

Investigation of the surface of light-curing dental materials after pre-polymerization heating

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Abstract:

Despite the great variety and wide application of light-cured composite restorative materials for the treatment of hard tissue diseases, the issue of longevity of restorations remains relevant to this day. One of the criteria for the long-term durability of a composite restoration is the finish of the restoration. Poor polishing and surface roughness significantly reduce the service life of the restoration and provide a good environment for biofilm adhesion, loss of luster of the restoration, and caries recurrence. Thus, methods affecting the reduction of surface relief in restorations remain a current issue.

Aim. To evaluate the surface roughness and porosity of composite filling materials after pre-polymerization heating.

Materials and methods. Sixty composite samples were prepared in the form of 1 mm thick, 1.2 cm diameter disks. The samples were divided into 2 groups depending on the presence of pre-polymerization heating. Atomic force microscopy (AFM) was used to measure the roughness and to visualize the surface morphology of the samples. Porosity was measured using a vacuum sputtering unit, and a scanning electron microscope was used to obtain micrographs of the sample.

Results. The use of pre-polymerization heating of composite filling materials in dental practice will allow dentists to significantly reduce the expression of the relief of the composite restoration by reducing the roughness and pore volume in the matrix of the composite filling material, which will certainly optimize the finishing of the restoration.

Conclusions. Pre-polymerization heating has a positive effect on the surface topography of the composite restoration, reducing its roughness and porosity, which improves the stage of finishing: obtaining a dry gloss, ensuring the duration of color stability, as well as reducing the adhesion of microorganisms to the filling material, thereby increasing the longevity of the composite restoration.

Keywords: pre-polymerization heating, roughness, porosity, polymerization, light-cured composite.

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Исследование поверхности светоотверждаемых стоматологических материалов после предполимеризационного нагрева

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Резюме:

Несмотря на большое разнообразие и широкое применение светоотверждаемых композитных реставрационных материалов, вопрос долговечности реставраций остается актуальным и по сей день. Одним из критериев долговечности композитной реставрации является ее качество. Плохая полировка и шероховатость поверхности значительно сокращают срок службы реставрации и создают благоприятную среду для адгезии биопленки, потери блеска реставрации и рецидива кариеса.

Цель. Оценить шероховатость и пористость поверхности композитных пломбировочных материалов после предварительного полимеризационного нагрева.

Материалы и методы. Было подготовлено 60 образцов композитов в виде дисков толщиной 1 мм и диаметром 1,2 см. Образцы были разделены на 2 группы в зависимости от наличия предварительного полимеризационного нагрева. Атомно-силовая микроскопия (АСМ) использовалась для измерения шероховатости и визуализации

морфологии поверхности образцов. Пористость измеряли с помощью вакуумной напылительной установки, а для получения микрофотографий образцов использовали сканирующий электронный микроскоп.

Результаты. Использование в стоматологической практике предполимеризационного нагрева композитных пломбировочных материалов позволяет стоматологам значительно уменьшить выраженность рельефа композитной реставрации за счет уменьшения шероховатости и объема пор в матрице композитного пломбировочного материала, что, безусловно, оптимизирует финишную обработку реставрации.

Выводы. Предполимеризационный нагрев положительно влияет на рельеф поверхности композитной реставрации, уменьшая ее шероховатость и пористость, что улучшает этап финишной обработки: получение сухого блеска, обеспечение длительности цветостойкости, а также уменьшение адгезии микроорганизмов к пломбировочному материалу, тем самым увеличивая долговечность композитной реставрации.

Ключевые слова: предполимеризационный нагрев, шероховатость, пористость, полимеризация, светоотверждаемый композит.

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INTRODUCTION

Composite restorative materials have excellent handling properties, good polishability, high wear resistance, the ability to be used for the restoration of any group of teeth due to the variety of shades and transparency, ease of use, easy to model regardless of the design of the restoration, give teeth a natural look, including the preservation of enamel shine. [1] However, despite the large number of positive physical and mechanical properties of light-cured composite restorative materials the results of clinical studies demonstrate that the biggest problem of composite is the loss of surface gloss and increased roughness. Under the influence of various factors, the color stability of the composite material also changes. The color stability of composite resin is an important property affecting its clinical longevity, which remains an inherent requirement for the material. [2] All staining solutions (tea, coffee, wine, energy drinks and so on) affect the color stability of the tested filling materials: solutions, even distilled water, darken the tested composite samples [3-6]. Authors investigating the effect of coffee on color stability and surface roughness of single-shade resin-based composite materials [7] also noted that composite type and immersion time had a marked effect on color stability and surface roughness of single-shade composite filling material and showed "extremely" high thresholds for perceptibility and acceptability as well as surface roughness even when distilled water was used. The quality of aesthetic restorations depends to a large extent on the finishing and polishing process. A perfectly polished surface of the composite material minimizes plaque accumulation, gingival irritation, and discoloration of the restoration, i.e., it improves color stability and, consequently, the aesthetics of the restoration [8]. Color stability also depends on the surface roughness of the composite restoration, as increased roughness ($> 0.3 \mu\text{m}$) can lead to greater plaque retention and pigment absorption than relatively smooth surfaces [9-11]. Due to the above factors affecting the properties of composite restorative materials, the issue related to the longevity of the restoration remains relevant to this day.

MATERIALS AND METHODS

Composite materials of different groups widely used in therapeutic dentistry were selected for this study. These materials were also selected based on the composition of the polymer matrix, filler particle size and country of manufacture. Composites from different manufacturers with

similar filler particle size were used to obtain relevant results. (Table 1). Samples in the form of disks with thickness of 1 mm and diameter of 1.2 cm were prepared for the study.

The prepared samples were divided into 2 groups according to the presence of thermal prehistory:

Group 1 (A, B, C)-the specimens were made of composite materials, polymerization of which was carried out without thermal prehistory (A – domestic light-cured microhybrid composite material Unirest (Stomadent, Russia), B – imported light-cured microfilled composite material Enamel Plus HRi (Micerium, Italy), C – imported light-cured microhybrid composite material Esthet X HD (Dentsply Sirona, USA).

Group 2 (A1, B1, C1) samples were prepared after preliminary pre-polymerization heating of composite material in a special furnace (Micerium; Avegno, Italy) providing two temperature modes (the first mode – heating to 39°C , the second mode – heating to 55°C). In our study, the second mode – heating to 55°C was used (A1 – domestic light-cured microhybrid composite material Unirest (Stomadent, Russia), B1 – imported light-cured microfilled composite material

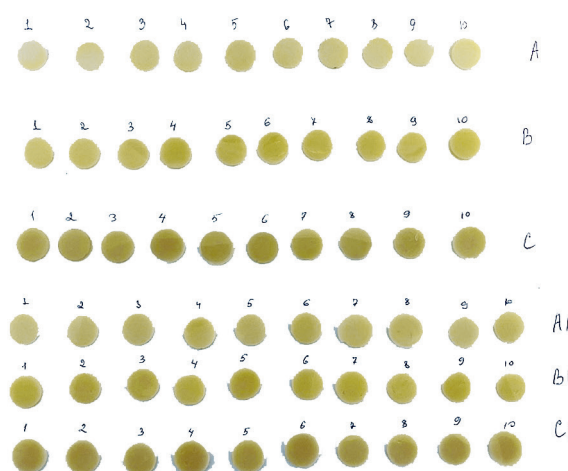


Fig. 1. Under letters A, B, C are prepared samples that have not undergone pre-polymerization heating; under letters A1, B1, C1 are prepared samples heated in an oven at 55°C .

Рис. 1. Под буквами А, В, С – подготовленные образцы, не подвергавшиеся предварительному полимеризационному нагреву; под буквами А1, В1, С1 – подготовленные образцы, нагретые в печи при 55°C .

Table 1. Characteristics of the investigated light-cured composite restorative materials.

Таблица 1. Характеристики исследуемых светоотверждаемых композитных реставрационных материалов.

Composite material	Manufacturer	Composition of the monomer matrix	Filler	Material group
Unirest	Stomadent (Russia)	Urethane methacrylate, bisphenol A glycidyl methacrylate (Bis GMA), triethylene glycol dimethacrylate (TGM), butylated hydroxytoluene, camphoroquinone, trimethacrylate triethanolamine (TMATEA), fluorescent pigment, barium aluminoborosilicate glass PM-3, glass filler GM32087, glacial acetic acid, aluminum oxide, silane A-174, iron oxide pigments: Red 7067, Yellow 7055, Hema Phosphate, acetone	Hybrid with an average filler particle size of 0.7 microns, glass filler filler organic matrix filling degree of 78±1%.	Light-curing microhybrid composite filling material
Enamel plus HRi	Micerium (Italy)	Diurethandimethacrylate, Iso-propylidene-bis (2(3)-hydroxy-3(2)-4(phenoxy) propyl)-bis(methacrylate)(Bis-GMA); 1,4 – Butanedioldimethacrylate	75% by weight (53% by volume). Glass filler: average particle size 0.7 µm; Highly dispersed silica: average particle size 0.04 µm	Light-cured microhybrid composite filling material
Esthet X HD	Dentsply Sirona (USA)	Bis-GMA, Bis-EMA accession product, triethylene glycol dimethacrylate, camphoroquinone (CQ), photoinitiator, stabilizer, pigments	Barium fluoroborosilicate crystals with an average particle size of less than 1 µm and silicon nanofiller (particle size 0.04 µm)	Light-curing microfilled composite restorative material



Fig. 2. Furnace (Micerium; Avegno, Italy) for heating composite materials. T2- mode 55°C.

Рисунок. 2. Печь (Micerium; Avegno, Италия) для нагрева композитных материалов. T2-режим 55°C.

Enamel Plus HRi (Micerium, Italy), C1 – imported light-cured microhybrid composite material Esthet X (Dentsply Sirona, USA). (Fig 1, 2).



Fig. 3. Bruker Innova.

Рис. 3. Bruker Innova.

Roughness evaluation

Atomic force microscopy (AFM) was used to measure the roughness and to visualize the surface morphology of the samples. The atomic force microscope was a Bruker Innova instrument (Fig.3)

Image acquisition was performed in the semi-contact mode. The probe sensor was TESP-V2 with a triangular-shaped probe with a tip radius of 7nm. The scanning area was 50x50µm. Frequency 0.5 Hz. The study of roughness was reduced to the processing of image data obtained using AFM. For this purpose, three arbitrary sections were made

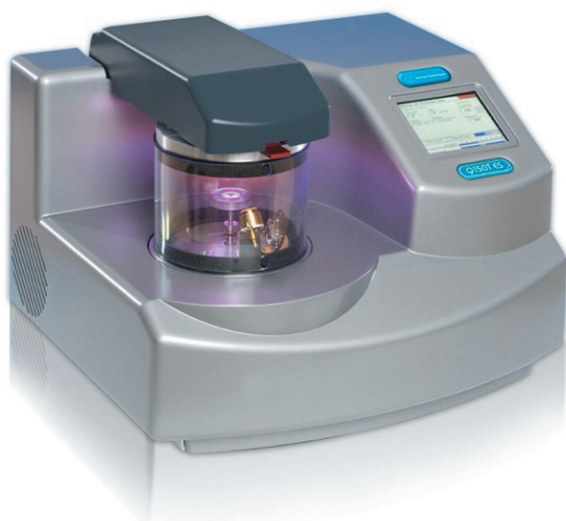


Fig. 4. Quorum Q150T ES – vacuum spraying machine.

Рис. 4. Quorum Q150T ES – вакуумная распылительная машина.

in the height map channel and the necessary roughness parameters Ra, Rz were calculated by means of software.

Porosity estimation

To measure porosity, the sample preparation process included:

Samples were placed in the vacuum chamber of a Quorum Q150T ES vacuum sputtering machine to form a thin conductive film. (Fig. 4). Graphite was chosen as the material. The thickness of the thin film as a result of sputtering was 10-15nm. The thin conductive film is formed in order to remove excessive surface charge arising in the



Fig. 5. Carl Zeiss Auriga Crossbeam electron microscope

Рис. 5. Электронный микроскоп Carl Zeiss Auriga Crossbeam

dielectric sample in the process of its exposure to a focused electron beam.

Measurement technique

A Carl Zeiss Auriga Crossbeam scanning electron microscope was used to obtain micrographs of the sample (Fig. 5).

During electron microscopic analysis, a secondary electron detector was used to visualize the surface of the samples.

An in-lens secondary electron detector (InLens) was used to visualize the internal structure of the samples.

Mode of operation of the electron microscope:

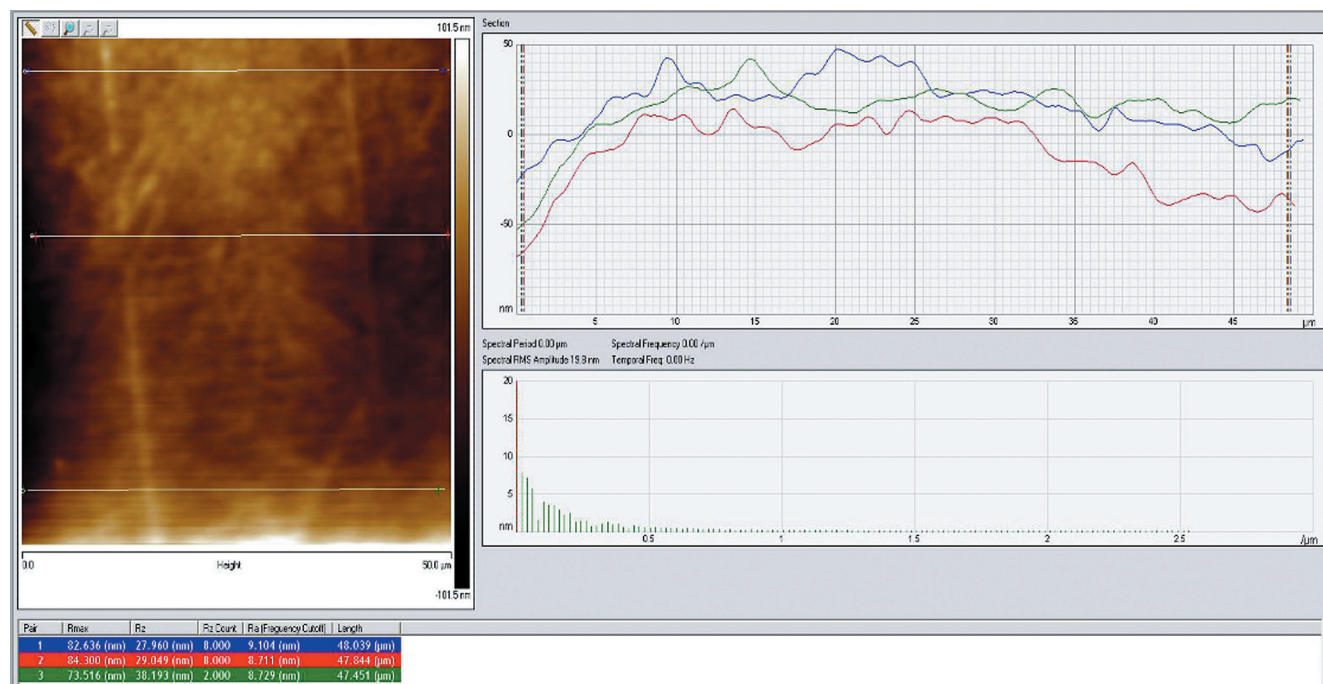


Fig. 6. Enamel Plus HRI heated. No sharp peaks are visually detected on the graph, almost equal width and uniformly decreasing height as the distance from the origin indicates a decrease in surface roughness and roughness of the sample after heating.

Рис. 6. Enamel Plus HRI в нагретом состоянии. На графике визуально не обнаруживается резких пиков, практически одинаковая ширина и равномерно уменьшающаяся высота по мере удаления от начала координат свидетельствует о снижении шероховатости поверхности и шероховатости образца после нагрева.

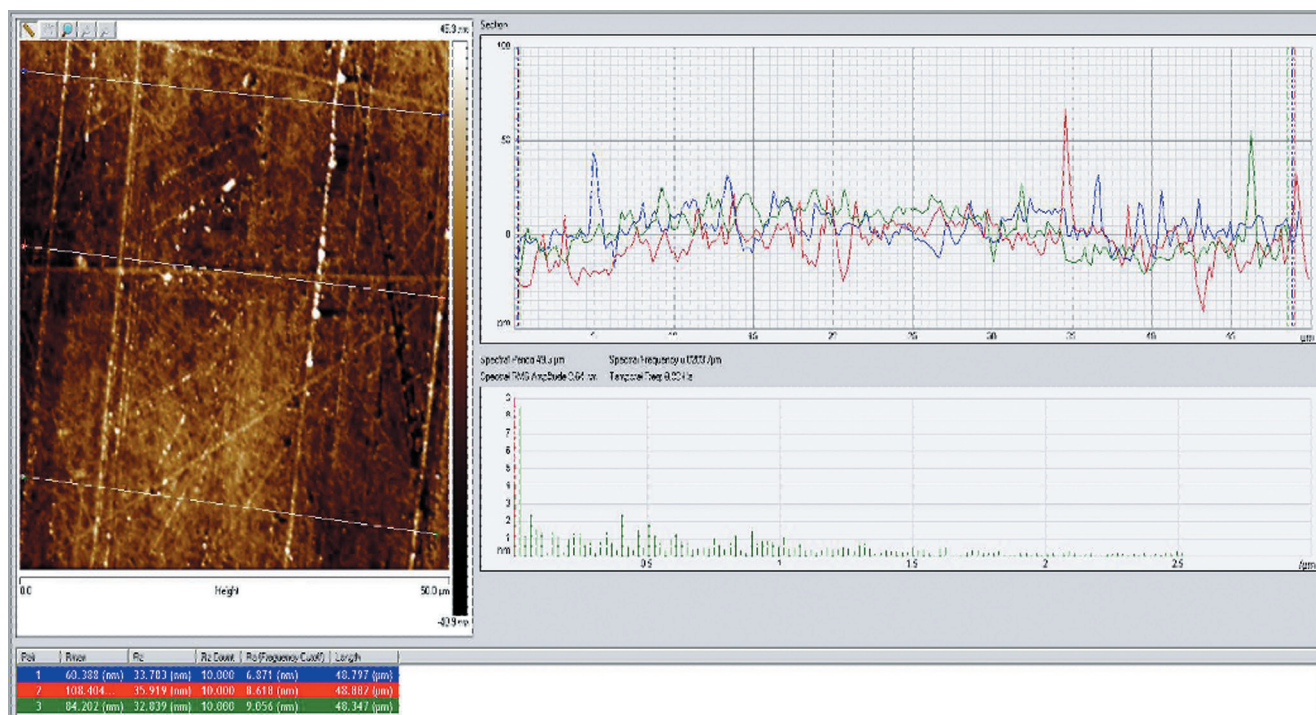


Fig. 7. Enamel Plus HRI unheated. The graph visually shows a large number of sharp peaks throughout, which indicates the rough and uneven surface of the structure of this material.

Рис. 7. Enamel Plus HRI без нагрева. На графике визуально видно большое количество острых пиков, что говорит о грубой и неровной поверхности структуры этого материала.

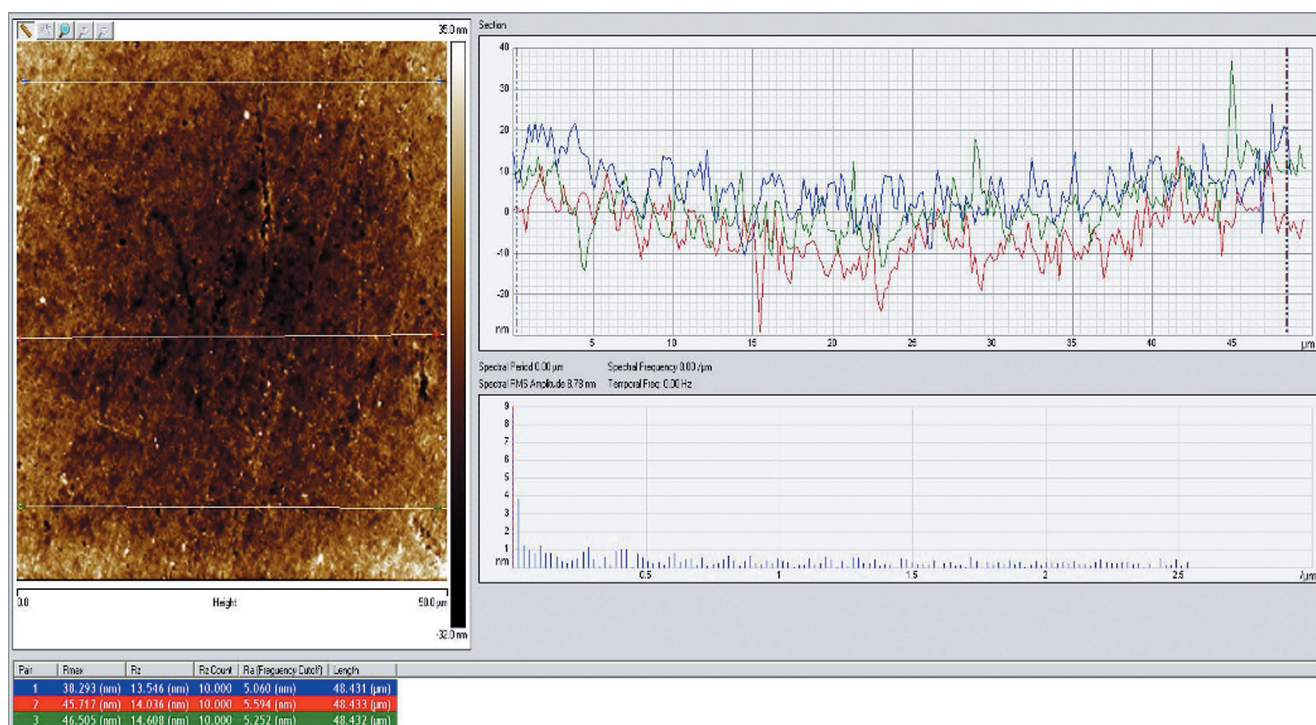


Fig. 8. Esthet X HD heated. From the graph, it can be visually determined that the nature of the peak height and width, as well as the nature of the scatter of the Rmax parameter between neighboring measurements for this material showed approximately the same results as the Esthet X HD material without thermal prehistory.

Рис. 8. Esthet X HD после нагрева. Из графика можно визуально определить, что характер высоты и ширины пика, а также характер разброса параметра Rmax между соседними измерениями для этого материала показал примерно те же результаты, что и для материала Esthet X HD без термической предыстории.

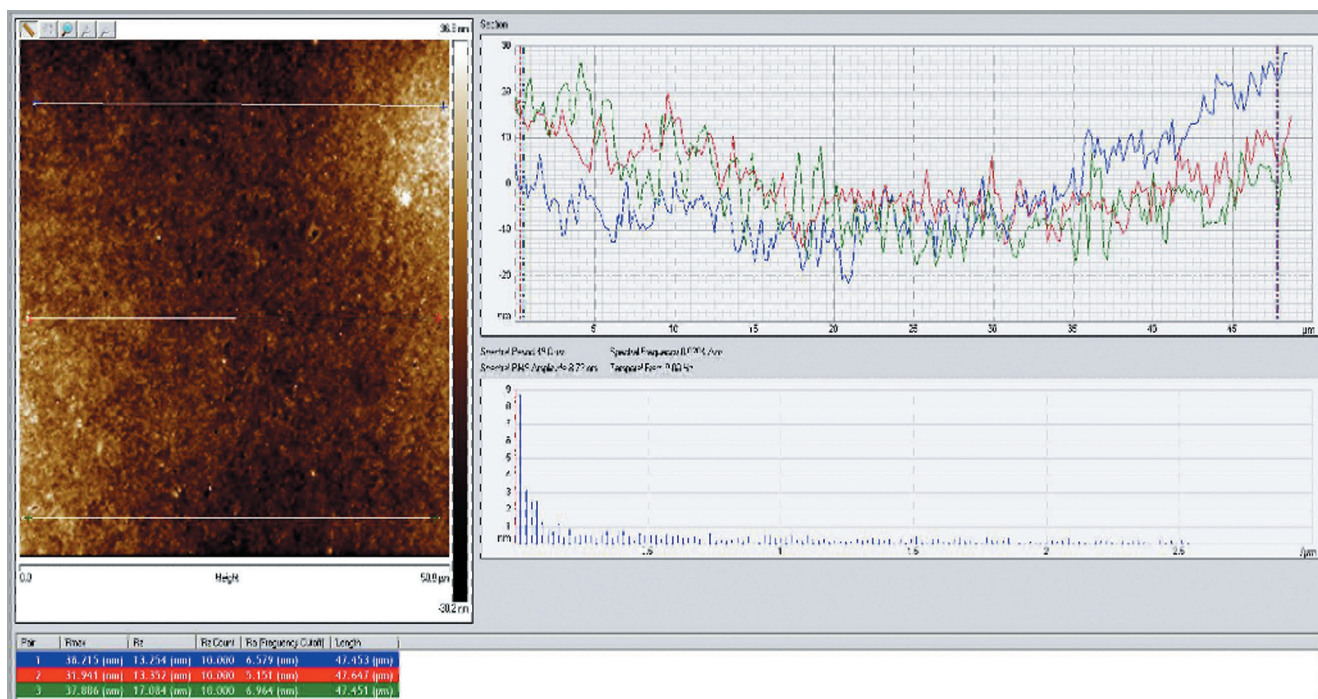


Fig. 9. Esthet X HD unheated. From the graph, it can be visually determined that the nature of the peak height and width, as well as the nature of the scatter of the Rmax parameter between neighboring measurements for this material showed approximately the same results as the Esthet X HD preheat-treated material.

Рис. 9. Esthet X HD без нагрева. Из графика можно визуально определить, что характер высоты и ширины пика, а также характер разброса параметра Rmax между соседними измерениями для этого материала показал примерно те же результаты, что и для материала Esthet X HD, прошедшего предварительную термообработку.

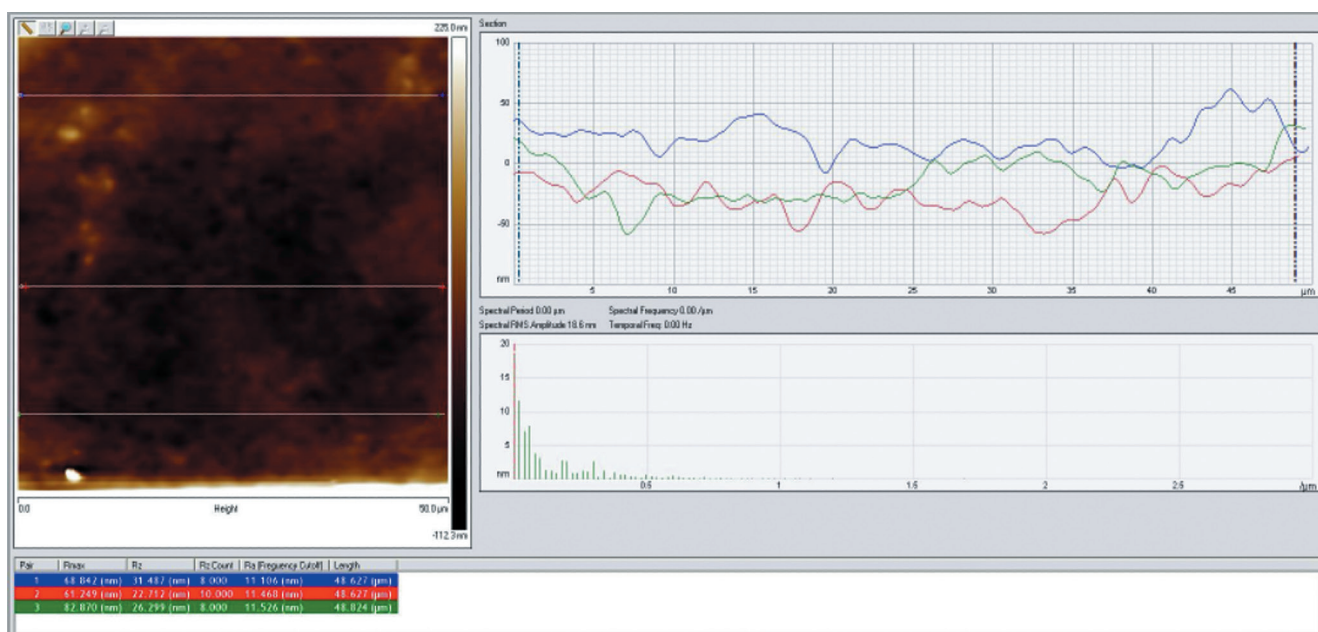


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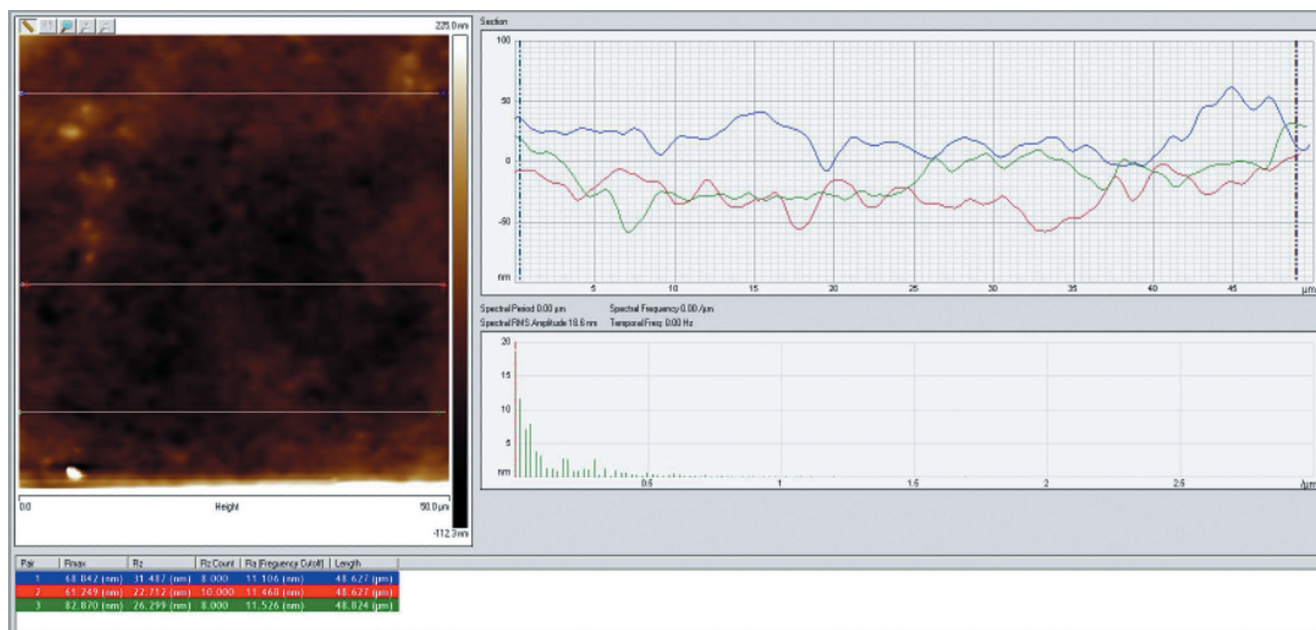


Fig. 10. Unirest heated. On the graph it is possible to visually determine smoother peaks of the same width and small variation of Rmax parameter between neighboring measurements in comparison with Unirest material not preheated.

Рис. 10. Нагретый материал Unirest. На графике можно визуально определить более гладкие пики одинаковой ширины и небольшой разброс параметра Rmax между соседними измерениями по сравнению с материалом Unirest без предварительного нагрева.

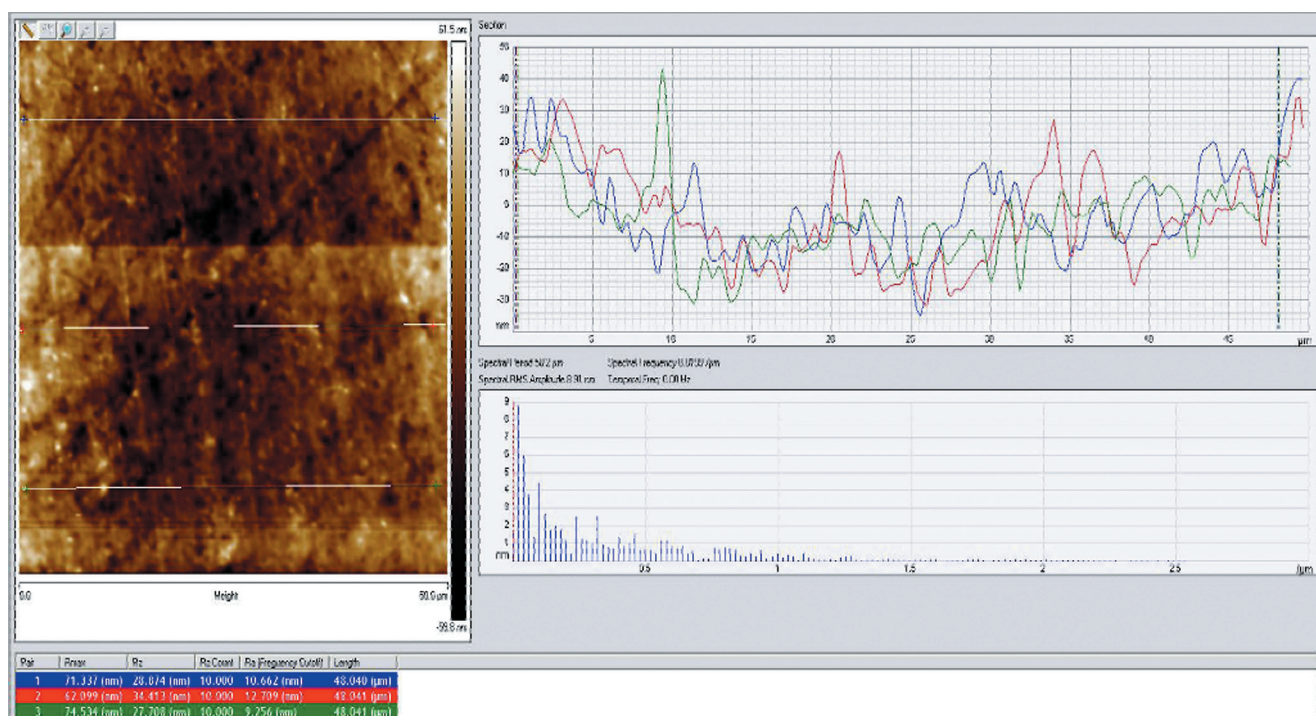


Fig. 11. Unirest unheated. On the graph visually it is possible to note a large number of sharp irregular peaks, which indicates the expression of rough and uneven surface of the structure of this material.

Рис. 11. Unirest без нагрева. На графике визуально можно отметить большое количество резких неравномерных пиков, что свидетельствует о выраженности грубой и неровной поверхности структуры данного материала.

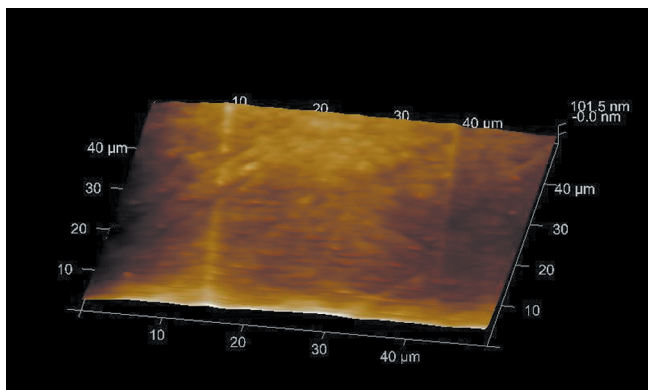


Fig. 12. Enamel Plus HRi heated. According to the results of AFM images, a more homogeneous and smoothed surface structure of the material is visually noted.

Рис. 12. Enamel Plus HRi в нагретом состоянии. По результатам АСМ-изображений визуально отмечается более однородная и сглаженная структура поверхности материала.

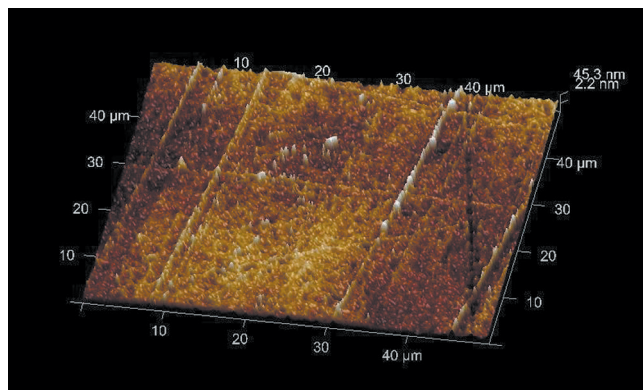


Fig. 13. Enamel Plus HRi not heated. According to the results of AFM images, visually more rough and uneven surface of the material is noted.

Рис. 13. Enamel Plus HRi без нагрева. По результатам АСМ-изображений визуально отмечается более шероховатая и неровная поверхность материала.

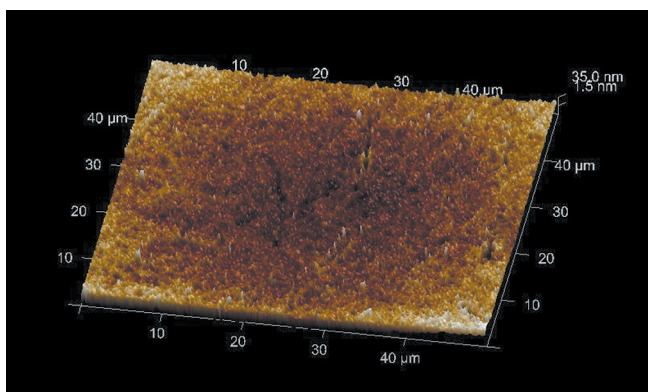


Fig. 14. Esthet X HD heated. The surface structure of the material visually before and after heating on AFM images showed no significant differences.

Рис. 14. Esthet X HD в нагретом состоянии. Структура поверхности материала до и после нагрева на АСМ-изображениях не показала существенных различий.

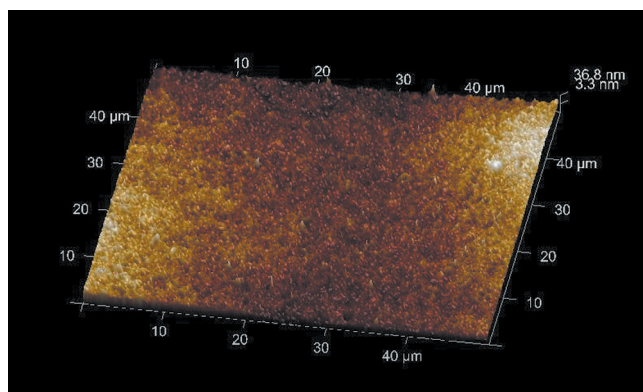


Fig. 15. Esthet X HD unheated. The surface structure of the material visually before and after heating on AFM images showed no significant differences.

Рис. 15. Esthet X HD без нагрева. Структура поверхности материала до и после нагрева на АСМ-изображениях не показала существенных различий.

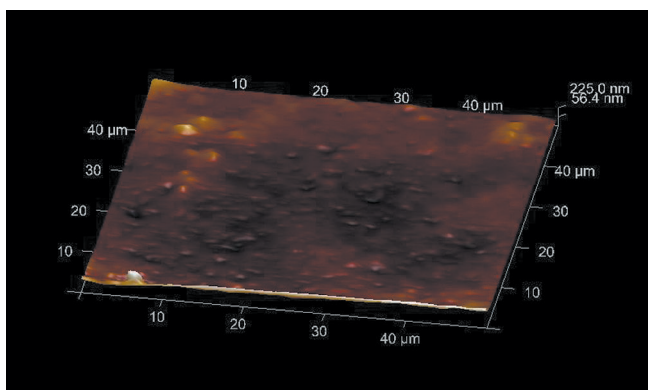


Fig. 16. Unirest heated. Based on the results of AFM images, a more homogeneous and smoothed surface structure of the material is visually noted.

Рис. 16. Нагретый Unirest. По результатам АСМ-изображений визуально отмечается более однородная и сглаженная структура поверхности материала.

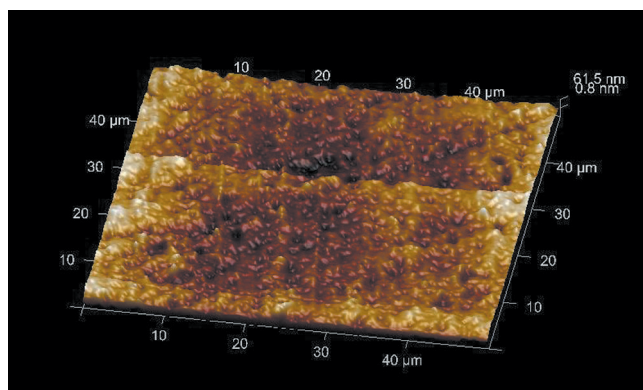


Fig. 17. Unirest not heated. According to the results of AFM images, a more rough structure of the material surface is visually noted.

Рис. 17. Unirest без нагрева. По результатам АСМ-изображений визуально отмечается более шероховатая структура поверхности материала.

Accelerating voltage -5 kV; Aperture diaphragm size – 30 mm; Working distance – 6 mm; Electron beam current – 30pA.

Roughness measurement results are demonstrated and described on the Figures 6-11.

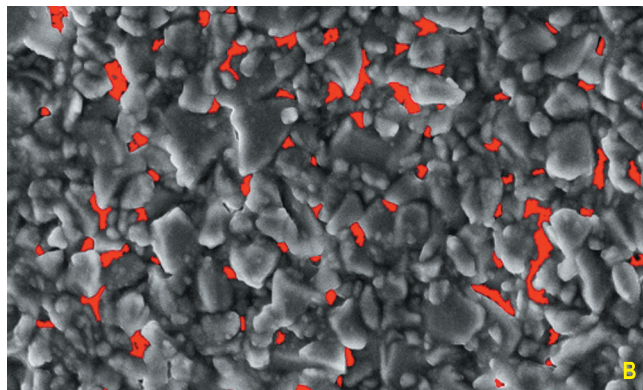
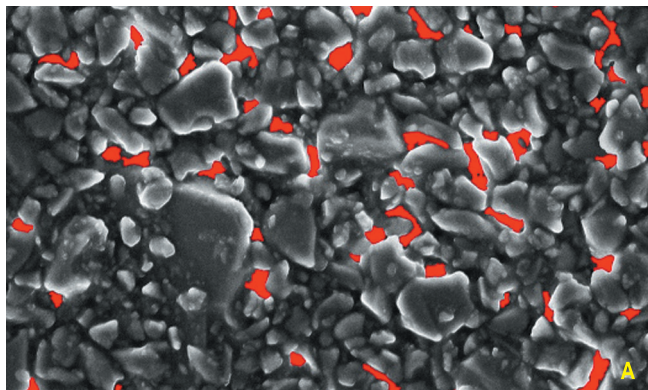


Fig. 18. On electron scanning microscopy of the composite material that has not undergone pre-polymerisation heating, we note a visually larger pore size in the matrix structure (A). In SEM images of the composite material with pre-thermal preheating (B), we note a smoother dispersed structure of the composite matrix and smaller pore size.

Рис. 18. На электронной сканирующей микроскопии композитного материала, не прошедшего предварительный предполимеризационный нагрев, мы отмечаем визуально больший размер пор в структуре матрицы (A). На СЭМ-изображениях композитного материала с предварительной термической предысторией (B) мы отмечаем более гладкую дисперсную структуру композитной матрицы и меньший размер пор.

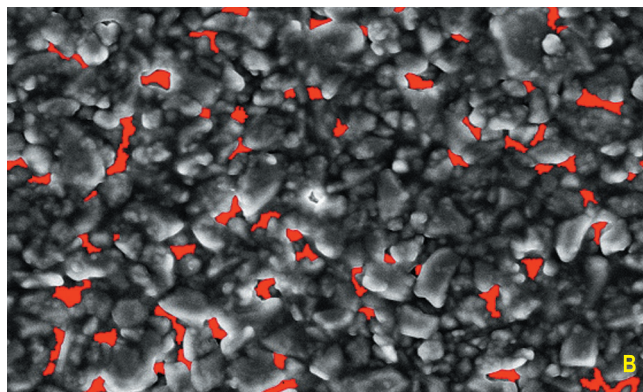
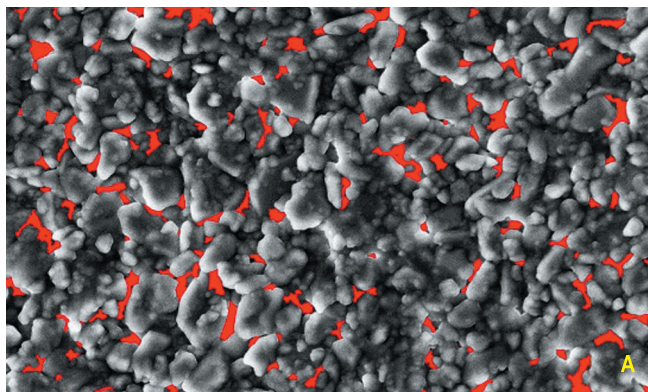


Fig. 19. Esthet X HD without pre-polymerisation heating (A), according to electron scanning microscopy data, has a higher number and size of pores than material with thermal preheating (B), but this material showed no significant change in surface structure before and after pre-polymerisation heating.

Рис. 19. Esthet X HD без предварительного полимеризационного нагрева (A), согласно данным электронной сканирующей микроскопии, имеет большее количество и размер пор, чем материал с предварительным термическим нагревом (B), однако этот материал не показал значительного изменения структуры поверхности до и после предварительного полимеризационного нагрева.

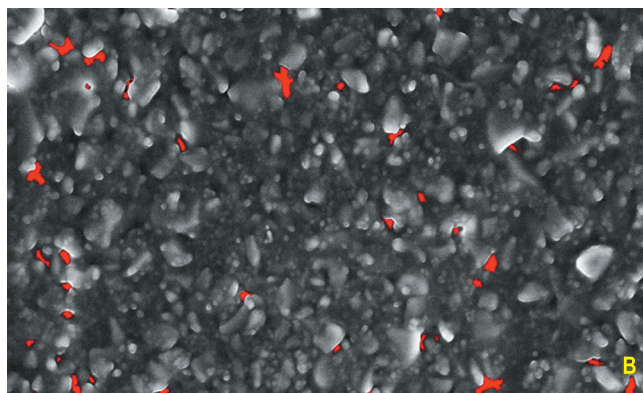
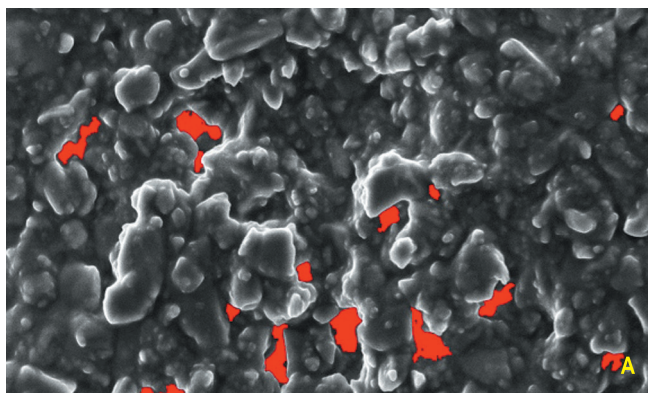


Fig. 20. Electron scanning microscopy of Enamel Plus HRi composite not subjected to pre-polymerisation heating shows a visually larger pore size of the matrix structure (A) SEM images of the composite material with thermal preheating (B) show a smoother and more homogeneous composite matrix structure and a small number of small isolated pores.

Рис. 20. На электронной сканирующей микроскопии композитного Enamel Plus HRi, не подвергнутого предварительному предполимеризационному нагреву, отмечается визуально больший размер пор матричной структуры (A) На СЭМ-изображениях композиционного материала с термической предысторией (B) отмечается более гладкая и однородная структура композитной матрицы и небольшое количество мелких изолированных пор.

Table 2. The results of porosity measurement.
Таблица 2. Результаты измерения пористости.

	Unirest	Enamel HRI	Esthet-X HD
Percent porosity to	Area 1- 3,66%	Area 1- 2,52%	Area 1 – 5,35%
	Area 2 – 4%	Area 2– 2,25%	Area 2– 5,40%
	Cf. value. – 3,83%	Cf. value. – 2,38%	Cf. value. – 5,37%
Percent porosity after	Area 1– 4,05%	Area 1- 1,23%	Area 1 – 4,1%
	Area 2– 3,37%	Area 2 – 2,37%	Area 2 – 3,77%
	Cf. value. – 3,71%	Cf. value. – 1,8%	Cf. value. – 3,93%

Results of atomic force microscopy are demonstrated and described on the Figures 12-17.

Measurement of porosity by SEM image processing method are demonstrated and described on the Figures 18-20. According to the scanning electron microscopy data, the material with thermal preconditioning has higher density and thus lower roughness due to greater smoothing of the dispersed matrix structure after the composite conversion from heating.

Discussion.

From the results of the roughness study and AFM images, it can be seen that the pattern of surface roughness has changed for the Unirest and Enamel Plus HRI specimens. The number of irregularities per unit length became smaller and the specimens appear more "smoothed". This is evidenced by the small scatter of the Rmax parameter between neighboring measurements.

The Esthet-X sample shows no significant changes in surface morphology.

The porosity of the samples was measured by SEM image processing. The results of porosity measurement are given in the table 2.

It was found that as a result of pre-polymerization heating of the composite, the porosity of each manufacturer's material decreased. The decrease in porosity in the case of Unirest had the smallest value and amounted to 0.12%. In the case of Enamel Plus HRI 0.58% and in the case of Esthet-X HD, the change was the most significant and amounted to 1.44%.

It should also be noted that in the case of the Enamel manufacturer's samples, the "after" sample shows a decrease in the size of the particles in the composite material.

CONCLUSIONS

Thus, from the results of the study, we can conclude that preheating of composite material reduces the roughness and porosity of the composite, making its surface smoother and more homogeneous, which in turn has an impact on the quality and durability of restorations. The use of the composite preheating method will be very relevant among dentists, as to this day, doctors in their clinical practice are still searching for effective polishing options to create a smooth surface of composite restorations.

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