Evaluation of surface characteristics of direct composite resins after finishing and polishing using fractal analysis

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Abstract. Surface characteristics of two composite resins (Filtek Ultimate Universal Restorative, 3M ESPE and Filtek Ultimate Flowable Restorative, 3M ESPE) after finishing and polishing procedure using three systems: two steps Sof-Lex system, 3M ESPE, multi-steps Super Snap system, Shofu, Inc. Kyoto, Japan and multi-steps OptiDisc system, KerrHawe SA were analyse using SEM and AFM investigations. Fractal analysis was performed on SEM and AFM images and the mean fractal dimension was calculated for each sample using FracLac software. For both tested materials decreased fractal dimension with the increase of SEM magnification was observed. Multi-steps OptiDisc system determined the biggest decrease of fractal dimension for both materials, followed by Sof-Lex system and Super Snap. Fractal analysis seems to be an appropriate method to investigate the complex structure of composite resins.

1. Introduction
In the last years fractal or multifractal geometry was largely applied in medicine, in recognition of human features and in texture analysis [1-5]. Application of such geometry generally consists in recognition of fractal characteristics. Different models were proposed to estimate the fractal dimension or the multifractal spectrum of a certain signal [4].

The idea of natural phenomena description by scale measurement in statistic analysis is not a novelty, a lot of studies being published over the time [6-9]. A recent interest on this idea was observed because a big number of physical systems present similar behaviour on different observation scales. In the 60s Benoît Mandelbrot was the first that used the notion of “fractal” to name the objects with complex geometry that cannot be characterised by an integral dimension [7]. Fractal geometry allows the description of irregular shapes, fragments of natural features or complex objects which traditional Euclidean geometry fails to evaluate. The concept offers a simple geometrical interpretation and it can be applied in different fields as biology, geophysics or fluid mechanics [2,3].

A major characteristics of fractal objects is the fact that metric measured properties as the length or the area are dependent on the measurement scale. Fractal dimension is a measure of the object complexity [10]. Geometric objects have topological dimension: for example a line has the dimension of 1, plane surfaces (circles, squares) have the dimension of 2. Figures which cannot be characterised by known dimensions were created and their evaluation became difficult. Fractal dimension should be analysed in conjunction with Euclidean dimension and with topological dimension [9]. From the Euclidean geometry point of view, Euclidean dimension of a geometrical object is the number of coordinates needed to determine that element. Euclidean dimension and the topological dimension are
the limits for fractal dimension, according to the following relation: \( E_D \geq D_B \geq T_D \), where \( D_B \) is fractal dimension, \( T \) is the topological dimension, and \( E_D \) is the Euclidean dimension. There are many ways to define fractal dimension: Hausdorff-Bezicovici dimension, Minkowski-Bouligand dimension, Kolmogoroff dimension, etc [9]. Unfortunately, there is a lack of studies in the literature that evaluate the fractal dimension of composite resins surface.

Composite resins have become the mostly used materials for direct restorations. The quality of the restoration surface has a significant importance in ensuring the longevity of the restoration in oral cavity. The surface roughness of composite resins influences: the bacterial plaque adhesion, the resistance to abrasion and wear kinetics, the tactile perception, the resistance of restoration to discoloration, the natural shine of the restoration. Finishing and polishing are mandatory steps in direct restoration procedure which have as major goal to obtain a smooth and glossy surface of the material. Previous studies have shown that finishing procedure determines increased surface roughness of composite resins when compared to the surface condition of the material when no such procedures were made [11, 12]. Different effects on composite surface were recorded when one step, two steps or multi-steps finishing and polishing systems were evaluated [13-15].

The aim of the study was to evaluate by fractal analysis the surface characteristics of different direct restorative resin based materials after finishing and polishing procedures.

2. Experimental procedures

2.1. Sample preparation

Two composite resins were chosen for this study: a high viscosity, nano-filled, universal composite resin- Filtek Ultimate Universal Restorative (3M ESPE) and a flowable, nano-filled, composite resin-Filtek Ultimate Flowable Restorative (3M ESPE). Details regarding the chemical composition of the composite resins are presented in Table 1.

| Name                  | Producer   | Organic matrix | Filler            | Filler size |
|-----------------------|------------|----------------|-------------------|-------------|
| Filtek Ultimate Universal Restorative | 3M ESPE    | bis-GMA, UDMA, TEGDMA | Silica oxide, Zirconia oxide, Clusters | 20 nm, 4-11 nm, 0.6-20 µm |
| Filtek Ultimate Flowable Restorative | 3M ESPE    | BisGMA, TEGDMA | Yterbiu fluoride, Silica oxide, Clusters | 0.1-0.5 µm, 20 nm, 0.6-10 µm |

Twenty cylindrical samples of each material having the high of 2 mm and the diameter of 5 mm were prepared in metallic molds, in direct contact with glass slabs. Transparent matrix was placed between the glass slab and the molds in order to obtain samples with flat surfaces and having no bubbles inside. After filling the mold with the material, a second matrix was applied and a glass slab was pressed on top to remove the excess of the material. The samples were lightcured for 40 second from the bottom and from the top through the glass slabs to obtain complete material polymerization. LED (light emitting diode) lamp (Optilight LD MAX, Gnatus) having the wavelength of 470-480 nm was used to lightcure the materials.

After removing the materials from the molds, finishing and polishing procedures were performed using different systems. The samples were randomly and equally split in 4 groups (one control group- the samples were not finished and polished and other 3 groups according to the system used for finishing and polishing: two steps Sof-Lex system (3M ESPE) (group 1), multi-steps Super Snap
system (Shofu, Inc. Kyoto, Japan) (group 2) and multi-steps OptiDisc system (KerrHawe SA) (group 3). For Sof-Lex system beige and white lamellar disks were used without paste or water, for 1 minute (30 seconds for each disk) at 10,000-20,000 r.p.m (revolutions per minute), according to the instructions provided by the producers. In the group where multi-steps Super Snap system was used, the samples were finished and polished using purple, green, and pink disks. Each disk was used only one time for a single sample, without paste or water, for 1 minute (20 seconds for each disk) at 10,000-20,000 r.p.m. For multi-steps OptiDisc system, the samples were finished using beige, orange and yellow disks and polished using Optishine brush. Each disk or brush was used 20 seconds, at 10,000-20,000 r.p.m (the disks) and at 5000 r.p.m (the brush).

Each sample was cut in two halves using a diamond disk at low speed. A half was used for SEM evaluation and the other half for AFM evaluation.

2.2 Evaluation of the samples surface

The surface microstructure of the samples was assessed by SEM (VEGA II LSH TESCAN microscope) and AFM (NTEGRA SPECTRA, NT-MDT) evaluation. Images at different magnifications: 500, 1000, 2000, and 5000 were recorded for each sample. In AFM analysis the root mean square roughness of the surface of each sample was determined.

Fractal analysis was performed on SEM and AFM images and the mean fractal dimension was calculated for each sample using FracLac software. This software was designed to make the fractal analysis of the images and it was developed as a plugin for Java Image J software. The fractal analysis is used for complex and heterogeneous binary digital images [16]. Generally, as a measure of the complexity dimension, fractal analysis offers values named fractal dimension or “box-counting” (D_0). D_0 is measured in relation to the increasing of the details with the increasing of the analysis scale (ε). On a specific image to be evaluated, grids of descending caliber were applied over the image and the number of boxes that contain pixels inside were counted N(ε). D_0 was calculated using the following formula:

\[ D_0 = \lim_{\varepsilon \to 0} \left( \frac{\log(N(\varepsilon))}{\log(\varepsilon)} \right) \]

3. Results and discussions

An example of SEM image conversion in binary format and of using “box-counting” method to calculate the fractal dimension from the slope of the regression line Ln (N(ε)) according to Ln(ε) are presented in Figure 1 a and b.

![SEM image](image1)

Figure 1. a) The conversion of SEM image in binary format; b) Slope of the regression line determination for fractal dimension calculation.

Some numeral results obtained by fractal analysis of the SEM images at different magnification of Filtek Ultimate Universal Restorative are presented in Table 2.
Table 2. Numerical results obtained by fractal analysis for Filtek Ultimate Universal Restorative.

| Martor | 1. Fractional Lacunarity (FracLac) | 2. Number of Scan Positions | 3. Scan Type | 4. Formula for Fractal Dimension | 5. Y₀ for the SET (Ø₁₀) | 6. β = Σ(ln(Y₍₀₎)/ln(Ø₁₀)) for Da | 7. Standard Deviation (SD) for Da | 8. ± SD with highest r² for Da | 9. r² for Da | 10. ± SE for Da |
|--------|----------------------------------|----------------------------|-------------|---------------------------------|------------------------|-----------------------------|-----------------------------|-----------------------------|------------------|------------------|
| 500x10f500 | 500 | 12 | Da₀₀₀ = Box × Regression Y₍₀₎₀₀₀ = C₀₀₀ | 1,8448 | 0,0409 | 1,9021 | 0,9958 | 0,1087 |
| 1kxf12f12ti | 1000 | 12 | Da₀₀₀ = Box × Regression Y₍₀₎₀₀₀ = C₀₀₀ | 1,8015 | 0,0369 | 1,8629 | 0,9981 | 0,0711 |
| 2kxf2kxti | 2000 | 12 | Da₀₀₀ = Box × Regression Y₍₀₎₀₀₀ = C₀₀₀ | 1,7758 | 0,0400 | 1,7474 | 0,9946 | 0,1142 |
| 5kx5kx5kx | 5000 | 12 | Da₀₀₀ = Box × Regression Y₍₀₎₀₀₀ = C₀₀₀ | 1,7118 | 0,0397 | 1,6922 | 0,9923 | 0,1314 |

The mean fractal values as a result of 12 determination and its variation with the increase of microscopic magnification are presented in Figure 2 and Figure 3 (for Filtek Ultimate Universal Restorative and Filtek Ultimate Flowable Restorative, respectively). The fractal values with the minimal square deviation for Filtek Ultimate Universal Restorative and Filtek Ultimate Flowable Restorative are presented in Table 4 and Table 5.

Figure 2. Mean fractal values variation for Filtek Ultimate Universal Restorative.

Figure 3. Mean fractal values variation for Filtek Ultimate Flowable Restorative.

Decreasing fractal dimension with increasing SEM magnification was observed for both tested materials. The biggest variation of mean fractal dimension at all magnification degrees was recorded for the samples in group 3. The samples in groups 2 and 1 showed the same tendency of fractal dimension variation with the increase of SEM magnification. The samples of both composite resins in group 2 presented the lowest fractal variation.
Fractal analysis of AFM microscopic images was performed using $80 \times 80 \mu m^2$ surfaces. The root mean square roughness values for Filtek Ultimate Universal Restorative and Filtek Ultimate Flowable Restorative composite resins were presented in Table 3. Also, fractal dimension for both resin materials are showed in Table 3.

**Table 3.** Fractal dimension and root mean square roughness for both composite resins.

| Fractal dimension, $D_B$ | Root mean square roughness, $R_a (\mu m)$ | Control | Filtek Ultimate Universal Restorative | Group 1 | Filtek Ultimate Flowable Restorative | Group 2 | Group 3 |
|-------------------------|------------------------------------------|--------|--------------------------------------|--------|--------------------------------------|--------|--------|
| 1.8095                  | 0.04115                                  |        | Control                              |        | Filtek Ultimate Flowable Restorative |        |        |
| 1.7991                  | 0.48920                                  |        | Group 1                              |        |                                      |        |        |
| 1.8097                  | 0.85260                                  |        | Group 2                              |        |                                      |        |        |
| 1.7933                  | 0.41110                                  |        | Group 3                              |        |                                      |        |        |
| 1.8116                  | 0.90600                                  |        | Control                              |        | Filtek Ultimate Flowable Restorative |        |        |
| 1.8094                  | 1.10220                                  |        | Group 1                              |        |                                      |        |        |
| 1.8096                  | 1.12180                                  |        | Group 2                              |        |                                      |        |        |
| 1.4073                  | 1.18580                                  |        | Group 3                              |        |                                      |        |        |

When we analyse the distribution of fractal dimension according to surface roughness an exponential distribution was obtained (Figure 6).

The maximum value of fractal dimension for the surface that we analyse is 2. Asymptotically increase of fractal dimension with the increase in surface roughness was observed. Pearson correlation coefficient was calculated in order to establish the correlation between surface roughness and fractal dimension. The value of 0.41486 with a significant coefficient of 0.17992 correspond to a moderate correlation, but the relationship is not linear.

Previous studies that aimed to establish a possible correlation between the fractal dimension and composite microstructure focused on the type of composite resin according to the filler particle size. The results have shown that the fractal dimension increases as the matrix-filler interface irregularities increase and the adhesion between the two components become stronger (Irina).

In the study of Nica I et al. increased fractal dimension was reported with the decrease of clusters dimension. Other studies focused on fractal analysis to understand the fracture behaviour of the composite resins [14,15,18-20].
The results confirmed the potential of fractal analysis to explain the complex mechanisms involved in the fracture of brittle materials.

4. Conclusions
Determination of fractal dimension on AFM images can be correlated to the mean square roughness and this investigation can be used to quantitative characterization of composite resins surface after finishing and polishing. Without making any quantitative correlation on fractal analysis of SEM images, we can conclude that multi-steps OptiDisc system determined the biggest decrease of fractal dimension for both materials, followed by Sof-Lex system. Super Snap system did not led to significantly changes of fractal dimension. Fractal analysis seems to be an appropriate method to investigate the complex structure of composite resins.

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Figure 6. Distribution of fractal dimension according to the surface roughness.
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