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## ORIGINAL ARTICLE

## COMPARATIVE ANALYSIS OF THE EFFECT OF ADHESIVE SYSTEMS ON THE MICROELEMENT COMPOSITION OF THE DENTIN OF THE CROWN PART OF THE TOOTH

Galina Chistyakova\*

Associate Professor Belarusian State Medical University, Department of Dental Propaedeutics and Materials Science, Minsk, Belarus

\* Corresponding author: Galina Chistyakova, Belarusian State Medical University Minsk, Belarus;  
e-mail: [galinach2018@mail.ru](mailto:galinach2018@mail.ru)

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## Abstract

**Title:** Comparative analysis of the effect of adhesive systems on the microelement composition of the dentin of the crown part of the tooth.

**Purpose:** To research the microelement composition of the «filling-tooth» interactive zone, the morphological characteristics of its border, to assess the effect of adhesive systems of the V and VII (self-etching) generations on the ion-exchange composition of the dentin of the crown part of the tooth, and to qualitatively assess the microelement composition of the hybrid dentin zone.

**Materials and methods:** Extracted teeth with preserved crown part (n=20), enamel etching gel (Vladmiva), V generation Singlebond - 2 adhesive system (3M ESPE), self-etching adhesive system Prime-Bond Universal (Dentsply Sirona), light-cured composite material Migrofil (Republic of Belarus). The research of the microstructure of samples with formed and sealed Class I cavities according to Black was carried out on a certified high-resolution scanning electron microscope «Mira» by Tescan (Czech Republic). The research of the microelement composition of the «filling-tooth» border was carried out by X-ray spectral analysis, which consisted of 2 blocks: using the adhesive system of the fifth generation and the seventh generation of the adhesive system.

**Conclusions:** 1. A comparative analysis of the results of using the fifth-generation adhesive system and the self-etching adhesive system showed that when using the total etching technique, trace elements Ca and P migrate from the tooth dentin structure into the hybrid zone. When using the adhesive system VII, the migration of elements was not established, due to the lack of demineralization of the dentin structure and the modification of the smeared layer. 2. Qualitative assessment of planar spectrograms revealed an increase in the carbon element in the modified adhesive layer. which is due to the presence of a smeared layer after odontopreparation of hard tissues of teeth destroyed by a carious process

3. The migration of the Si element indicates the chemical interaction of the adhesive and the filling material, as well as the filling material and the dentin of the tooth through the adhesive layer.

4. The use of the adhesive system of the fifth generation provides a higher quality of the marginal fit of the filling material to the surface of the dentin than when using a self-etching adhesive system.

**Keywords:** adhesive system, trace element composition, hybrid zone.

## Introduction

In modern restorative dentistry, adhesive systems are used, designed not only to ensure better adhesion of the filling (micromechanical adhesion), but also to migrate useful microelements into the hard tissues of the tooth (chemical adhesion) and vice versa.<sup>2,3</sup>

After preparation of the carious cavity, a so-called smear layer is formed on the surface of the dentin, which forms a film 2–10 µm thick on the surface of the dentin, closing the intra- and peritubular spaces of the dentin, thereby preventing the penetration of the components of the adhesive system into its structures. In the early 90s. XX century in dental practice, the sixth and seventh generations of adhesive systems appeared, represented by self-etching systems. Their development for use was determined by the desire of manufacturers to reduce the number of stages of working with adhesive systems and, thereby, reduce the number of dentist errors when working with adhesive systems. The development of the strategy of self-etching of dental tissues was due to the high sensitivity to violations of adhesive preparation when using adhesive systems of the IV and V generations and, as a consequence, the frequent occurrence of postoperative sensitivity. In addition, the use of self-etching systems reduced the number of work steps and, accordingly, reduced the number of errors at the stages of working with the adhesive system.

The advantages of self-etching systems are a shorter adhesive preparation time, a very simple operating procedure, and a low risk of postoperative sensitivity. However, the absence of the stage of etching and removal of the smear layer from the surface of the dentin of the tooth after preparation of the carious cavity doubt remains full. The disadvantages of using self-etching adhesive systems were considered to be large differences in adhesion to enamel and dentin. These indicators, presented by manufacturing companies as higher values than those of the IV and V generations, as well as the low efficiency of etching of intact enamel and sclerotic dentin, the high hydrophilicity and acidity of the components did not always lead to good final results. In connection with the above, when using self-etching systems, a selective etching technique is used.<sup>1</sup> At present, the degree of influence of various adhesive

systems on the ion-exchange composition of dentin of the tooth crown has not been fully revealed.

Thus, studies of the effect of adhesives not only on the structural characteristics of the tooth (components of the hybrid zone), but also on the metabolism of its tissues, in particular, on the mineral component, and the possibility of penetration of tooth microelements into the bonding system, remain relevant.<sup>3</sup>

This research is aimed at studying the replacement therapy of the tooth crown in the context of establishing the microelement composition of the dentin of the crown of the tooth after restoration using adhesive systems of the V and VII (self-etching) generations in vitro.

## Purpose

The purpose of the work was to research the microelement composition of the interactive «filling-tooth» zone, the morphological characteristics of its border, to assess the influence of adhesive systems of the V and VII (self-etching) generations on the ion-exchange composition of dentin in the coronal part of the tooth and to qualitatively assess the microelement composition of the hybrid dentin zone.

## Materials and methods

Research materials: extracted teeth with the crown part preserved (n=20), gel for etching enamel (Vladmi-va), fifth generation adhesive system Singlebond - 2 (3M ESPE), self-etching adhesive system Prime-Bond Universal (Dentsply Sirona), light-curing composite material Migrofil (Republic of Belarus). The experiment used teeth removed for medical reasons. The score of tested samples for the research is divided into two groups: group 1 (n=10) and group 2 (n=10) - experimental with cavities of Black class I formed, depending on the type of adhesive system used. In the first group, total etching and a fifth-generation adhesive system were used to fill cavities; in the second experimental group, a seventh generation (self-etching) adhesive system was used. After filling, the teeth were placed in a thermostat for 72 hours at a temperature of 37°C and 100% humidity. To obtain samples, longitudinal and transverse cuts of the tooth crown were made.

The microstructure of the samples was studied using a certified high-resolution scanning electron microscope «Mira» from Tescan (Czech Republic) with a field emission cathode. To research in a scanning electron microscope, the surface of polished sections of tooth samples was sprayed with conductive cathode chromium under vacuum conditions for 10 s. The research of the elemental composition was carried out using a micro-X-ray spectral analyzer from Oxford Instruments (England).

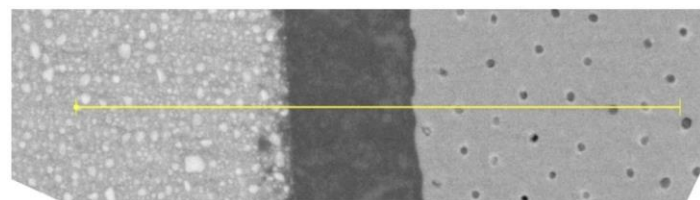
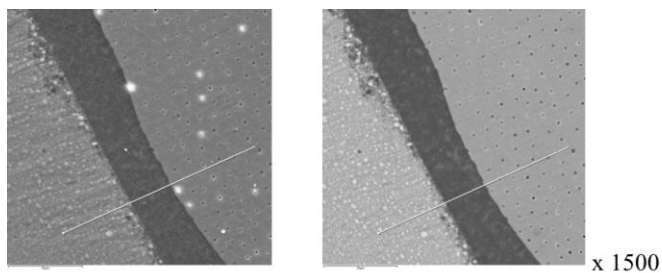
Sections of tooth samples were placed in a scanning electron microscope camera for X-ray spectral analysis at various magnifications.

The scanning areas were the following zones: «composite material - adhesive layer - hybrid zone - dentin».

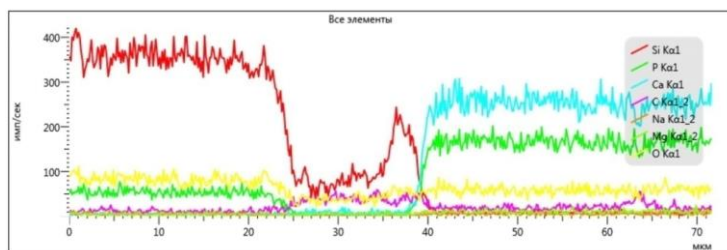
The research of the microelement composition of the «filling-tooth» interface was carried out using X-ray spectral analysis, which consisted of 2 blocks:

### 1-block

**1-block: Micro-X-ray spectral analysis of the «filling-tooth» boundary when using the fifth generation of adhesive system.**



**Figure 1.** Scanning area within which MRSA was performed (1 block of the research - when using the V generation adhesive system)



**Figure 2.** Planar spectrogram 1 - Concentration curves of distribution of elements (1 block of research - when using adhesive system of the fifth-generation)

The figure 1 shows the scanning area of the test sample of block 1.

Based on the sounding performed, planar spectrogram 1 was obtained (Figure 2), which reflects the microelement composition in the scanning area.

Interpretation of planar spectrogram 1.

On the Oy axis: changes in the intensity value of the secondary characteristic radiation excited by a directed electron beam within the designated scanning line. The line thickness is about 1 μm. On the Ox axis: distance and total length of the scan line in 1 μm.

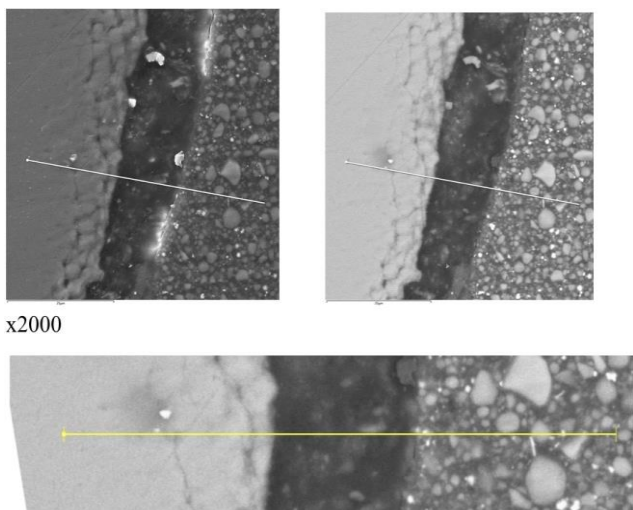
### Research Results of the 1-block

- 1) Concentration curves of the following elements were obtained: Si, P, Ca, C, Na, Mg. Migration is determined only for Si, P, Ca.
- 2) Qualitative analysis of planar spectrograms in filling areas using V generation adhesive systems revealed accumulation of P and especially Ca in the hybrid zone. The increased concentration of Ca in the hybrid zone is primarily due to decalcification of dentin after exposure to 37% orthophosphoric acid. At the border of the adhesive layer and the filling material, a high concentration of trace elements calcium and phosphorus was also established, but trace elements such as Si, P, Ca, C, Na, Mg were also identified. Interactive zones, where a dense contact zone is established, confirms the presence of chemical adhesion, which is determined by the mutual penetration of elements from the adhesive system into the structure of the material, and vice versa. The chemical interaction of the adhesive and the filling material, as well as the filling

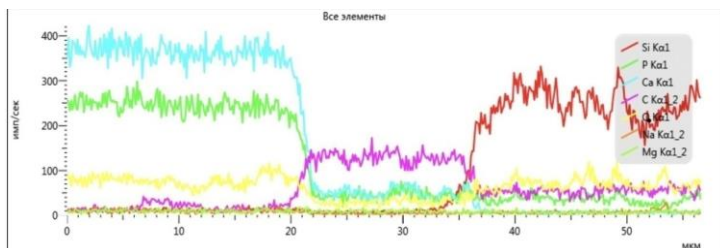
material and tooth dentin through the adhesive layer is evidenced by the migration of the Si element (Figure 1).

**2-block**

**2-block: Micro-X-ray spectral analysis of the «filling-tooth» interface using a self-etching adhesive system.**



**Figure 3.** Scanning area within which MRSA x2000 was performed (2 block of the research - when using a self-etching adhesive system)



**Figure 4.** Planar spectrogram 2. Concentration curves of distribution of elements (2 block of research - when using a self-etching adhesive system)

The interpretation of Figure 3 and planar spectrogram 2 (Figure 4) is similar to the interpretation of Figure 1 and planar spectrogram of block 1.

**Research Results of the 2-block**

- 1) Concentration curves of the elements Si, P, Ca, C, Na, Mg were obtained. Traces of microelements Si, P, Ca are determined in the modified zone.
- 2) A qualitative analysis of spectrogram 2 revealed the absence of Ca and P in the hybrid zone in a comparative analysis with spectrogram 1 when using the fifth-generation adhesive system, which is due to the non-removal of the smear layer, and the modification of the smear layer when using a self-etching adhesive system.

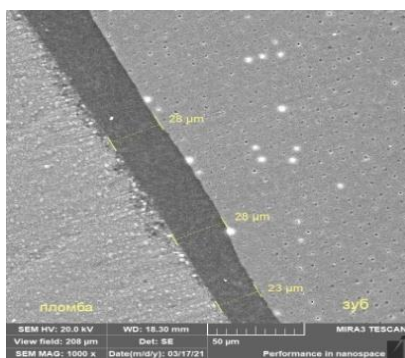
**Discussion**

In this research, it is not possible to assess the migration of microelements, as well as the depth of their penetration, but it allows us to give a qualitative assessment of the migration of microelements from the adhesive system to dentin and in the opposite direction.

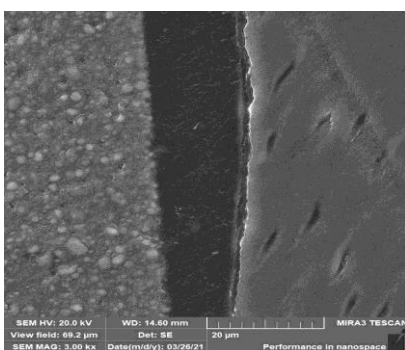
Qualitative analysis of the spectrogram revealed the absence of Ca and P in the hybrid zone, which is due to the presence of a modified smear layer when using a self-etching adhesive system.

Figures 5 and 6 show sections of teeth showing the «filling-tooth» boundary when using the fifth-generation adhesive system (Figure 5) and a self-etching adhesive system (Fig. 6). A qualitatively tighter fit of the filling material and the adhesive system was established in samples using the V generation of the adhesive system (Figure 5). When analyzing samples from the second block of research (using a self-etching adhesive system), areas of impaired contact of the modified adhesive layer with the surface of the dentin of the tooth were identified, as well as single areas of impaired contact of the modified adhesive layer with the filling material (Figures 3, 6). During the analysis of planar spectrograms, an increase in element C in the zone of modified adhesive layer, which is due to the presence of a smear layer after odontopreparation.





**Figure 5.** Section of the «filling-tooth» border when using the adhesive system of the V generation



**Figure 6.** Section of the «filling-tooth» border when using the adhesive system of the VII generation

Thus, during the research was established that the sealing density of the filling to the dentin when using a self-etching adhesive system is lower than when using a V generation adhesive system, which is due to the modification of the smear layer on the dentin surface after odontopreparation.

When conducting comparative analysis of the results, it was found that only when using the fifth-generation adhesive system does migration of trace elements Ca and P occur from the structure of tooth dentin into its hybrid zone, as well as migration of the element Si from the filling material through the adhesive layer into tooth dentin. The data obtained are combined with previously conducted studies in the mode of total etching of hard dental tissues.<sup>3</sup>

## Conclusions

Based on the results of the research, we formulated the following conclusions:

1. A comparative analysis of the results of using the fifth-generation adhesive system and a self-

etching adhesive system showed that when using the total etching technique, trace elements Ca and P mi-grate from the tooth dentin structure to the hybrid zone.

When using the VII generation adhesive system, no migration of elements was detected due to the lack of demineralization of the dentin structure and the formation of a modified smear layer.

2. A qualitative assessment of planar spectrograms revealed an increase in the carbon element in the modified adhesive layer. which is due to the presence of a smear layer after odontopreparation of hard dental tissues destroyed by the carious process
3. Migration of the Si element indicates the chemical interaction of the adhesive and the filling material, as well as the filling material and tooth dentin through the adhesive layer.
4. The use of a V generation adhesive system ensures a higher quality of marginal seal of the filling material to the dentin surface than when using a self-etching adhesive system.

## Declarations

### **Conflict of interest and financial disclosure**

The author declares that he has no conflict of interest and there was no external source of funding for the present study. None of the authors have any relevant financial relationship(s) with a commercial interest.

### **Ethical approval**

Research protocol was approved by the local Ethical Committee (2018/23) and in accordance with those of the World Medical Association and the Helsinki Declaration.

### **Informed consent**

Informed consent was obtained from all individual participants included in the study.

### **Source of Funding**

Non funding.

### **Availability of Data and Materials**

Not applicable.

REFERENCES

1. Hramchenko SN, Kazeko LA, Goreglyad AA. Sovremennye adgezivnye sistemy. *Uchebno-metodicheskoe posobie. Minsk: BGMU. 2008;2:47*
2. Chistyakova GG, Sahar GG, Koltun KG, Sikorskogo AV, Doroninoy OK. Innovacii v medicine i farmacii - 2016. *Materialy distancion. nauch.-prakt. konf. studentov i molodyh uchenyh, Minsk, Belorus. gos. med. un-t. pod red. BGMU. 2016;513-517*
3. Chistyakova GG. Ocenka adgezionnoj svyazi fotokompozitov s tvyordymi tkanyami koronok zubov na osnovanii metoda skaniruyushchej elektronnoj mikroskopii i mikrorentgenospektral'nogo analiza. *Sovremennaya stomatologiya Minsk. 2019;4:56*

ԱՏԱՄԻ ՊՍԱԿԻ ԴԵՆՏԻՆԻ ՄԻԿՐՈՏԱՐՐԵՐԻ ԿԱԶՄԻ ՎՐԱ ԱԴՎԵԶԻՎ ՀԱՄԱԿԱՐԳԵՐԻ ԱԶԴԵՑՈՒԹՅԱՆ ՀԱՄԵՄԱՏԱՏՎԱՆ ՎԵՐԼՈՒԾՈՒԹՅՈՒՆ

Գալինա Չիստյակովա

Բելառուսի պետական բժշկական համալսարանի ստոմատոլոգիական պրոպեդևտիկայի և նյութագիտության ամբիոնի դոցենտ, Մինսկ, Բելառուս

Ամփոփում

**Նպատակը.** Ուսումնասիրել «լցանյութ-ատամ» ինտերակտիվ գոտու միկրոտարրերի կազմը, դրա եզրագծի մորֆոլոգիական բնութագրերը, գնահատել V և VII սերնդի ադեզիվ համակարգերի ազդեցությունը իոնափոխանակման բաղադրության վրա, ատամի դենտինի և դենտինի հիբրիդային գոտու միկրոտարրերի բաղադրության որակական գնահատում:

**Նյութեր և մեթոդներ.** Հեռացված ատամներ՝ պահպանված պակով (n=15), էմալը մշակելու համար նախատեսված գել (Վլադմիր), հինգերորդ սերնդի ադեզիվ համակարգ Singlebond - 2 (3M ESPE), բոնդ Prime-Bond Universal (Dentsply Sirona), լուսակարածրացող կոմպոզիտային նյութ Migrofil (Բելառուսի Հանրապետություն): I դասի ձևավորված և լցված խոռոչներով նմուշների միկրոկառուցվածքի ուսումնասիրությունը՝ ըստ Black-ի, իրականացվել է Tescan-ի (Չեխիա) հաստատված սկանավորող էլեկտրոնային մանրադիտակի միջոցով (Չեխիա):

«Լցանյութ-ատամ» ինտերակտիվ գոտու ուսումնասիրությունը իրականացվել է ռենտգենյան սպեկտրային անալիզով, որը բաղկացած է եղել 2 բոլկից՝ օգտագործելով հինգերորդ սերնդի ադեզիվ համակարգ և յոթերորդ սերնդի ադեզիվ համակարգ:

**Եզրակացություններ.** 1. Հինգերորդ սերնդի ադեզիվ համակարգի և ինքնակող ադեզիվ համակարգի արդյունքների համեմատական վերլուծությունը ցույց է տվել, որ ընդհանուր մշակման տեխնիկա կիրառելիս Ca և P միկրոտարրերը ատամի դենտինի կառուցվածքից տեղափոխվում են հիբրիդային գոտի:

VII սերնդի ադեզիվ համակարգ օգտագործելիս տարրերի միգրացիա չի հայտնաբերվել դենտինի կառուցվածքի դենտինալիզացիայի և քսուքի շերտի փոփոխության պատճառով:

2. Պլանային սպեկտրոգրամների որակական գնահատումը բացահայտեց փոփոխված ադեզիվ շերտում ամֆաժնի տարրի ավելացում, ինչը պայմանավորված է կարիեսային պրոցեսից քայքայված ատամի կոշտ հյուսվածքների օդոնտոպատրաստումից հետո քսուքի շերտի առկայությամբ:

3. Si տարրի միգրացիան ցույց է տալիս ադեզիվ և լցանյութի, ինչպես նաև լցանյութի և ատամի դենտինի քիմիական փոխազդեցությունը ադեզիվ շերտի միջով:

4. V սերնդի ադեզիվ համակարգի օգտագործումը ապահովում է լցանյութի մարզինալ կաշուկայի ավելի բարձր որակ դենտինի մակերեսին, քան ինքնակող ադեզիվ համակարգ օգտագործելիս:

КОМПАРАТИВНЫЙ АНАЛИЗ ВЛИЯНИЯ АДГЕЗИВНЫХ СИСТЕМ НА МИКРОЭЛЕМЕНТНЫЙ СОСТАВ ДЕНТИНА КОРОНКОВОЙ ЧАСТИ ЗУБА

Галина Чистякова

Доцент Белорусского государственного медицинского университета, кафедра стоматологической пропедевтики и материаловедения, Минск, Беларусь

Абстракт

**Цель:** Исследование микроэлементного состава интерактивной зоны «пломба-зуб», морфологической характеристики ее границы, оценка влияния адгезивных систем V и VII (самопротравливающего) поколений на ионно-обменный состав дентина коронковой части зуба и качественная оценка микроэлементного состава гибридной зоны дентина.

**Материалы и методы:** Экстрагированные зубы с сохранённой коронковой частью (n=15), гель для травления эмали (Владмива), адгезивная система V поколения Singlebond - 2 (3M ESPE), самопротравливающая адгезивная система Prime-Bond Universal (Dentsply Sirona), светоотверждаемый композиционный материал Мигрофил (Республика Беларусь). Исследование микроструктуры образцов со сформированными и запломбированными полостями I класса по Блэку проводили на аттестованном сканирующем электронном микроскопе высокого разрешения «Mira» фирмы «Tescan» (Чехия) Исследование микроэлементного состава границы «пломба-зуб» проводили методом рентгеноспектрального анализа, которое состояло из 2 блоков: с применением адгезивной системы V поколения и VII поколения адгезивной системы.

**Выводы:** 1. Компаративный анализ результатов использования адгезивной системы V поколения и самопротравливающей адгезивной системы показал, что при использовании техники тотального травления происходит миграция микроэлементов Са и Р из структуры дентина зуба в гибридную зону.

При использовании адгезивной системы VII миграции элементов не установлено, из-за отсутствия деминерализации структуры дентина и модификации смазанного слоя.

2. При качественной оценке планарных спектрограмм установлено повышение элемента углерода в модифицированном адгезивном слое. что обусловлено присутствием смазанного слоя после одонтопрепарирования твердых тканей зубов, деструктированных кариозным процессом

3. Миграция элемента Si свидетельствует о химическом взаимодействии адгезива и пломбировочного материала, а также пломбировочного материала и дентина зуба через слой адгезива.

4. Использование адгезивной системы V поколения обеспечивает более высокое качество краевого прилегания пломбировочного материала к поверхности дентина, чем при использовании самопротравливающей адгезивной системы.