness in preventing caries. Studies have demonstrated the efficacy of applying MI Varnish™, which contains 5% sodium fluoride or CPP-ACP, to damaged enamel surfaces. This application increases the release of calcium, fluoride, and phosphate ions onto the tooth surface, promoting remineralization [18]. Fluoride protector 0.7% is a highly soluble solution that facilitates the formation of fluoride deposits, aiding in the remineralization of the tooth surface by incorporating fluoride ions into the crystal lattice [19]. In this study, we observed that the addition of MI Varnish™ and fluoride varnish led to a decrease in the microhardness of HV GIC. Similar findings regarding the reduction in microhardness of HV GIC after the application of fluoride varnishes and the addition of CPP-ACP have been reported in previous literature [12]. Potential adverse effects of dental varnish application might be material dependent [20], as in our study. Conversely, some studies have indicated that fluoride, calcium, and phosphate ions can increase the microhardness of RM GIC [21]. F protector S varnish, containing 1.5% ammonium fluoride, ethanol, and water, has a greater impact on the microhardness of RM GIC compared to MI Varnish™. After the evaporation of the alcoholic component, the concentration of fluoride on the treated surface is approximately ten times higher [22]. The formation of a fluorapatite compound, which is more resistant to dissolution than hydroxyapatite, is one possible explanation for the increase in microhardness observed in the treated material. Te-Econom Plus® composite contains bisphenol A-glycidyl methacrylate (Bis-GMA), urethane dimethacrylate, and triethylene glycol dimethacrylate (TEGDMA), which makes it an aesthetically pleasing and mechanically robust restorative material. In this study, we observed a significant increase in microhardness with the application of F protector S varnish and a slight increase with MI Varnish™. Previous literature has reported a decrease in microhardness when using pH-modifying compounds [23]. However, our study utilized different topical varnishes, which may yield varying effects on the material [24]. Some studies have shown an increase in microhardness after the application of fluoride-based varnishes [25]. Additionally, it is worth considering that the surface polishing of the composite samples involved partial coverage with paraffin used for insulation, which could contribute to the observed increase in microhardness [26]. This is a potential caveat of the study.

After treatment, the roughness of all samples showed an increase [27]. The MI Varnish™, due to its density, required physical removal from the sample. Even after drying with a duster, the remaining residue had to be carefully dried with cotton balls to ensure accurate profilometer measurements, similar to previous studies [28]. While increased roughness is an undesirable characteristic that facilitates the adhesion of food and bacteria to the material surface, the benefits of fluoride and calcium outweigh this concern, especially for patients at risk of caries [29].

The surface microhardness of restorative dental material increases after fluoride varnish application. Although an increase in the surface roughness of restorative dental material is an undesirable effect of this varnish application, the formation of fluorapatite and remineralization of demineralized enamel outweigh this risk.

The limitation of this study is that the use of paraffin as an insulator should be quantified in the following research on the microhardness of the materials. Furthermore, the lack of use of thermocycling could be a caveat, and hence direct extrapolation of these results in a clinical setting is not possible.

Conclusion

This study provides evidence that varnishes such as F protector S and MI Varnish™ can affect the microhardness and roughness of restorative materials. Despite the limitations of the study, which is reflected in the use of paraffin as an insulator, these findings highlight the importance of considering material compatibility when selecting varnishes for different types of dental restorative materials.

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