A COMPARATIVE IN VITRO STUDY OF COMPOSITES: COLOUR STABILITY

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INTRODUCTION:
Composites are currently some of the most widely used restorative materials in clinical practice due to their mechanical properties and the increasing aesthetic demands of patients. The first step when it comes to obtaining a composite restoration with good aesthetics requires an understanding of the characteristics of both the material to be used and the teeth to be restored.

Tooth colour is influenced by numerous factors, such as lighting conditions and the translucency, opacity, light dispersion and shininess of the teeth.
Similarly, there are numerous types of composite, which are polymeric materials comprising two phases, namely an organic matrix and an inorganic filler. The fact that the former is based on diacrylate and methacrylate polymers means that it tends to absorb water, thus meaning that when exposed for a long period of time in the oral medium, its physical, mechanical, surface or colour properties can be affected.

This water absorption, and therefore the susceptibility of composites to staining, is related to the hydrophilic or hydrophobic nature of the resin matrix. It is well known in the literature that Bis-GMA- and TEGDMA-based composites tend to absorb most water, whereas UDMA-based composites absorb the least and are therefore most resistant to staining. This fact has also been related to the degree of preservation of these composites.

As far as the filler is concerned, several significant breakthroughs in the production of new composites have been achieved in the past few years by applying nanotechnology, which allows particles with a size of between 1 and 100 nm to be introduced. As a result, there is currently a wide variety of composites containing filler particles with sizes ranging from 100 nm to 2-3 μm.

The main advantages of nanoparticle-containing composites include reduced contraction during polymerisation and improved mechanical and optical properties as they allow a better shine to be obtained and less wear after polishing. However, several authors have noted that their colour stability has not been well studied.

There are considered to be three main types of composite discoloration: 1) external staining due to the build-up of plaque, which stains the surface; 2) as a result of water absorption or degradation of the material; and 3) intrinsic colouration resulting from physicochemical reactions inside the restoration. It is vital to use polishing systems to obtain stain resistance and improved aesthetics.

AIM:
To evaluate the colour stability of 8 colour A2 composites (5 nanohybrid, 2 microhybrid and 1 ormocer) after an ageing process involving thermal cycling and immersion in different colouring solutions for 30 days, followed by a comparison with untreated samples.
MATERIAL AND METHODS:

MATERIAL:
Composites:

<table>
<thead>
<tr>
<th>composite</th>
<th>study code</th>
<th>manufacturer</th>
<th>type</th>
</tr>
</thead>
<tbody>
<tr>
<td>Ventura Nanolux</td>
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<td>Madespa, S.A, Toledo, Spain</td>
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</tr>
<tr>
<td>Grandio</td>
<td>GR</td>
<td>VOCO, Cuxhaven, Germany</td>
<td></td>
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<td>Ceram X</td>
<td>CX</td>
<td>Dentsply, Konstanz, Germany</td>
<td></td>
</tr>
<tr>
<td>Tetric EvoCeram</td>
<td>TEC</td>
<td>Ivoclar Vivadent, Schaan, Liechtenstein</td>
<td></td>
</tr>
<tr>
<td>Synergy D6</td>
<td>SD6</td>
<td>Coltène/Whaledent, Altstätten, Switzerland</td>
<td></td>
</tr>
<tr>
<td>Filtek Z250</td>
<td>FZ250</td>
<td>3M, St. Paul, MN, USA</td>
<td>microhybrid</td>
</tr>
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<td>Quadrant</td>
<td>Quadrant</td>
<td>Cavex, Haarlem, the Netherlands</td>
<td></td>
</tr>
<tr>
<td>Admira</td>
<td>ADM</td>
<td>VOCO, Cuxhaven, Germany</td>
<td></td>
</tr>
</tbody>
</table>

METHODS
A total of 320 composite discs with a diameter of 10 mm and a height of 2 mm were prepared (Figure 1).

Once prepared, all discs were polished using a Hitech Europe polisher equipped with a silicon carbide polishing disc with a grain size of 600 at 150 rpm for 30 seconds. They were subsequently polished with clinical counter-angle polishing discs for a further 30 seconds. All discs were then placed in distilled water at room temperature for 48 hours before being divided into two groups:

- Group 1 (n=160): the initial colour was determined using a spectrophotometer and the samples placed directly into the colouring solutions such that each group of composites (n=20) was sub-divided into 4 sub-groups (n=5): 1) distilled water (control); 2) coca cola, 3) coffee and 4) red wine (Figure 2). Samples were stored in an incubator at 37 ºC (± 1 ºC) throughout the study period. The liquids were changed every 2-3 days and fungal growth was monitored to prevent its appearance, (Figure 3).
- Group 2 (n=160): samples were thermally cycled for 5000 cycles (5 ºC/55 ºC) and, once this ageing process was complete, the initial colour was measured and the discs immersed in the colouring solutions as described above.

**RESULTS:**

![Interaction scheme](image1.png)

The relationship between composite type and colouring solution studied is shown in Figure 3. A very similar behaviour was observed, with red wine and coffee causing the most discolouration and coca cola and distilled water the least, although coca cola was found to discolour the GR composite much more than the other composites.

![Interaction scheme](image2.png)

Figure 3. Composite-colourant interaction.

Figure 4. Interaction with thermally cycled composites.
Figure 4 shows the interaction between the composites and the thermal cycling treatment. In general, the values observed are higher for all groups after the ageing process, except for SD6 and VTN, where the values are not affected by ageing. GR exhibits the highest values for the non-thermally cycled group.

**CONCLUSIONS:**

- Red wine has been shown to produce the highest degree of discolouration, followed by coffee, coca cola and distilled water, the latter of which was used as a control.
- The Ventura Nanolux composite exhibits a very similar staining behaviour to Ceram X, Tetric EvoCeram, Admira, Filtek Z250 and Quadrant, all of which exhibit a stable and uniform behaviour.
- All composites except Sinergy D6 and Ventura Nanolux exhibit greater staining after the ageing treatment, with the latter two exhibiting similar values.
AN IN VITRO STUDY OF COMPOSITES: FLEXURE

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INTRODUCTION
The use of aesthetic materials for restorations in the posterior sector has increased significantly over the past few years. As a result, various new materials with varying chemical compositions for the organic matrix and different particle sizes, such as micro- and nanohybrid, nanofilled, ormocer and silorane composites, have been developed.

The choice of composite for use in a reconstruction currently requires an evaluation of the characteristics and properties of the following aspects of the different materials available: 1) at a functional level, their ageing behaviour; 2) their mechanical properties, which must be excellent; 3) their optical and aesthetic properties; and 4) their biocompatibility.

The clinical behaviour of these materials depends on their mechanical properties. The main causes of restoration failure in the 1970s and 1980s were lack of wear resistance, loss of anatomical shape and proximal contacts, and degradation of the restoration. An important property related to the above is the so-called flexural strength, which simultaneously measures the stress of compression and shear forces. Indeed, this is one of the in vitro tests that allows us to determine the resistance of the material to chewing forces (5) and is one of the tests included in the ISO 4049 standard for polymer-based filling, restorative and luting materials.

It has been observed that one of the factors most closely related to the mechanical properties during chewing is the percentage of inorganic filler contained in the composite (8). Similarly, various studies have demonstrated the influence of particle size and shape on the mechanical properties of composites.

As a result of intensive research efforts in the field of dental composites, there is now a wide range of direct restoration materials, such as microhybrid, nanohybrid, nanofilled, silorane, ormocer and compomer composites. The differences between these composites reside in the composition of the organic matrix, the size, shape and composition of the filler and the chemical agents that bind these two components together (3,9), all of which mean that these materials exhibit different mechanical and chemical behaviours when subjected to ageing processes.

Thermal cycling is one of the most widely used ageing processes in in vitro studies. This technique simulates the in vivo ageing of materials by subjecting them to repeated cycles at different temperatures (hot and cold) and, in general, shows that the physical properties of the materials worsen.

AIM
To evaluate the flexural strength of 8 composites (5 nanohybrid, 2 microhybrid and 1 ormocer) subjected to a thermal cycling-based ageing process and to compare the results with those obtained for their untreated counterparts. 3
MATERIALS

Composites

<table>
<thead>
<tr>
<th>composite</th>
<th>study code</th>
<th>manufacturer</th>
<th>type</th>
</tr>
</thead>
<tbody>
<tr>
<td>Ventura Nanolux</td>
<td>VTN</td>
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</tr>
<tr>
<td>Grandio</td>
<td>GR</td>
<td>VOCO, Cuxhaven, Germany</td>
<td></td>
</tr>
<tr>
<td>Ceram X</td>
<td>CX</td>
<td>Dentsply, Konstanz, Germany</td>
<td>nanohybrid</td>
</tr>
<tr>
<td>Tetric EvoCeram</td>
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<td>Ivoclar Vivadent, Schaan, Liechtenstein</td>
<td></td>
</tr>
<tr>
<td>Synergy D6</td>
<td>SD6</td>
<td>Coltène/Whaledent, Altstätten, Switzerland</td>
<td></td>
</tr>
<tr>
<td>FIltex Z250</td>
<td>FZ250</td>
<td>3M, St. Paul, MN, USA</td>
<td>microhybrid</td>
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<tr>
<td>Quadrant</td>
<td>Quadrant</td>
<td>Cavex, Haarlem, the Netherlands</td>
<td></td>
</tr>
<tr>
<td>Admira</td>
<td>ADM</td>
<td>VOCO, Cuxhaven, Germany</td>
<td>ormocer</td>
</tr>
</tbody>
</table>

METHODS

A total of 160 composite bars measuring (25±2) mm x (2±0.1) mm x (2±0.1) mm were prepared using a mould manufactured according to the instructions set out in the ISO 4049 standard (6) (Figure 1 B). A glass plate was placed on top of these samples and they were polymerised for 40 seconds, using an LED lamp, at three different points, namely the two ends and the centre of the bar.

Upon completion of this process, the samples were polished using 600-grain silicon carbide discs to remove excess material and burrs, then stored in an oven at 37 (±1) ºC for 24 hours. After this time the samples were divided into two groups:

- Group 1 (n=80): 10 bars for each composite were immediately subjected to the flexural test.

- Group 2 (n=80): samples (10 for each composite) were thermally cycled in water at 5 ºC/55 ºC (±1ºC) for 5000 cycles, remaining at each temperature for 30 seconds. They were then subjected to the flexural test.

Flexural test:

Samples were placed on a Quasar 5 universal tester at a constant rate of (0.75±0.25) mm/min.
The flexural strength is measured in MPa.

RESULTS.
A multi-factor ANOVA showed statistically significant differences between the variables studied (composite and thermal cycling) and the interactions between them.

It can be seen from Figure 2 that the Filtek Z 250 composite exhibits the highest flexural strength, followed by Ceram X and Grandio. The Admira and Quadrant composites have the lowest flexural strengths. The remainder (Tetric EvoCeram, Synergy D6 and Ventura Nanolux) exhibit an intermediate behaviour in terms of flexural strength.

Figure 3 shows that the samples which were not submitted to the ageing process exhibit higher flexural strengths than those that underwent this process, thus meaning that the aged samples fractured sooner.
CONCLUSIONS
The Ventura Nanolux composite does not present statistically significant differences with respect to the nanohybrid samples (Ceram X, Synergy D6, Grandio, Tetric EvoCeram) in terms of flexural strength.

All composites exhibit higher flexural strengths if not submitted to an ageing treatment (thermal cycling).
**IN-VITRO COMPOSITES STUDY. FINAL REPORT. MICROTENSILE BOND STRENGTH**

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**Introduction**
Adhesive dentistry has had a rapid development in recent years. Among the advantages of adhesive techniques that enable states to recover the missing tooth anatomy and various functions performed by the teeth. In addition, some of the biomaterials that are used with adhesive techniques like composite resins achieve the aesthetic parameters required by patients. The ideal material from the point of view of its adhesion to the tooth structure is one that achieves primary chemical bonding to the tooth. The achievement of such a union would avoid the presence of interface and thus circumvented the problems that cause this critical area in dentistry today. In the case of composites, they achieve in the enamel, mainly micromechanical joints due to the implementation of the total etching technique and the use of adhesives in the hard tissues of the tooth.

The total etching technique at the level of the dentin has not yielded results similar to those achieved in enamel due to its composition with a higher proportion of organic material and water. Add to this the formation of a smear layer during etching acid that acts as a barrier to the adhesive directly to a dentin. Research in this area seeks to achieve a dentin adhesive capable to reach high adhesion values to the mineral and the organic component of dentin.

Currently, these adhesives achieve the formation of a hybrid layer by preparing dentin through a primer and then the placement of an adhesive that provides a surface compatible with the restorative material.

Some of the current adhesives fail to make these two steps (preparation of dentin and adhesive placement) in a single step. In general the vehicle of these components has been a solvent such ethanol or acetone. The main function of this vehicle is to give greater fluidity to the adhesive, allowing more humectancy. It have appeared on the market some solvent free adhesives who, according to the manufacturer, improve the clinical application of the product while maintaining the adhesion values of single component adhesives with solvent.

The advantage of using these kind of adhesives would be facilitate the clinical process of dentin bonding, to prevent over blowing on the adhesive and dentine before curing, reducing further the possibility of drying dentin, thereby improving the adhesion values of fillings made with this type of adhesive.
Objectives
Main objective
- The aim of this study is to evaluate the values of microtensile bond strength in different types of composite resins using a one solvent free single component adhesive.

Specific objectives
- Measure and compare the values of microtensile bond strength in different types of composites using a solvent free single component adhesive.

Materials
Samples will consist in forty eight (n=48) extracted third human molars free of any alteration in its anatomical integrity, which are free of caries and filling materials of any kind. During the study period will be stored hydrated in distilled water at room temperature.

The test adhesive will be Unibond2 Ventura, single component solvent free adhesive manufactured by Madespa (Toledo, Spain).

For the control group will be used the solvent component adhesive Adper Scotchbond1 XT manufactures by Kerr.

37% phosphoric acid will be used.

<table>
<thead>
<tr>
<th>Product name</th>
<th>Group</th>
<th>Type</th>
<th>Manufacturer</th>
</tr>
</thead>
<tbody>
<tr>
<td>Admira</td>
<td>1</td>
<td>Nanoceramic</td>
<td>VOCO (Cuxhaven, Germany)</td>
</tr>
<tr>
<td>Grandio</td>
<td>2</td>
<td>Nanohybrid</td>
<td>VOCO (Cuxhaven, Germany)</td>
</tr>
<tr>
<td>Nanolux, Ventura</td>
<td>3</td>
<td>Nanohybrid</td>
<td>Madespa (Toledo, Spain)</td>
</tr>
<tr>
<td>Ceram X</td>
<td>4</td>
<td>Nanoceramic</td>
<td>Dentsply (Konstanz, Germany)</td>
</tr>
<tr>
<td>Tetric Evoceram</td>
<td>5</td>
<td>Nanohybrid</td>
<td>Ivoclar Vivadent (Schaan, Liechtenstein)</td>
</tr>
<tr>
<td>Filtek Z 250</td>
<td>6</td>
<td>Microhybrid</td>
<td>3M (St.Paul,MN, EEUU)</td>
</tr>
<tr>
<td>Synergy D6</td>
<td>7</td>
<td>Nanohybrid</td>
<td>Coltène Whaledent (Altstätten, Switzerland)</td>
</tr>
<tr>
<td>Quadrant</td>
<td>8</td>
<td>Hybrid</td>
<td>Cavex (Haarlem, The Netherlands)</td>
</tr>
</tbody>
</table>

Table 1: Composites used in this study.

Method
All Teeth will be mounted in acrylic resin to facilitate the preparation of the samples. It deletes any occlusal surface by cutting the tooth with the cutting machine Isomet 100 and wear down after sanding with 180 grit discs under a constant stream of water until a flat dentin surface parallel to the occlusal surface.

The molar preparations are kept at room temperature and divided randomly into eight experimental groups of six (n = 6) teeth each. Each of the eight (8) of the groups are divided in turn into an experimental group and a control group of 3 teeth each (n = 3) to test different types of composite research.

In each of the groups, teeth are prepared following total acid etching with phosphoric acid 37% (Ventura gel conditioner, Madespa, Toledo, Spain), applies for 15 seconds, remove and wash for 15 seconds with water. Subsequently placed the single component solvent free adhesive. (Ventura Unibond 2) for 20 seconds wetting all surfaces and light cured for 30 seconds with a curing lamp, after that it will be made the build-up with composite resin for a composite block of 5 mm high, using the incremental technique and following the manufacturer's instructions.

In the case of controls prepared teeth with total etching with phosphoric acid 37% (Ventura gel conditioner), applies for 15 seconds, remove and wash for 15 seconds with water. Subsequently placed the single component adhesive with solvent Adper Scotchbond1 XT, for 15 seconds wetting all surfaces, will be extended with clean dry air for three seconds and then
light cure for 20 seconds with the lamp. After that it will be made the build-up with composite resin for a composite block of 5 mm high, using the incremental technique and following the manufacturer's instructions.

All samples will be stored in distilled water at room temperature before making the blocks for stress testing.
All restored teeth are both longitudinally sectioned (Figure 1) in the "x" and "and" directions across the bonded interface using a diamond saw in an 1000 machine to obtain Isomet sticks, Each with a cross-sectional area and with 1mm2 approximately 5 mm of composite and 5 mm of dentin tissue. (Following Pashley method) (19).

The two groups and their corresponding experimental and control subgroups will be formed as follows:

Group A: Adhesive Ventura Unibond2
- A2: Grandio.
- A3: Ventura Nanolux.
- A4: CeramX.
- A5: Tetric EvoCeram.
- A6: Filtek Z 250.
- A7: Sinergy D6.
- A8: Quadrant.

Group B: Adhesive Adper Scotchobond1 XT.
- B1: Admira.
- B2: Grandio.
- B3: Ventura Nanolux.
- B4: CeramX.
- B5: Tetric EvoCeram.
- B6: Filtek Z 250.
- B7: Sinergy D6.
- B8: Quadrant.
Results by composites

Fig 3. Mean values of microtensile bond strength for each group

Results by adhesive

Conclusions
Ventura Unibond 2 Adhesive performed having better values compared Adper Scotchbond1 XT with all composites groups used in this study.
In nanohybrid groups Sinergy D6 (nanohybrid) with Unibond2 had the highest value and Tetric Evo Ceram with Adper Schotbond1 Xr had the lowest value. Quadrant (microhybrid) with Adper Schotbond1 XT had the lowest values among all groups.
The use of the free solvent adhesive system (Unibond2) can influence the adhesive values using different types of composites systems. In this case highest values were found.
Introduction
A smooth surface finish of resin composites is highly valuable in achieving a good restoration, regardless of the location or type of the cavity to which the restoration is applied. Surface irregularities, which may be brought about by the composite itself, or the manipulation of materials, may bring about clinical problems such as gingival irritation, plaque accumulation, staining, bacterial adhesion and even secondary caries on the margins of the said restoration. Furthermore, rough restorations may cause patient discomfort, and may even cause premature wear on the enamel that is in contact with the rough restoration. Surface Roughness properties is a manifestation of an interaction of different factors such as the filler of the composites used (the size, shape, type and distribution of the particles), the type of the resin matrix, the degree of polymerization and the bond efficiency of the filler-matrix interface. Other factors that may affect surface roughness may be the type of polishing system employed during treatment, flexibility of the material in which the abrasives are embedded, the geometry of the instruments used to manipulate the composites and the hardness of the abrasives used during polishing.
In this line, a lot of studies have been conducted in the hopes of coming up with better ways of analyzing the surface roughness of composites, whether the goal is to compare the available composites in the market: packable or flowable, according to the filler size of the composites such as nanofilled, nanohybrid or microhybrid, or may be between dimethacrylate based composites, cationic or organic modified ceramics or other available composites in their different categories. Different polishing systems may also be evaluated: the single step type that employs the use of the fine diamond polishing system or multiple step polishing systems available like aluminum oxide abrasives, the silicone multigrit system discs, and finishing foam wheel impregnated UDMA among the many other polishing systems available. These, along with the use of polishing pastes may also be evaluated in connection to its effect on the surface roughness of composites. The effect of the duration in doing polishing procedures on surface roughness may also be evaluated by varying the time set for polishing a certain composite. Previous studies have evaluated surface roughness qualitatively through optical or scanning electron microscopy or quantitatively with the use of a stylus contact profilometry machine, or both qualitatively and quantitatively with the use of a non-contact 3D optical profilometry machine.
The aim of this study was to compare surface roughness of various composites after carrying out a uniform method of polishing, and employing the use of a non-contact 3D optical profilometry machine.

Objectives
1. General Objective
   - To evaluate the surface roughness values (Ra) of various dental resin composites.

2. Specific objectives
   - To evaluate whether there is a significant difference of surface roughness values between the different types of composites using the same polishing procedure.
   - To compare the differences in surface roughness values between different Nanohybrid, Microhybrid and Ormocer type of composite.

Materials and Methods
1. Samples preparation:

   Composite discs were made with a metal mold, 10mm in diameter and 3mm in depth, on top of a glass slab. Molds were packed with composite to a slight excess and covered with another glass slab (Figure 1). A total number of 35 specimens were fabricated; specifications of each are shown on Table 1. Through the glass slabs for 20 seconds each on both sides. Samples were pre-ground flat on wet 600-grit SiC disc with slight pressure for 10 seconds on each side by a single operator, using a polishing machine. Samples were washed to remove any debris created from the pre-polishing procedure and then stored in water and kept at a temperature of 37°C for 24 hours and were polished with a 1200-grit SiC polishing disc with slight pressure for 10 seconds on each side.

   ![Composites discs samples preparation](image)

   **Fig 1.** Composites discs samples preparation

   Samples were washed before storage at 37°C until the first analysis of surface roughness which was carried out after 24 hours.
### Table 1. Various Composites used in the study, Filler Particle Size and Filler weight and volume and their respective manufacturers.

<table>
<thead>
<tr>
<th>Composite</th>
<th>Type</th>
<th>Filler wt./ vol.</th>
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<tr>
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<td>Madespa S.A., Toledo</td>
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<td>87/71.4</td>
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<td>Synergy D6</td>
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<td>Nanohybrid</td>
<td>76, 57</td>
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<td>Admira</td>
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<td>77, 61</td>
<td>VOCO, Cuxhaven, Germany</td>
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<td>Filtek Z250</td>
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<td>82, 60</td>
<td>3M, St. Paul MN, U.S.A.</td>
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<tr>
<td>Tetric Evoceram</td>
<td>Nanohybrid</td>
<td>82.5/68</td>
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#### 2. Surface Roughness Analysis:
After the 24h period of water storage, surface roughness analysis using a Leica Dual Core 3D non-contact profilometer machine was started. One reading at the center of each sample was taken note of using the Leica scan software version 3.2 with a confocal objective magnification of 150x. This phase and analysis were common to all groups. The parameter of surface roughness, which is the Ra Value, was recorded as representative value for each specimen.

#### Results

![Means and 95.0 Percent LSD Intervals](image)

Figure 2. Mean values of the different Ra values of 7 composites.
Conclusions
Based on the results and statistical analysis, the following may be stated about the study:
1. There are no significant statistical differences between the surface roughness values obtained between the different groups of nanohybrid, microhybrid and organic modified ceramic composites.

2. Nanohybrid Ventura Nanolux, showed no significant differences on surface roughness between the other nanohybrid groups, in other hand, a tendency for lower values on surface roughness was showed compared with Sinergy and Grandio nanohybrid resin composites.

Fig 3. Mean Ra Values of the different composites used in this study.