



COMPARATIVE ANALYSIS OF SMEAR LAYER REMOVAL TECHNIQUES IN THE TREATMENT OF DENTAL CARIES

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ABSTRACT

The purpose of this systematic literature analysis is to review the application features of various types of acids and to determine the most effective etching technique to achieve maximum elimination of the smear layer.

Materials and methods: the analysis of up-to-date Russian and English literature was carried out in Google Scholar and PubMed electronic databases. The analysis identified the features of various etching techniques using phosphoric, maleic and polyacrylic acids to achieve maximum elimination of the smear layer.

Results: during the analysis, 67 articles were examined. After analyzing the literature on the inclusion criteria, the total number of publications amounted 31.

Conclusion: smear layer should be removed using phosphoric or maleic acids according types of tissues: phosphoric acid application used on the enamel, maleic acid is applied on the dentine due to ensure a high-quality adhesive protocol, taking into account the sparing effect on the structural and functional units of tissues. Recommended exposure: applied to the enamel for 15-30 seconds, taking into consideration the type and the molecular weight of the acid. The exposure time of acid on dentin is recommended from 10-15 seconds, but an increase in the exposure time of maleic acid on dentin did not lead to negative changes in the structure of dentin, which significantly distinguishes the use of orthophosphoric acid. Also, static batching is not enough for high-quality treatment, because, in case of static etching, only the internal areas consisting of enamel prisms can be fully etched. Consequently, the use of dynamic etching is recommended. All authors contributed equally to the writing of the article.

KEYWORDS: etching, phosphoric acid, smear layer, dynamic etching.

INTRODUCTION

Working with composite restorative materials involves the use of acids to demineralize the surface of dental hard tissues. Such changes of surface imply the removal of the smear layer, an increase in both permeability, microporosity and chemical changes in the surface composition [Barkmeier W et al., 2009]. After preliminary treatment of the enamel, the wettability improves, the area of the outer surface increases and a micro-

retention relief is formed. Thus, an improved bond of the composite with the tooth tissues is achieved. Unfortunately, conditions are not always optimal, and organic residues, as well as the aprismatic structure of the enamel, can reduce the etching efficiency and, thus, jeopardize the adhesion of the material [Ciucchi P et al., 2015].

The effect of acid etching depends on the type of acids and their concentration, the form of acid

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application (gel, semi-gel), rinsing time, instrumental treatment of the enamel surface before etching, chemical composition and state of the enamel. Besides, the application technique is also different: enamel etching, dentine conditioning, total etching [Barkmeier WW et al., 2009].

The purpose of this systematic literature analysis is to review the application features of various types of acids and to determine the most effective etching technique to achieve maximum elimination of the smear layer.

MATERIALS AND METHODS

The research was written in the course of the analysis of Russian and English literature in Google Scholar and PubMed electronic databases.

The analysis includes publications that meet the following selection criteria:

1. Publications dated from 2001 to 2019;
2. The study examined the properties of three basic acids used to remove the smear layer in the treatment of dental caries;
3. The features of various etching techniques to achieve maximum elimination of the smear layer are described;

The publications were selected and included in the analysis in several stages. The first step was to exclude publications dated 2000 and earlier. Subsequently, the title and summary of the publications were analyzed. Further, there was an acquaintance with the content and the review of full-text versions of the selected publications. At each stage, the authors worked independently.

During the data extraction process, the risk of systematic error was evaluated using a two-component Cochrane Collaboration instrument [Higgins J et al., 2008; Higgins J et al., 2011]. According to Higgins et al. systematic error levels were categorized as follows: low risk if all criteria are met; moderate risk if only one criterion is missing; high risk if two or more criteria are missing; unclear risk if there were

few details to evaluate the risk.

RESULTS

In the course of a systematic literature analysis, 67 literature sources were identified, 13 of which were from PubMed and 54 from Google Scholar. After the analysis of the selected works according to the inclusion criteria, removal of duplicates, the total number of articles amounted 31. The studies included in the analysis identified the features of various etching techniques using phosphoric, maleic and polyacrylic acids to achieve maximum elimination of the smear layer.

DISCUSSION

The traditional etching of dental hard tissue includes the use of phosphoric acid. Compared to other acids such as citric, hydrofluoric or hydrochloric, phosphoric acid is the most effective in stimulating of the adhesion of enamel to dental materials in vitro. The optimal concentration is considered to be 35-37% [Zhu J et al., 2014].

For correct etching, the time of application of the acid exposure to the enamel is extremely important. For many years, 60 seconds of acid etching of enamel has been traditional. However, studies with a scanning electronic microscope have shown that etching of enamel for 15 seconds leads to the formation of the same porous surface as etching for 60 seconds [Nanjannawar L et al., 2012]. In addition, spending less time on acid etching can save patient time in the dental chair, reduce the possibility of surface contamination, minimize the dissolution of subsurface substance, and be more biocompatible when etching enamel and dentin at the same time [Perdigão J et al., 2006]. Still, the main reason, why the etching time dropped from 60 seconds to the current 15, is because adhesive dentistry has adopted the total-etch concept. This concept was based on etching enamel and dentine at the same time. Sixty seconds of etching was optimal for the enamel, but it demineralized the dentin too deeply. A 60-second dentin etching will demineralize the dentin to a depth of 15 μm . However, penetration of the adhesive monomer is difficult and can miss a free of adhesive zone in the lower part of the hybrid layer. When acid exposure lasts 15 seconds, the enamel is still sufficiently etched, and



To overcome it is possible, due to the uniting the knowledge and will of all doctors in the world

the dentin is demineralized only for 5-8 μm , which facilitates infiltration [Zhu J et al., 2014].

Another factor to consider while etching is the diffusion of the acid solution through the dentin surface and therefore its conditioning capacity. This factor depends on the viscosity of the acid solution and the molecular weight of the acid. In terms of viscosity, gel solutions, applied for the same time and in the same concentration as liquid solutions, have less effect. But in practice, it is better to use gel forms, because it is easier to control their application. Thus, in order to obtain the same etching pattern of the gel solution as the liquid solution, it is only necessary to use it for a longer period. In addition, the diffusion of a solution is inversely proportional to the molecular weight, for example, polyacrylic acids diffuse less than acids with a lower molecular weight, such as phosphoric and nitric [Rontani R et al., 2014].

It is also necessary to consider the generation of the applied adhesive system. Thus, the use of self-etching systems made possible the reduction of the operating time, since they simultaneously ensure the processes of demineralization and penetration into the enamel [Ciucchi P et al., 2015]. As these systems are composed of weaker acidic monomers compared to phosphoric acid, interprismatic enamel is selectively decalcified and does not improve the bond strength [Perdigão J et al., 2003]. It was found that preliminary etching of enamel with phosphoric acid increases the adhesion strength of self-etched systems. These observations indicate that eliminating the etching step of the enamel can jeopardize the optimum bond strength and durability. Fatigue tests of enamel compounds have also shown that self-etching systems do not function at the same level as traditional phosphoric acid conditioning systems [Barkmeier W et al., 2009]. The need for mechanical treatment of the enamel to ensure effective etching can be considered as a significant disadvantage of self-etching adhesive systems. Typically, the bond strength to enamel with self-etch adhesives is lower than with total-etch systems. In some cases, self-etching systems lead to an increase of staining in the edge of adhesion to the enamel, while the state of adhesion to dentin is satisfactory [Tsujimoto A et al., 2016; Oz FD et al., 2019]. Thus, in a number of clinical cases,

self-etching adhesives are more preferable than total-etch systems, when the residual enamel structures are subjected to mechanical treatment, and the surface with which the bond is formed is mainly represented by dentin. Also, these systems are preferable in those areas of the oral cavity where it is difficult to isolate the working field from oral fluids [Pisuk D, 2020].

In addition, the choice of acid should be based on dissociation constants. As a rule, acids with a lower pK_a (maleic acid $pK_a = 1.8$) are used in a more dilute solution than acids with a higher pK_a (phosphoric acid $pK_a = 2.1$) [Di Hipólito V et al., 2005]. Tooth tissue is etched more often with 37% phosphoric acid. When it is used, a more remarkable and retaining pattern is found on the enamel surface [Perdigão J et al., 2003]. If the acid concentration is less than 30%, then hardly soluble brushite is formed on the tooth enamel. This compound is poorly removable with a water jet and prevents the composite from adhering to the enamel of the tooth. If the acid concentration exceeds 40%, then there is a rapid precipitation of calcium phosphate compounds, which prevent the conditioning of the enamel. However, even when etching is carried out with 30-40% phosphoric acid, calcium phosphate is formed on the enamel, which must then be carefully removed by rinsing [Di Hipólito V et al., 2005].

Residual acids can also interfere with the bonding of the adhesive to the dental hard matter. Therefore, after etching the enamel, they must be carefully removed [Barkmeier W et al., 2009]. According to Perdigão J. and co-authors, the surface residue is partly silica used to thicken the gel and is not completely removed by irrigation with water [Di Hipólito V et al., 2005].

After etching, due to the different degrees of solubility of enamel prisms and interprismatic substance, a micro-retention relief is formed, characterized by a different pattern. If the enamel prisms dissolve, type I of etching is obtained. If the periphery (interprismatic substance) is etched with acid, then type II etching is formed. If enamel prisms and interprismatic substances are etched in this way, then mixed type III is formed. Types I and II are more preferable for holding adhesives on the enamel surface by micromechanical retention. Type III pattern is less conducive to the ad-

hesion of the material to the enamel surface. The subsequent development of the acid etching technique was based on the ideas of maximizing type I and / or type II by optimizing the concentration of the solution and the duration of exposure [Zhu J et al., 2014].

When etching, the enamel layer with a thickness of about 10 μm is irretrievably lost. Histological changes (pits, cracks) can reach a depth of 30-50 μm [Durmus B et al., 2017]. As a result of conditioning, as mentioned above, the total surface increases, the enamel structure's reaction activity increases and the wettability improves (up to 400%). If such enamel is moistened with a low-viscosity composite material or a bond, the latter penetrates into the spongy structures and micromechanically adheres to the tooth enamel due to the rheological and geometric effect. After polymerization, typical layers are formed, which are discernible under a microscope on a cut of the cavity edge [Sheets J et al., 2012].

Compared to enamel, dentin is a more complex bonding substrate due to its complex and variable histological features. Physiological and pathological changes after etching can further lead to the formation of sclerotic dentin, which is less susceptible to adhesion protocols designed for use on healthy dentin [Kwong S et al., 2002].

It should be remembered that in some cases dentine conditioning is also necessary. Buonocore was the first to apply acid to the dentin surface in an attempt to improve the adhesion of restorative materials using 7% perchloric acid for 1 minute. He did not achieve good results with this technique, but it is important to note that at that time the available aesthetic restorative materials were mainly acrylic resin and silicate cement, quite different from modern ones. Only a few years later, when Bowen developed the bisphenol A-glycidyl methacrylate (Bis-GMA) molecule, research on dentine conditioning was recommenced. This research was dedicated to the development of an effective dentin bonding agent. The most common classification of dentine bonding agents is divided into generations. Thus, the fourth generation is used to etch both enamel and dentin at the same time, this method is called "total etching" followed by the application of a hydrophilic primer and, as a final step, by the application of a bonding adhe-

sive. The result is a hybrid layer formed due to the final adhesion of collagen fibers and a bonding agent - adhesive. It is important to make this layer continuous in order to prevent possible micro-cracks [Rontani R et al., 2004].

However, dentin cannot be treated in the same way as enamel. The use of 37% phosphoric acid in some cases can be the cause of excessive demineralization, avoiding proper infiltration and adhesive impregnation in collagen fibers, thus, there is a threat of disconnection between dissimilar materials [Perdigão J et al., 2003].

But there is a need for etching dentin, which is associated with the presence of a smear layer that forms on the surface of dentin during the preparation of tooth tissues and consists of hydroxyapatite particles, odontoblast processes, collagen fibers and microbial cells. When etching dentin, the acid gel acts on the smear layer, dissolving it and ensuring the penetration of the adhesive system deep into the dentin with the formation of a hybrid zone. This zone is formed when the adhesive system penetrates into the dentinal tubules and intercollagen spaces [Puppin-Rontani J et al., 2017].

After conditioning the dentin, the tissue becomes poor in minerals and rich in proteins, and also exposes collagen and increases fluid flow and permeability. Some purposes of acid etching are to facilitate bonding to the underlying dentinal matrix, demineralize the dentinal matrix to allow adhesive infiltration, expose inter- and peritubular dentin, and clean the dentin surface [Rontani R et al., 2004].

With all this, hydroxyapatite crystals dissolve with acid, and dentin turns into a structure consisting of intertwining collagen fibers. These fibers are deprived of their inorganic support and therefore the etched dentin must be handled with particular care. A strong air jet from an air spray develops a collapse - disorientation of collagen fibers deprived of inorganic support. The longer and stronger the impact of the jet, the more notable the collagen fibers collapse [Sai K et al., 2018]. To avoid collapse, if possible, direct the jet onto the enamel. In this case, reflected jets fall on the dentin, which does not cause a collapse of collagen fibers. It is very important that after etching the dentin retains its "lacy structure" as much as possible. This structure allows the primer of the adhe-

sive system to penetrate deep into the dentin within 30 seconds and form a hybrid zone there. The hybrid zone is very important to achieve good adhesion. In the case of the development of a strong collapse of collagen fibers, the formation of a hybrid zone is difficult [Ram D et al., 2005].

Gwinnett and Kanca reported that conditioning of dentin with 37% phosphoric acid for 15 seconds followed by a unique combination of hydrophilic primers and an ambiphilic bonding agent leads to a gap-free material-tissue interface in vivo and in vitro [Yazici A et al., 2009].

The latest generation of adhesive systems use different types of acids to etch enamel and dentin at the same time. Among them, there are 10% and 35% solutions of phosphoric acid and 10% maleic acid. Acid application time is reduced to 15-30 seconds. Application of these conditioning agents to the dentin surface leads to partial or complete removal of the smear layer, opens the dentinal tubules and causes demineralization of peritubular and intertubular dentin. Thus, the microporosity of the intertubular dentin and its strength increase and are within the normal range recorded for enamel [Di Hipólito V et al., 2005].

Even the research shows that acid etching improves the penetration of universal adhesives, it is worth to notice that organic matrix exposure can occur in dentin. Thus, it becomes more difficult to form the 10-methacryloyloxydecyl dihydrogen phosphate (MDP) - Ca salt, which provides the chemical interaction of the nanomaterial. This chemical interaction in dentin is facilitated by its partial demineralization, which preserves the integrity of collagen fibers, which chemically interact with 10-methacryloyloxydecyl dihydrogen phosphate (MDP) - Ca, forming a stable layer [Perdigão J et al., 2003].

It is important that after etching and washing off the acid, the dentin is not overdried, because this leads to purely mechanical damage to the collagen fibers that are left without support. But you cannot work with too wet dentin, excess water dissolves the primer and reduces the effectiveness of its effect. You need to work with slightly moistened dentin, the so-called "sparkling" (containing randomly scattered water molecules), with which adhesive systems that have acetone in their composition are well connected, which per-

fectly penetrates the moistened collagen fibers and leads to a penetration deep into the primer [Yazici A et al., 2009].

The total-etch technique involves the simultaneous application of an acidic gel to enamel and dentin. Acid is applied to the tooth tissue, starting with the enamel. The time is counted after the end of the application of acid to the enamel. The main requirement: the enamel is etched for at least 15 seconds, dentin - no more than 15. Then the acid is removed with a stream of water for 20 - 30 seconds, and then the cavity is dried with a booster for 10 seconds. In this case, the enamel is completely dry, and the dentin is not overdried. The quality of the etching of tooth tissues is determined by different criteria. Firstly, the colored acid gel is completely washed off with water, and secondly, after drying, the enamel surface becomes chalky, and the dentin shines. To solve this problem, materials have been proposed to moisturize and fix the network of collagen fibers in dentin, thanks to an aqueous solution of 2-hydroxyethyl methacrylate (HEMA) and stabilizers (Aqua-Prep, Bisco; Gluma Desensitizer, Gluma; Creafil SA Primer, Kuraray; Tubulicid Red, Global Dental Products) [Cadenaro M et al., 2019].

When using adhesive systems for total etching, there is a problem of controlling the depth of dentin demineralization, which is compatible with the diffusion depth of hydrophilic primers. If the primers are diffused into all of the demineralized dentin, the bonding agent can penetrate the entire depth of the dentin, creating an effective hybrid layer. Initially, it was believed that the thicker the hybrid layer and the longer the resin marks, the better adhesion is achieved. However, it is now believed that there is no significant difference in the strength of adhesion from the length of these marks, the main role belongs to the continuity of the hybrid layer. But it is still controversial whether the adhesive systems penetrate the total depth of dentin demineralization [Rontani R et al., 2004].

In addition, difficulties in performing the total-etch technique may arise during the stage of rinsing and drying the dentin. According to many clinicians, it is difficult to determine whether a surface is sufficiently wet before the adhesive is applied. In medicine, there is even such a thing as "sparkling dentin". But, as practice shows, every

dentist puts his own meaning in this concept. Excessive or insufficient wetting leads to a decrease in bond strength.

Therefore, it can be said that the inclusion of an etching in the functionality of the adhesive may be justified. This makes it possible to remove the difficult-to-control step of sequential etching of enamel and dentin from the technological chain. Thus, self-etching composite adhesives are the treatment of choice. They provide less procedure time, a decrease in the patient's stress level, and a decrease in the amount of mental and physical work of the dentist. However, since the acidity of self-etching adhesives is less high, the removal of the smear layer is often incomplete; therefore, dentinal tubules may not be as open as in the case of total-etch systems [Pisuk D, 2020].

There is also a number of features in the treatment of non-carious lesions of hard dental tissues, since these tissues are characterized by sclerotic changes. It significantly interferes with the penetration of the adhesive into the dentin layer. In such a situation, total etching is the method of choice. Based on encouraging clinical results of the use of the total-etch technique for non-carious defects, it is suggested that the bonding of primers from self-etch systems to sclerotic dentin can also be enhanced with the additional use of phosphoric acid for preconditioning. Although sclerotic dentin has no smear layer, it has been shown that self-etching primers cannot sufficiently etch the surface hypermineralized layer in sclerotic dentin. Interaction with sclerotic dentin can be improved by modifying the protocols commonly used for healthy dentin. These include the removal of the surface hypermineralized layer or the use of stronger acids [Kwong S et al., 2002; Karakaya S et al., 2008].

Maleic acid can also be used as the etching agent. Etching with 10% maleic acid gel for 15 seconds partially removes hydroxyapatite crystals from the prism core, and with a delay of up to 60 seconds, greater dissolution of the prism core material develops. Despite the absence of differences in the type of etching pattern obtained on enamel with any exposure, after 60 seconds of etching, the topographic view of the structure of the prism core approached to the one obtained with 35% phosphoric acid gel. This is because maleic acid is or-

ganic and has a higher molecular weight, which requires more time for the acid reaction on the dentin surface [Di Hipólito V et al., 2005].

Maleic acid is able to demineralize the surface of hard tissues, but its effect is directly related to the concentration of protons. This organic acid forms a complex fibril network of dentin (in particular, on the intertubular and peritubular areas), which is very interesting when considering the formation of a hybrid layer after application and curing of the adhesive system. When tissues are treated with maleic acid, two different layers are formed: the first is the surface layer of compacted collagen fibers and the second is a thick layer of well-separated fibrils, which forms a very porous structure of the collagen net.

The large number of fibrils and the porosity created by etching (in particular, after treatment with maleic acid at 0.7 pH) showed possible ways of penetration of the adhesive agent into the decalcified surface of human dentin. In particular, considering the development of self-etching adhesive systems that involve the use of weak organic acids to demineralize the dentin surface, maleic acid at pH 0.7 may be a reasonably effective agent to demineralize the dentin surface. The predominant dissolution of the hypermineralized peritubular dentinal matrix with maleic acid and, thus, the dilatation of the tubule can be explained by the ability of the acid to etch peritubular dentin from the inside of the tubular lumen more than through the demineralized side of dentin.

Samples etched with maleic acid at a lower proton concentration (i.e. pH 1.4) show a small amount of residual particles on the annular dentin. Observations of maleic acid at pH 0.7 showed perfectly clear and porous intertubular dentin with no smear residue even at high increasing. This suggests that the ability of maleic acid to clean the surface of the dentin from the smear layer and to demineralize the intertubular dentin is related to the pH of the acid used [Breschi L et al., 2002].

Thus, maleic acid is able to remove the smear layer, but there is no plug formed in the dentinal tubules. Also, this organic acid in most cases does not demineralize dentin in its depth. When using maleic acid with an exposure of 15 seconds (the standard time for etching with phosphoric acid), it can only provide a tissue change of about 1 mm,

which is not enough for good adhesion. Therefore, it is necessary to increase the time of its application, as well as to use the dynamic etching technique [Rontani R et al., 2004].

Since Buonocore first introduced the use of phosphoric acid for etching enamel, various alternative acids and chelating agents have been studied, including polyacrylic acid. Summitt reported several advantages of using polyacrylic acid over conventional phosphoric acid etching: the enamel surface is not significantly damaged, the smear layer is easier to remove, and there is minimal loss of fluoride-rich outer enamel [Raggio D et al., 2010]. However, polyacrylic acid does not remove the entire structure of the smear layer, the plugs in the smear layer remain intact. A number of studies show the low efficiency of polyacrylic acid when etching through the relatively denser structures of the smear layer [Kharouf N et al., 2019].

Etching with 20% sulfated polyacrylic acid can be carried out with an exposure time of 30 or 60 seconds. Crystal growth is observed after 30 seconds. These crystals, apparently, form a sufficient area for the penetration of the adhesive, forming a retaining retention net [Sena LMF et al., 2018]. Samples etched with 20% sulfated polyacrylic acid for 60 seconds show cracking and pitting. In general, sulfated polyacrylic acid removes significantly less healthy enamel, which is good for the concept of maximizing dental hard tissue preservation. But at the same time, it provides a lower strength of the bond compared to 37% phosphoric acid [Devanna R, Keluskar K., 2008; Nogawa H et al., 2015].

When working with acids, dynamic etching can be used. This is an active and vigorous rubbing of acid into the tooth tissue. A simple application of acid to the enamel (the so-called static etching of the enamel) is not enough for high-quality etching. This type of etching provides good etching of only the inner areas consisting of enamel prisms. In this case, the etching of aprismatic areas of the enamel is irregular. As a result, islets of non-etched enamel remain on the enamel surface, with which the adhesive does not interact. It leads to the formation of micro-spaces, the appearance of a white line, and edge staining of the restoration. This problem is most relevant for aesthetic filling and critical when etching unprepared enamel: in such a situa-

tion, these "islets" make up most of the bonding surface [Naidyonova O, 2020].

It has been proven that statistically higher bond strength is achieved with the dynamic application of an acidic agent. The etching and monomer penetration rates are greater with dynamic conditioning than with static conditioning. This increased bond strength can apparently be attributed to the higher degree of etching. This is not surprising, since mixing and replenishing the active ingredient ensures that the enamel surface is exposed to fresh acid and etching by-products are removed more efficiently [Glasspoole E et al., 2001].

Based on the above, preference should be given to dynamic etching of enamel, which implies constant rubbing of the etching gel into the enamel surface with a brush or applicator. With this etching technique, an even micro-roughness of its surface is achieved without reference to the initial structure of the enamel. In addition, it gives the best adhesion results in the immediate or long term [Barkmeier W et al., 2009].

CONCLUSION

The stage of etching of dental hard tissues is an important element in the treatment of dental caries, since it largely determines the subsequent adhesion of dissimilar materials - tooth tissue and composite filling material. smear layer should be removed using phosphoric or maleic acids according types of tissues: phosphoric acid application used on the enamel, maleic acid is applied on the dentine due to ensure a high-quality adhesive protocol, taking into account the sparing effect on the structural and functional units of tissues. Recommended exposure: applied to the enamel for 15-30 seconds, taking into consideration the type and the molecular weight of the acid. The exposure time of acid on dentin is recommended from 10-15 seconds, but an increase in the exposure time of maleic acid on dentin did not lead to negative changes in the structure of dentin, which significantly distinguishes the use of orthophosphoric acid. Also, static batching is not enough for high-quality treatment, because, in case of static etching, only the internal areas consisting of enamel prisms can be fully etched. Consequently, the use of dynamic etching is recommended. All authors contributed equally to the writing of the article.

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CONTENTS

4. **ZILFYAN A.V., AVAGYAN S.A., MURADYAN A.A., BARSEGHYAN E.S.**
RECOMMENDED TACTICS FOR MASS VACCINATION OF HEALTHY INDIVIDUALS AND COVID-19 CONVALESCENTS
13. **MAGHAKYAN S.A., AGHAJANOVA E.M., HOVHANNISYAN A.H., ASOYAN V.A., BARSEGHYAN E.S.**
MYXEDEMA COMA ASSOCIATED WITH COVID-19 INFECTION: CASE REPORT
17. **HAKOBYAN H.H.**
ANXIETY AND CHRONIC PAIN IN CAREGIVERS OF CHILDREN WITH CEREBRAL PALSY IN ARMENIA: DESCRIPTIVE STUDY
23. **ISSAMATOV B.K., ZHOLDYBAY ZH.ZH., TAJIBAEV T.K., SERIKULY E.S., BAIMAKHANOV B.B., MEDEUBEKOV U.SH., SAGATOV I.Y.**
FEATURE ANALYSIS OF COMPUTED TOMOGRAPHIC SIGNS OF HEPATOCELLULAR CARCINOMA IN MULTIPHASE STUDIES
29. **TAJIBAYEV T.K., CHORMANOV A.T., MATKERIMOV A.ZH., TERGEUSSIZOV A.S., BAUBEKOV A.A., ZHAKUBAYEV M.A., SAGATOV I.Y., KANCHI M.**
CAROTID BODY TUMORS: CASE SERIES OF EXTREMELY RARE HEAD AND NECK PARAGANGLIOMAS.
35. **KHANCHI MEAD, MATKERIMOV A.ZH., TERGEUSSIZOV A.S., DEMEUOV T.N., ZHAKUBAYEV M.A., KHANCHI M.M., SHAMSHIEV A.S., SAGATOV I.Y.**
SURGICAL TREATMENT OF ANEURYSMS OF AORTIC ARCH BRANCHES AND VESSELS OF THE UPPER EXTREMITIES
43. **SARKISYAN N.G., KATAEVA N.N., AKHMETOVA A.I., KUKHAREVA A.R., CHUMAKOV N.S., KHLYSTOVA K.A., MELIKYAN S.G.**
PHYSICO-CHEMICAL INDICATORS OF DENTAL PATIENT SALIVA WHO HAVE UNDERGONE AN UNCOMPLICATED CORONAVIRUS INFECTION
49. **KHABADZE Z.S., NEGORELOVA YA.A., GEVORKYAN A.A., NAZAROVA D.A., SHILYAEVA E.S., KOTELNIKOVA A.P., MORDANOV O.S.**
COMPARATIVE ANALYSIS OF SMEAR LAYER REMOVAL TECHNIQUES IN THE TREATMENT OF DENTAL CARIES
58. **KULIKOVA A.A., KHABADZE Z.S., GENERALOVA YU.A., MOKHAMED EL-KHALAF R., NAZAROVA D.A., YOLLYBAYEV YA.A.**
APPLICATION OF POLYHEXANIDE AS A NEW HIGHLY EFFECTIVE ANTISEPTIC COMPOSITION.
64. **MORDANOV O.S., KHABADZE Z.S., NAZAROVA D.A., SHILYAEVA E.S., KOTELNIKOVA A.P., MORDANOVA A.V.**
TEMPERATURE EFFECT ON THERMAL CHANGES AND PHASE ANALYSIS OF 3Y-TZP ZIRCONIA RESTORATIONS
70. **BASSEL J.A., EYAD M.S.**
EVALUATION OF MARGINAL ADAPTATION OF (CAD/CAM) LAVA PLUS HIGH TRANSLUCENT ZIRCONIA AND (CAD/CAM) IPS-EMAX FULL CROWNS
76. **KHABADZE Z.S., NAZAROVA D.A., SULEIMANOVA Z.M., GENERALOVA YU.A., KOTELNIKOVA A.P.**
MICROBIAL BIOGENOSIS OF APICAL PERIODONTITIS IN THE ROOT CANAL SYSTEM (PART 1)
81. **KHABADZE Z.S., NAZAROVA D.A., SULEIMANOVA Z.M., GENERALOVA YU.A., KOTELNIKOVA A.P.**
MICROBIAL BIOGENOSIS OF APICAL PERIODONTITIS IN THE ROOT CANAL SYSTEM. (PART 2)
87. **TIUNOVA N.V., NABEREZHNOVA S.S., SAPERKIN N.V., VDovina L.V., DAUROVA F.JU., TOMAEVA D.I., CHUVARKOVA I. M.**
RATIONALE BEHIND A MINIMALLY INVASIVE APPROACH IN THE TREATMENT OF DENTAL FLUOROSIS
94. **TSVETKOVA M.A., SOHOV S.T.**
ORTHODONTIC TREATMENT ALGORITHM FOR PATIENTS WITH POSITIVE DRUG ANAMNESIS. GLUCOCORTICOID.
101. **PANAHI S.R., SABZ G., JOKARTANGKARAMI A., AFROUGHI S., KARIMPOUR F.**
ANATOMICAL CHARACTERISTICS OF NASOPALATINE CANAL USING CONE BEAM COMPUTED TOMOGRAPHY IMAGES
109. **PURWANTI T., ERFAN KUSUMA M.S., YUDIANTO A.**
HEAD INJURY CASE WITH BLUNT FORCE TRAUMATIC: CASE REPORT AT BHAYANGKARA HOSPITAL KEDRII INDONESIA



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