Comparative Evaluation of Hardness and Elasticity Modulus of Tooth-Colored Materials for Dental Restoration

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The aim of this study was to compare Rockwell hardness (HRC) and modulus of elasticity (Young modulus) to different restorative materials. Three commercial composite resins: Filtek Z250 (3M ESPE Co.), Zmack Comp (Zermack SpA, Italy), Kalore (GC Corporation, Japan) and one compomer: Dyract eXtra (Dentsply De Trey Gmbh, Germany) were used. Six samples of each material were obtained by placing them in plastic rings having 5 mm inner diameter and 6 mm high. All composite samples were cured for 40 s using a lamp LEDidition - Ivoclaire Vivadent clinical, Austria. The samples were finished and polished and then stored in distilled water, at room temperature for 48 hours. Rockwell Hardness (HRC) measurements were realized using the UMT-2 Tribometer (CETR). The hardness was automatically calculated from the slope of unloading curve and expressed in GPa and transformed in HRC values (kgf/mm^2). Six indentations were performed on diametral direction and mean values were calculated for all tested samples. The device also measured the modulus of elasticity for each sample. Data were analyzed by ANOVA and Mann-Whitney test (significance level of $p < 0.05$). The mean HRC values were the following: Filtek Z250 > Zmack Comp > Dyract eXtra > Kalore (82.98 > 70.10 > 53.27 > 37.72 kg/mm^2). Regarding Young modulus, the rank from highest to lowest as follows: FiltekZ250 > Zmack Comp > Dyract eXtra > Kalore (16.24 > 14.05 > 12.41 > 7.86). The microhydrid composite resins have a significantly higher hardness than nano-hybrid composites or compomers.

Keywords: Rockwell hardness, Young modulus, Mann-Whitney test, resin composite, compomer

In the oral environment, restorative materials are exposed to chemical, thermal, and mechanical challenges. These challenges can cause deforma-tion of the material. Dental composites are expected to have mechanical properties comparable to those of tooth enamel and dentin and provide a long life of service [1, 2]. Since their introduction, composite resins have been continuously developed in an attempt to improve their properties and broaden their clinical applications. The properties of composite resins can be altered by variations in composition and amount of resin matrix, as well as size and distribution of filler particles [3-12]. The greatest inorganic filler content is present in traditional minifilled composites with the intention of increasing hardness and wear resistance [13].

Surface hardness is one of the most important properties used to compare restorative materials, and is defined as the resistance to permanent indentation or penetration [14]. It is a mechanical property of the restorations that should always be taken into account, especially when they are faced with large areas of masticatory force [15, 16].

Substantial surface microhardness of the restoration is one of the main requirements especially in posterior stress-bearing areas. One of the most important factors that affect dental restoration is that it undergoes wear during function or whereas being cleaned [17]. As wear is due to abrasion, surface hardness is an essential property. It is the mechanical property most frequently used to characterize the wear resistance of materials. A material that have a higher surface hardness, in general, considered to be more wear resistant [18-21].

The Rockwell hardness test was developed as a rapid method for hardness determinations. A ball or metal cone indenter is normally used, and the depth of the indentation is measured with a sensitive dial micrometer. The superficial Rockwell method has been used to test plastics used in dentistry [22].

In this study we used the Rock-well hardness test because has the advantage that hardness is read directly and it is good for testing viscoelasticity of materials. The aim of this study was to compare Rockwell hardness and modulus of elasticity to different restorative materials.

Experimental part

Materials and methods

Three commercial composite resins: Filtek Z250 (3M ESPE Co.), Zmack Comp (Zermack SpA, Italy), Kalore (GC Corporation, Japan) and one compomer: Dyract eXtra (Dentsply De Trey Gmbh Germany) were used for this study. The composition of the materials used in this study is in the table 1. Six samples of each material type having 5 mm in diameter and 6 mm deep, were obtained by condensed them in plastic rings having 5 mm inner diameter and 6 mm high. All composite samples were cured for 40 s using a lamp LEDidition - Ivoclaire Vivadent clinical, Austria. The samples were finished and polished and then stored in distilled water, at room temperature for 48 h.

Rockwell Hardness (HRC) measurements were realized using the UMT-2 Tribometer (CETR) illustrated in figure 1a and b. Small cylindrical specimens were fixed on the linear table of tribometer and indented with a normal force of 10 N for 30 s. The tests were made by first applying a preload of 1 N for 15 s. A cone indenter with a radius of 200 μm was pressed on the samples surface with an indentation velocity of 0.005 mm/s. The depth of penetration is recorded by a capacitive sensor. A typical load-depth indentation curve can be observed in figure 2a, b, c and d. The hardness is

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Materials	Category	Compositions	Manufacturer
Filtek Z250	Universal microhybrid composite	Bis-GMA, Bis-EMA, UDMA (0.01-3.5 μm zirconium/silica filler) 60% by volume	3M ESPE Dental Products, St.Paul, USA
Zmack Comp	Universal microhybrid composite	Dymethacrylate resin (EBDADMA), TEGDMA, photo initiators, stabilizer, fillers: barium-aluminium-borosilicate <1.5 μm; highly dispersed silicon dioxide 0.04 μm; iron oxide pigments; titanium oxide (57%)	Zermack SpA, Italy
Dyract eXtra	Compomer	Ethoxylated Bisphenol-A dimethacrylate, urethane resin, triethylene glycol dimethacrylate (TEGDMA), and trimethylolpropane trimethacrylate (TMPTMA); strontium fluorideglass 0.8 μm	Dentsply De Trey Gmbh Germany
Kalore	Nano-hybrid composite	Urethane dimethacrylate, DX-511 co-monomers, dimethracrylate (18%), Fillers: fluoroaluminosilicate glass, pre-polymerized filler (400 nm nano-sized modified strontium glass, 100 nm lanthanoid fluoride), silicon dioxide (82%); photo initiator <1%, pigment ,1%	GC Corporation, Japan

Table 1
THE COMPOSITION OF THE RESTORATIVE MATERIALS USED IN STUDY

automatically calculated from the slope of unloading curve and expressed in GPa and transformed in HRC values (kgf/mm^2). Six indentations were performed on diametral direction and mean values were calculated for all tested samples. The device also measured the modulus of elasticity for each sample. Data were analyzed by ANOVA and Mann-Whitney test (significance level of $p < 0.05$).

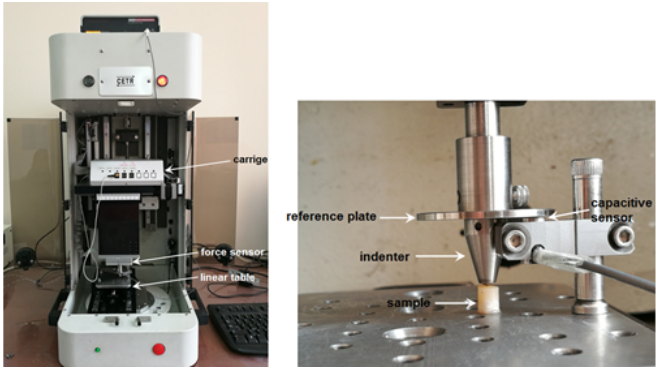
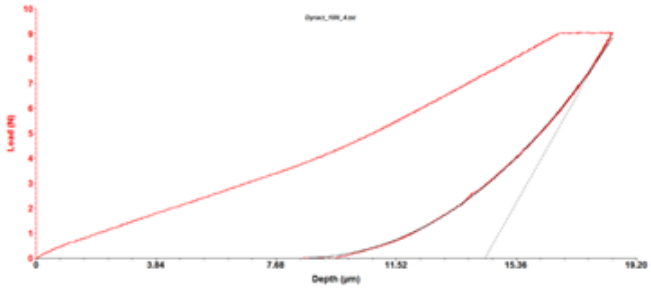
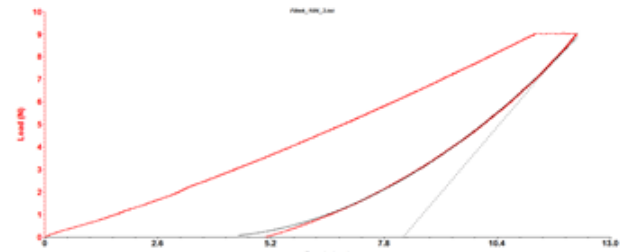


Fig. 1. UMT-2 Tribometer: a) general view of equipment; b) indentation test for hardness measurements



a.

Fig. 2. Hardness Rockwell curves: a. Dyract eXtra



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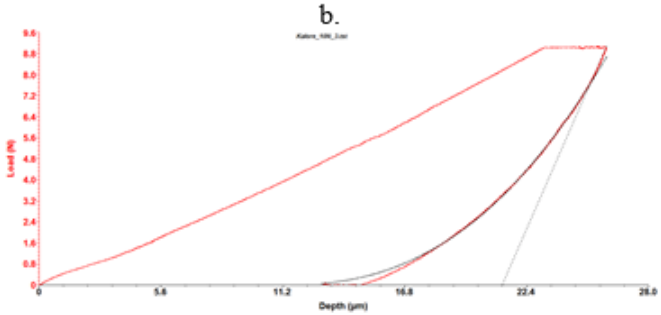
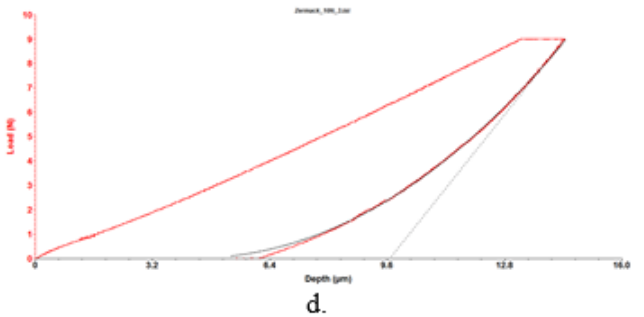


c.



d.

Fig. 2. Hardness Rockwell curves: b. Filtek Z 250; c. Kalore; d. Zmack Comp)

Results and discussions

The mean HRC values and standard deviation recorded for all the samples are presented in table 2. The surface hardness of composite microhybrid Filtek Z250 was highest than composite microhybrid Zmack Comp (82.98 kg/mm^2 > 70.10 kg/mm^2). This is explained by the different composition in fillers to microhybrid composite resins. Thus, Filtek Z250 has zirconium particles that are much tougher than barium, aluminium and silicate from Zmack Comp compositions. The compomer Dyract eXtra having in filler composition strontium fluoride glass, has an average HRC 53.27 kg/mm^2, less than two composite microhybride, but higher than the composite nanohybride. The composite Kalore exhibited the lowest mean HRC (35.77 kg/mm^2), which would also be explained by the chemical composition of the fillers.

Table 2
MEAN HRC VALUES RECORDED TO SAMPLES

MATERIALS	HRC	
	Mean	Std. Deviation
DYRACT EXTRA	53.27827	0.816679
FILTEK Z 250	82.98494	0.914143
KALORE	35.72278	1.528646
ZMACK COMP	70.10297	1.729941

Table 3
MEAN SCORSE YOUNG MODULUS TO SAMPLE

MATERIALS	Young Modulus	
	Mean	Std. Deviation
DYRACT EXTRA	12.41306	0.367624
FILTEK Z 250	16.24694	0.879870
KALORE	7.86333	0.084013
ZMACK COMP	14.05167	0.157284

The mean Young's modulus values and standard deviation recorded for all the samples are presented in table 3. We observe statistically significantly higher values for microhybride composites compared to nanohybrid composite and compomer.

The modulus of elasticity represents the stiffness of the material when subjected to a compressive force. The interatomic or intermolecular forces of the material are respon-sible for the property of elasticity [23]. The composite Filtek Z250 exhibited the highest average Young's modulus (16.24 GPa), followed by Zmack Comp (14.05 GPa), Dyract eXtra (12.41 GPa) and Kalore (7.86 GPa).

These results can be explained by fact that the size and volume of the filler from materials used differ greatly. Thus, as the density of the filler is higher, the modulus of elasticity will be lower. Also, the size of fillers can influence the Young's modulus [24].

Mann-Whitney test indicated that the HRC and Young's modulus mean values of the all materials were significantly different from each other ($p < 0.0001$).

Hardness is therefore a measure of the resistance to plastic deformation and is measured as a force per unit area of indentation. Hardness influences ease of cutting, finishing, and polishing an object and its resistance to in-service scratch-ing. Finishing or polishing a structure is important for esthetical purposes and, as discussed previously, scratches can compromise fatigue strength and lead to premature failure [25, 26].

The fillers are made of quartz, ceramic and or silica. With increasing filler content the polymerisation shrinkage, the linear expansion coefficient and water absorption are reduced. On the other hand, with increasing filler content, the compressive and tensile strength, the modulus of elasticity and wear resistance are generally increased [27]. The filler content of a composite is sometimes determined by the shape of the filler. In a study with different types of composite, those materials with pre-polymerised composite fillers were shown to have the lowest filler content and thus also the lowest flexural strength and hardness. Composites with round fillers had the highest filler content, which was associated with higher hardness and high flexural strength. For mixed filler particles (hybrid composites) there was no linear relationship between filler content and flexural strength [28].

In one study of 72 restorative materials it was also shown that filler volume had a significant influence on the mechanical properties. The relationship between filler content, flexural strength and modulus of elasticity was most obvious [29].

Nanofillers and nanoclusters enhance the long-term stability and the polishing properties of micro-filler composites are made possible by the use of nanoparticles and nanoclusters. The mechanical stability achieved in hybrid composites is due to larger filler particles or *nanoclusters*. Superficial filler particles are lost due to abrasion. The nanoclusters of the nanocomposites are hereby broken down into nanoparticles. These particles are smaller than the wavelengths of visible light. It has been shown that surface polish is preserved longer after wear tests in composites with filler particles < 0.4 μm [30]. Nanoparticles can be incorporated into cells but their toxic potential is still largely unknown [31].

Compomers are composed of composite and glassionomer components. It is an attempt to take advantage of the desirable qualities of both materials: the

		Dyract Extra	Filtek Z 250	Kalore	Zmack Comp
Dyract Extra	HRC	-	0.0001	0.0001	0.0001
	YOUNG MODULUS	-	0.0001	0.0001	0.0001
Filtek Z 250	HRC	0.0001	-	0.0001	0.0001
	YOUNG MODULUS	0.0001	-	0.0001	0.0001
Kalore	HRC	0.0001	0.0001	-	0.0001
	YOUNG MODULUS	0.0001	0.0001	-	0.0001
Zmack Comp	HRC	0.0001	0.0001	0.0001	-
	YOUNG MODULUS	0.0001	0.0001	0.0001	-

**The mean difference is significant at the 0.05 level*

Table 4
MANN-WHITNEY STATISTICAL TEST RESULTS WHEN COMPARED HRC, YOUNG MODULUS TO SAMPLES

fluoride release and ease of use of the glassionomers and the superior material qualities and aesthetics of the composites. In addition to the various polymerizable monomers (e.g. UDMA) the material also contains dicarboxylic acids, which in contrast to those in traditional glassionomers have polymerisable double bonds. The reactive fluoroaluminiumsilicate glasses from the glassionomer technology are found in compomers. The particle size of fillers in these products varies from 0.2μm up to 10 μm [32]. Since the compomer has much lower filler contents of a different type they produce lower surface microhardness [33].

A comprehensive study on the physical properties of different restorative materials (flexural strength, compressive strength and tensile strength) was able to demonstrate considerable differences in the same material group. The filler content had the greatest influence on the material properties [29].

The most current composites are filled with silicate particles based on oxides of barium, strontium, zinc, aluminium or zirconium. The concentration rate of filler is generally 70-80% by Weight. The particle filler size is in the range from 0.04 to 85 μm [34]. The primary purpose of the filler particles is to increase the strength of composite and to decrease the amount of matrix material, resulting in increased hardness, decrease wear and reduction in polymerization shrinkage [35]. The filler content, filler size, morphology, and the distribution of filler particles influence the physical and mechanical properties of composite resin and many studies reported the relation between filler and flexural strength, compressive strength, diametral tensile strength, shear punch strength, fracture toughness, hardness, wear, shrinkage stress and thermal expansion [36-38].

Of great importance in the hardness of composite materials is the size and volume of the inorganic particles [39]. Thus, in the Filtek Z250 composite, particle size is 0.01-3.5 μm, 60% vol, higher than Zmack Comp (1.5 μm, 57% vol), and Kalore (400 nm, 80% vol). Our results confirmed this, namely that the hardness of the materials and the modulus of elasticity are directly proportional to the size of the fillets and their volume.

Decreasing filler particle sizes is model of better dispersion and increased interfacial area between matrix and filler. This can be translated into increased flexural strength, surface microhardness, and polishability of the finished restoration [40].

Podariu et all. [41] suggested that characteristics of the fillers particles such as size, texture, shape, the amount and the distribution mode in resin composite mass, can influence the properties of composite materials. They demonstrated that the percentage and the type of inorganic filler can modified the parameters of nano-composite materials.

On the other hand, the hardness of resin composite can be affected by acid attack of beverage [42]. The composite resins suffer a softening of the surface layer under the acids action, due to the changes of the organic component [43]. Wongkhantee, S. et all. [44] observed that organic acids induce the dissolution of BIS-GMA.

Kawai, K. et all. [45] suggested that for improving the wear resis-tance of composites is to increase the abrasive resistance of the resin matrix, rather than increasing in the hardness of the filler particles. The predominant base monomer used in commercial dental composites has been bis-GMA, which due to its high viscosity is mixed with other dimethacrylates, such as TEGDMA. UDMA corresponds to another alternative organic matrix composition and it is often pres-ent in current compositions, can be considered as increasing the resistance for resin composite.

Conclusions

In this study the results were presents higher values of the Rockwell hardness and Young‘s modulus at the microhybrides composites compared with nanohybrides composites and compomer. It is necessary to investigate in other studies the flexural strength, conversion and flexural modulus, to improve the understanding of mechanical properties of tooth-colored materials.

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