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Hardness investigation of conventional, bulk fill and flowable dental composites

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ABSTRACT

Purpose: of the present paper is to investigate the micro-hardness of three types of resin-based composites – conventional, bulk fill and flowable.

Design/methodology/approach: Cylindrical specimens with a diameter of 5 mm and thicknesses of 2, 3 and 4 mm were made from each composite. They were light cured for 20, 40 and 60 s with light intensity of 600, 1000 or 1500 mW/cm^2. The Vickers micro-hardness was measured on the top and bottom surface of the specimens.

Findings: The highest micro-hardness was measured in bulk fill composite, followed by conventional and the lowest was measured in the flowable one. Increasing the light intensity leads to increase of the micro-hardness on both surfaces of the three composites. The increase of the irradiation time results in increase of the micro-hardness mainly on the bottom surface of the composites. The change of the layer thickness influences the conventional and the flowable composites and almost does not affect the hardness of the bulk fill composite.

Research limitations/implications: The limitations of this study concerns to the values of the light intensity, which are defined by the light curing unit (LCU) used. There are many LCUs on the market; consequently, constant investigations of dental composites micro-hardness are needed.

Practical implications: The investigation of the micro-hardness of the three types of composites in different modes would be very helpful for clinicians to obtain successful polymerization of composite restorations in their everyday practice.

Originality/value: The micro-hardness of three types resin-based dental composites – conventional, bulk fill and flowable is investigated and compared in varying of three mode parameters – light intensity, curing time and layer thickness.

Keywords: Resin-based composites, Micro-hardness, Light intensity, Irradiation time, Layer thickness

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BIOMEDICAL AND DENTAL ENGINEERING AND MATERIALS

1. Introduction

Dental resin-based composites (RBCs) are the most preferable materials for direct restorations of tooth defects nowadays due to their high aesthetic and easy application [1,2]. The beginning of the modern composites can be attributed to 1962 when the Bis-GMA (bisphenol A glycydil dimethacrylate) monomer was developed by R. Bowen [3-5]. The first composite, containing Bis-GMA in its composition, was created one year later. The newly developed products have significant advantages over the materials used so far for direct restorations – silicate cements and self-cured polymers: less polymerization shrinkage and reduced toxicity. However, the problems related to marginal adaptation, discoloration and poor wear resistance have not yet been fully resolved. For this reason, most clinicians in the 1970s and 1980s used RBCs to restore Class III, IV and V cavities, and amalgam fillings were the first choice for posterior defects. With the improvement of RBCs over the years, their use has become more widespread and they gradually replace the amalgam, even when it is necessary to restore posterior teeth.

The main components of RBCs include organic matrix, inorganic part – fillers, coupling agents and initiators of the polymerization process [3-5]. The organic matrix consists mainly of the bifunctional monomers Bis-GMA and UDMA (urethane dimethacrylate). Due to their high viscosity, TEGDMA (triethylene glycol methacrylate) is used as a diluent. Bis-EMA (bisphenol A polyethethylene glycol dimethacrylate) is added to improve handling properties and reduce polymerization shrinkage. Fillers are the inorganic component of RBCs and consist mainly of glass particles - borosilicate, barium glass, strontium glass, silicon dioxide, etc. Depending on the particles size, the composites are classified as macrophilled, microphilled, nanophilled and hybrid (microhybrid and nanohybrid) [6-8]. The type and quantity of the fillers determine the composite type – flowable, compactable, bulk fill and its properties – hardness, wear resistance, polishability, radiopacity, viscoelasticity and polymerization shrinkage. Coupling agents (silanes) are usually bipolar compounds which are intended to provide a strong and stable chemical bond between the organic matrix and the inorganic fillers [4,6,7]. Two types of initiators are mainly used in RBCs – benzoyl peroxide in self-cured composites and most often camphoroquinone in light-cured composites, as dual cured materials contain both of them.

Nanofilled and nanohybrid RBCs have high polishing ability with a gloss comparable to that of the enamel, good wear resistance and transparency [6,7]. Due to their increased aesthetics, strength and durability, they are increasingly preferred by clinicians as a universal restorative material for both anterior and posterior restorations [2].

Flowable RBCs are characterized with lower amount of inorganic filler, which leads to more liquid consistency and low viscosity compared to conventional composites. Their main advantages include: high wettability of the tooth surface; ability to form layers with a minimum thickness; high flexibility; radiopacity and different colours of the material [2, 9-11]. But they have some disadvantages such as high polymerization shrinkage and poor mechanical properties. Therefore, they are used mainly for restoring V class cavities, very small occlusal defects, as liners in class I and II cavities [12].

Conventional RBCs are applied using incremental technique which is time consuming process, contributing to more inaccuracies. To simplify the procedure, the manufacturers create bulk fill composites. Bulk fill RBCs are developed in two consistencies – flowable and compactable. Using these composites it is possible to place layers up to 5 mm (compared to 2 mm for conventional ones), while ensuring sufficient depth of cure. This is achieved by optimization of the photoinitiator system, modification of fillers (larger size or higher particle translucency) or inclusion of various chemicals in the composition [13,14]. Flowable bulk fill composites have lower filler content than compactable ones and it is required the restoration to be completed with a composite layer with a larger filler amount, which has a higher hardness and wear resistance. The application of bulk fill composites in posterior restorations reduces cusp deflection [15-17] and polymerization stress [18], thus increasing the fracture resistance of the restoration and hard dental tissues (HDT) [15]. However, flowable bulk fill composites have poorer mechanical properties than compactable and conventional ones, so they should not be used as a surface layer of the filling, which is exposed to direct chewing load [19].

The successful polymerization of RBCs, characterizing with monomer-polymer conversion ratio, can be evaluated by their hardness. There is a positive correlation between the conversion ratio and hardness of dental composites [20,21]. It was found that 80% bottom-to-top hardness ratio corresponds to 90% conversion ratio [22]. On the other hand, the wear and fracture resistance as well as the durability of the restoration are defined by the composite hardness. Consequently, the hardness of RBCs is of great interest of many research groups.

El-Nawawy M. et al. [23] investigated the micro-hardness of three types of composites – nanofilled, packable and hybrid. It was found that the micro-hardness and depth of cure of the packable composite were better than these of the two ones. Good correlation was revealed between the micro-hardness and depth of cure for the three investigated composites. The micro-hardness of two flowable and two bulk fill composites were investigated in 4 mm thick samples and compared with the two conventional in the work of Jang J.H. et al. [24]. It was found that the mean bottom surface hardness of bulk fill flowable composites exceeded 80% of the top surface HV, while that of the highly filled flowable (G-aenial Universal Flo) and bulk fill non-flowable composite could not reach 80%HV. Therefore, sufficient polymerization could not be provided in 4 mm thick layer of the last two composites. Son S.A. et al. [14] investigated the micro-hardness of five bulk fill and two conventional resin-based composites. It was established linear micro-hardness decrease with specimen thickness increase, as the top-to-bottom decrease of bulk fill composites is lower (11.5-48.8%) compared to the conventional RBCs (57.3-71.5%). The bulk fill composites with lower filler content showed lower micro-hardness and higher polymerization shrinkage than the conventional ones. The same tendency for lower hardness of the low viscosity resins compared to that of high viscosity resins was revealed in the research of Rizzante F.A.P. et al. [25]. According to them, all bulk fill composites showed depth of cure higher than 4.5 mm and similar or even lower polymerization shrinkage compared to the conventional ones.

Nowadays there is a great variety of dental RBCs, which is one of the main reasons for the comparatively low level of knowledge of practitioners about the composites properties and the conditions for successful polymerization process [26,27]. Therefore, there is a need for constant study of the properties of dental composites. The aim of the present paper is to investigate the micro-hardness of three types of rasin-based composites – conventional, bulk fill and flowable. The micro-hardness is evaluated in varying of three factors – light intensity of the light curing unit (LCU), curing time and composite thickness.

2. Materials and methods

2.1. Materials and samples manufacturing

Three types of light cured resin-based composites were used in the research: Universal nanohybrid Composite (UC) Evetric (Ivoclar Vivadent, Lichtenstein), nanohybrid Bulk fill Composite (BC) for posterior restorations Filtek One Bulk Fill Restorative (3M, USA) and universal nanofilled Flowable Composite (FC) G-aenial Universal Flo (GC, Japan), recommended for Class I, II, III, IV and V Restorations. All composites were of A2 shade, but had a different composition and organic matrix/filler ratio (Tab. 1) [2]. Cylindrical specimens with a diameter of 5 mm and thicknesses of 2, 3 and 4 mm were made from each composite (Fig. 1a).

Table 1.
Composition of the investigated dental composites [2]

№	Composite	Composition: Component	Composition: Amount	Matrix/filler ratio, wt.%
1	UC *Evetric* [28,29]	Matrix: UDMA (Urethane dimethacrylate) BIS-GMA (Bisphenol A glycydil dimethacrylate) Bis-EMA (Bisphenol A polyethethylene glycol dimethacrylate) Fillers: Barium glass, Ytterbium Fluoride (YbF_3), Mixed oxides and prepolymers 40nm-3μm.	 10-25%; 3-10%; 3-10%	19-20/80-81
2	FC *Filtek One Bulk Fill Restorative* [30,31]	Matrix: AUDMA (Aromatic Urethane Dimethacrylate) DDDMA (1,12-Dodecane Dimethycrylate) UDMA (Urethane dimethacrylate) Fillers: Silane Treated Ceramic, Silica, Zirconia and Ytterbium Fluoride.	 10-20% <10% 1-10%	23.5/76.5
3	FC *G-aenial Universal Flo* [32]	Matrix: UDMA (Urethane dimethacrylate) Bis-EMA (Bisphenol A polyethethylene glycol dimethacrylate) Dimethacrylate component Fillers: Silicon dioxide (16 nm), Strontium glass (200 nm), pigments.	 10-20% 5-10% 5-10%	31/69

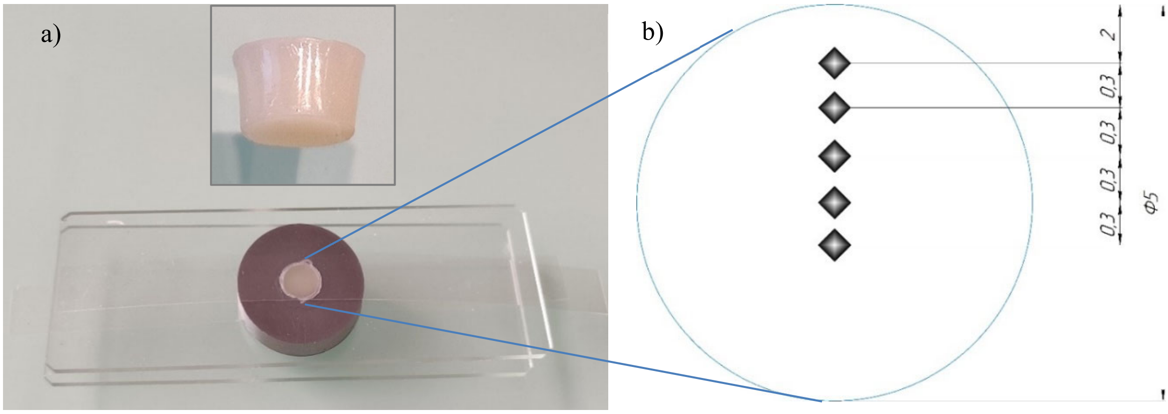


Fig. 1. Manufacturing of composite samples – a) and scheme of micro-hardness measurements – b)

Table 2.
Micro-hardness difference between top and bottom surfaces of the investigated composites

№	Layer thickness, mm	Light intensity, mW/cm^2	Irradiation time, s	Micro-hardness difference, %		
				UC Evetric	FC G-aenial Universal flo	BC Filtek One Bulk Fill Restorative
1	2	600	20	20	11	5
2	2	600	40	10	4	6
3	2	600	60	10	4	1
4	2	1000	20	12	11	3
5	2	1500	20	18	7	2
6	2	1500	60	11	4	1
7	3	600	20	50*	35*	13
8	3	1500	60	18	8	2
9	4	600	20	73*	69*	22*
10	4	600	60	34*	30*	4
11	4	1000	60	37*	18	4
12	4	1500	20	56*	40*	10
13	4	1500	40	38*	20	4
14	4	1500	60	28*	18	3

* Micro-hardness difference between top and bottom surfaces larger than 20%.

Polyurethane moulds with an inner diameter of 5 mm, an outer diameter of 20 mm and a thickness of 2, 3 and 4 mm, respectively, were used for the making of the samples. (Fig. 1a). A glass slide was used as a smooth base, on which a transparent celluloid strip was placed, and the polyurethane mould was placed on it. It was filled with composite, which was applied in one layer. Excess material was removed; a celluloid strip was placed on the composite and pressed with a new glass slide to obtain the smooth surface needed for the proper measurement of the hardness. The light guide tip of the LED LCU Curing Pen (Eighteeth, China) with a wavelength of 385-515 nm was placed in contact with the glass and the sample was polymerized for 20, 40 or 60 s with a light intensity of 600, 1000 or 1500 mW/cm^2 in continuous mode. The distance between the LCU's tip and the top surface of the composite was 1 mm, which is the exact thickness of the glass slide.

Three specimens were made for each combination of parameters (Tab. 2). The total number of samples was 126, or 42 samples for each composite. They were stored in a dry dark container at room temperature for 24 hours, after which the hardness measurements were performed [2].

2.2. Hardness measurements

The Vickers micro-hardness was investigated by ZHVμ-S (Zwick/Roell, Germany) hardness tester with 50 gr loading for 10 s. Five measurements (Fig. 1b) were performed on the top and bottom surfaces of each specimen. The average values wepe used in the analysis [2].

3. Results obtained

The hardness measurements (Fig. 2) have shown that the highest is the micro-hardness of BC Filtek One Bulk Fill Restorative (59.1-67.2 HV on top and 57.5-65.3 HV on bottom surfaces), followed by UC Evetric (top 42-62.7 HV and bottom 32.7-45 HV) and the lowest is that of FC G-aenial Universal Flo (top 42.4-50 HV and bottom 31.7-46.3 HV). For all investigated composites, the highest micro-hardness on the top surface was obtained using modes with the highest light intensity of 1500 mW/cm^2 and 60 s irradiation time. While for the values on the bottom surface, the layer thickness has a great influence. In that case, the highest is the micro-hardness on the bottom surfaces of the thinnest samples (2 mm) processed with the largest parameters – intensity and time.

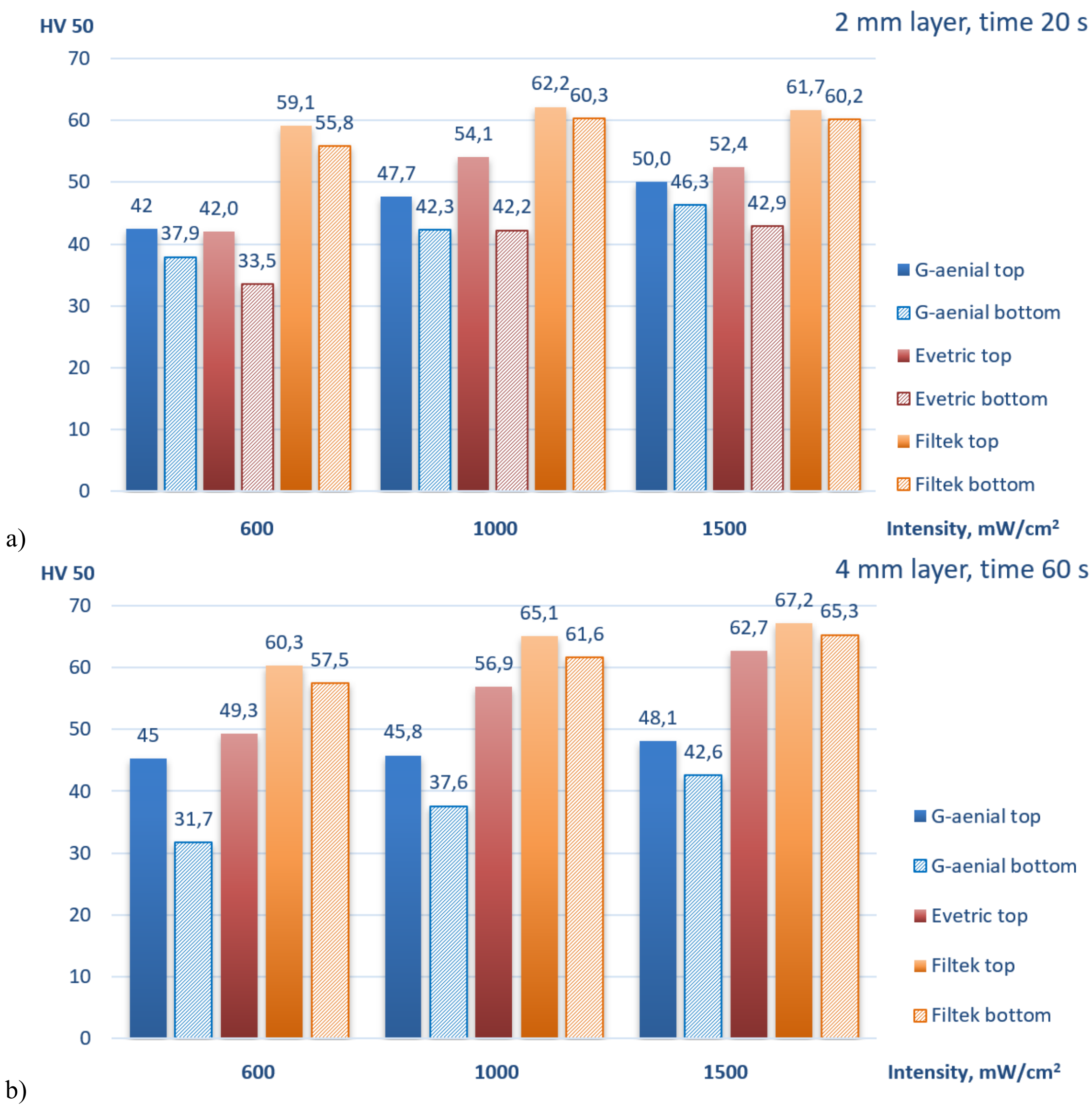
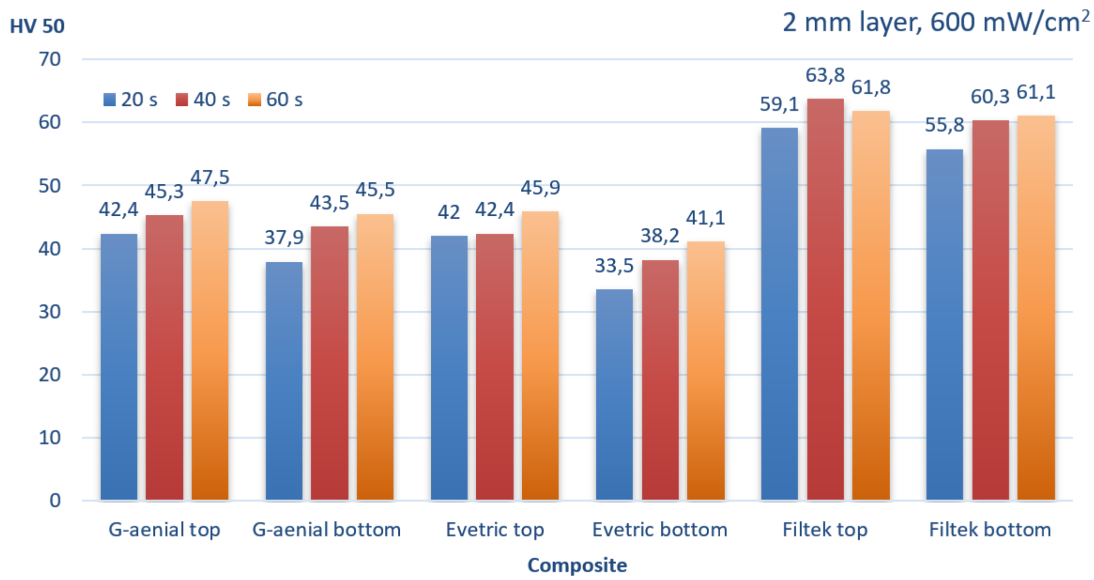


Fig. 2. Micro-hardness of investigated composites depending on the LCU intensity in 2 mm layer thickness, 20 s curing time – a) and 4 mm layer thickness, 60 s curing time – b)

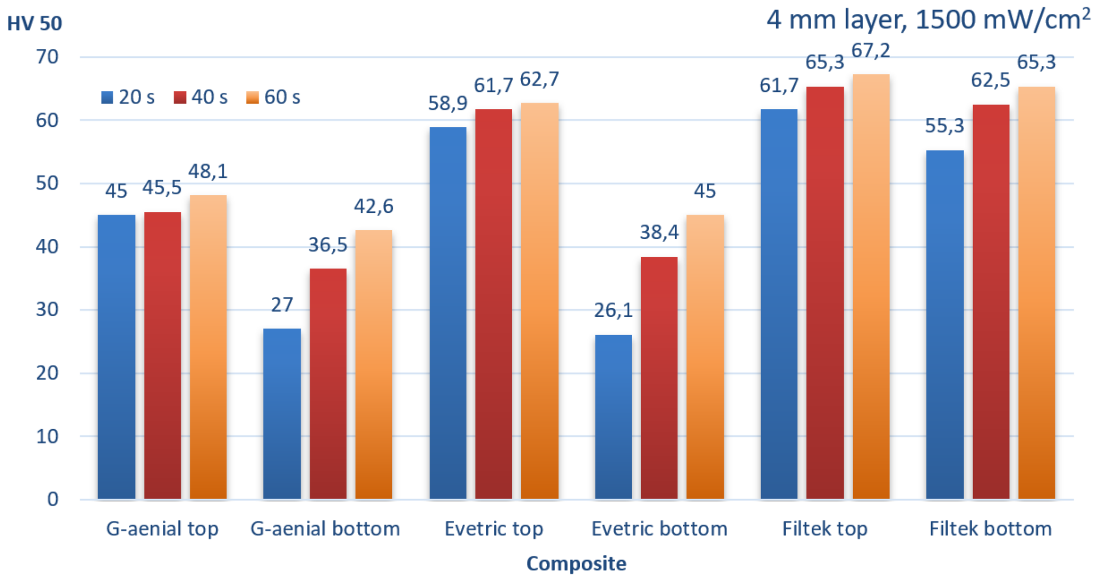
Increasing the light intensity leads to increase of the micro-hardness on both surfaces of the three composites (Fig. 2). The influence of intensity is weakest in all polymerization modes of BC Filtek One Bulk Fill Restorative. The strongest is the intensity influence on the micro-hardness of the top surface of UC Evetric in 60 s irradiation time.

The increase of irradiation time results in slight increase of micro-hardness on the top surfaces of all composites (Fig. 3). However, the micro-hardness increase of the bottom surface is more clearly pronounced, especially in the layer with the largest thickness of 4 mm of FC G-aenial Universal Flo and UC Evetric.

The change of the layer thickness influences mostly on the micro-hardness on the bottom surface (Fig. 4). It is decreased with the layer thickness increasing. This effect is more clearly pronounced in the polymerization modes with the lowest intensity and irradiation time (600 mW/cm^2 and 20 s). The strongest is the layer thickness influence in UC Evetric, characterizing with the highest difference of



a)



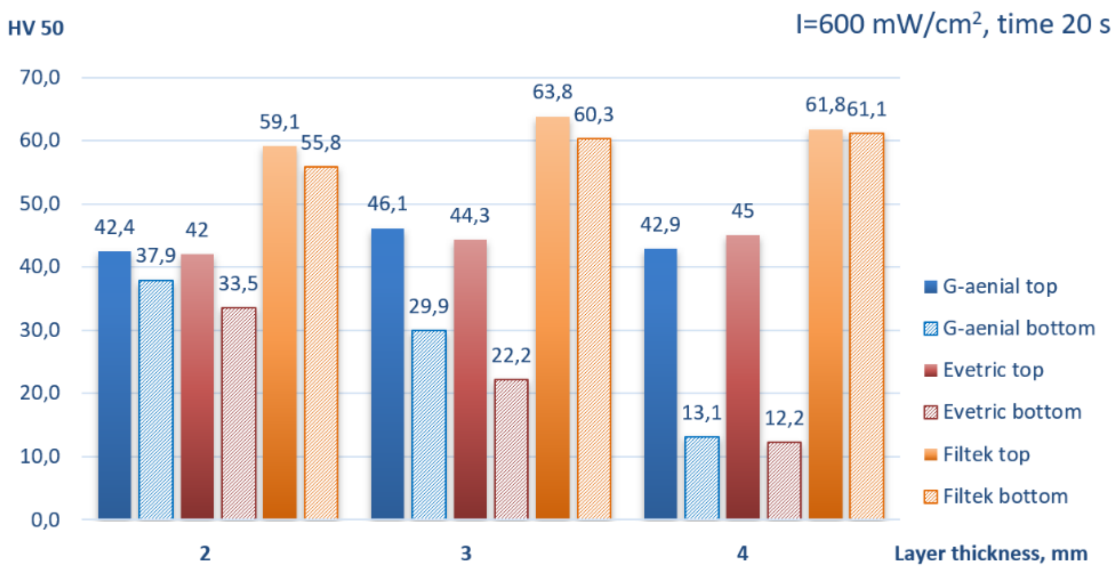
b)

Fig. 3. Micro-hardness of investigated composites depending on the change of irradiation time in 2 mm layer thickness, 600 mW/cm^2 light intensity – a) and 4 mm layer thickness, 1500 mW/cm^2 light intensity – b)

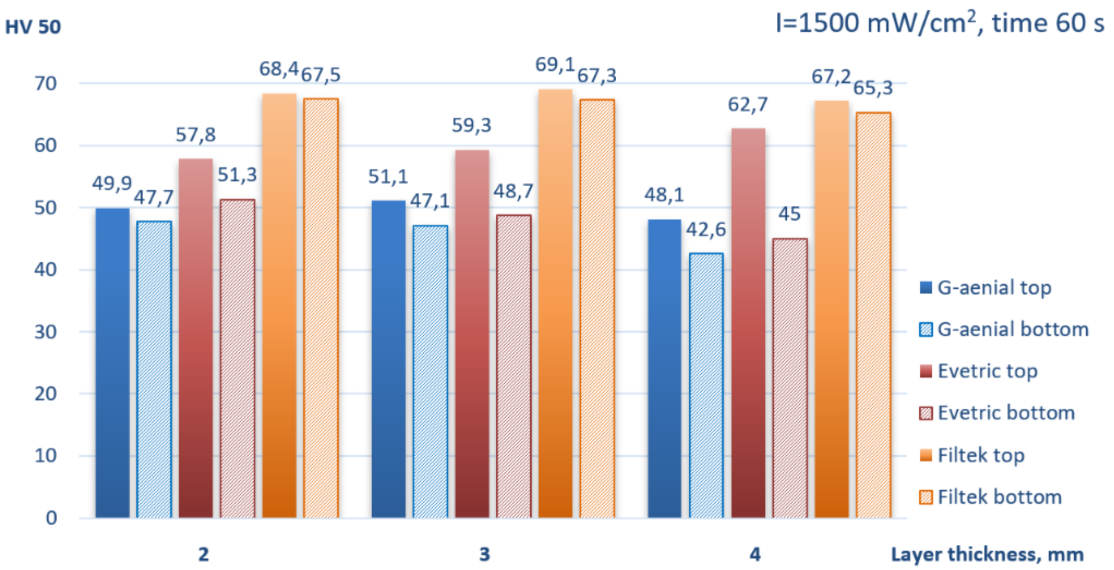
micro-hardness on top and bottom surfaces, which vary between 10-73% (Tab. 2). The micro-hardness difference in FC G-aenial Universal Flo is lower (4-69%), but comparable to that of UC. The lowest is the influence of layer thickness in BC Filtek One Bulk Fill Restorative with top-to-bottom micro-hardness difference of 1-22%. Our research have shown an interesting result – the highest micro-hardness on the top surface in BC Filtek One Bulk Fill Restorative and FC G-aenial Universal Flo is obtained in 3 mm layer thickness in the polymerization modes used (Fig. 4).

4. Discussion

The successful polymerization of resin-based dental composites, expressed in their high hardness, depends on many factors referring to the LCU, process parameters and composition and properties of the restorative material. The main of them are LCU intensity, irradiation time, distance and angulation of the LCU tip, composites thickness and their composition and optical properties [2].



a)



b)

Fig. 4. Micro-hardness of investigated composites depending on the layer thickness at 600 mW/cm^2 light intensity, 20 s curing time – a) and 1500 mW/cm^2 light intensity, 60 s curing time – b)

The hardness of the resin-based composites is defined by the monomer-polymer conversion ratio: the higher the polymerization ratio, the higher the hardness. Therefore, the hardness on the bottom surface of the composites is lower compared to the top due to the lower polymerization ratio owing to the lower light energy input [2,14,26]. Difference between the maximum hardness on the top surface of the composite and that at the bottom less than 20% is accepted as a guideline for determining whether the material at the bottom of the restoration is fully polymerized [21,22,24]. It is found that the KHN top-to-bottom ratio of 80% corresponds to a top-to-bottom conversion ratio of 90% [22].

It is noteworthy that under the same polymerization conditions – light intensity and irradiation time, composite thickness and colour, LCU tip distance and angulation different micro-hardness values are obtained for the three investigated composites. The lowest is the micro-hardness of FC G-aenial Universal Flo, followed by that of UC Evetric, as the highest is in BC Filtek One Bulk Fill Restorative (Fig. 2, Fig. 3 and Fig. 4). The obtained results are defined by the difference in the composition of the materials – the organic matrix/filler ratio on the one hand, and on the other – the type of inorganic filler particles.

In the FC G-aenial Universal Flo, the matrix/filler weight ratio is 31/69%. The high content of the organic matrix determines the lowest viscosity of the composite and its relatively low micro-hardness values. BC Filtek One Bulk Fill Restorative is the composite with the highest hardness, although it contains a larger amount of organic matrix than UC Evetric – the matrix/filler ratio for the first is 23.5/76.5%, and for the second – 19-20/80-81%. However, the composition of the inorganic component is different. The filler of the BC contains ceramics and zirconium, which have high hardness, while that of UC contains prepolymers characterizing by lower hardness.

When comparing the three composites with respect to the difference in hardness between the top and bottom surface, it can be seen that in UC Evetric a difference above the permissible 20% occurs nearly in the half of modes, mostly in the samples with 3 and 4 mm thickness (Tab. 2), confirming the results of Jang J.H. et al. [24]. The impossibility of sufficient polymerization of the material in its entire volume is mainly due to the increased thickness of the layer. This is expected, as the manufacturer's advice is incremental technique to be used in which the placement of the composite should be carried out in portions up to 2 mm thick [29].

Despite the lower hardness of FC G-aenial Universal Flo, better polymerization of the material is observed in its entire volume even with a greater layer thickness of 3 and 4 mm. Only in four modes, characterizing with lower energy input, there is hardness difference between the top and bottom surface over 20% (Tab. 2). The higher polymerization depth of FC G-aenial Universal Flo is due to the lower amount and smaller size of the fillers, allowing the light to reach the deepest layers of the restoration more easily.

In BC Filtek One Bulk Fill Restorative very close micro-hardness values on the top and bottom surfaces are obtained, as only in one mode there is hardness difference greater than 20% (Tab. 2). As a typical representative of the new generation of bulk fill composites, this material is characterized by a larger polymerization depth – up to 5 mm [30]. This is due to comparatively high matrix content and presence of nanoparticles in the filler, defining a very high translucency of the unpolymerized composite [33-36], which allows the light to penetrate easily to the deepest layers of the restoration.

In UC Evetric, the increase in the layer thickness leads to a high decrease in the hardness at the bottom of the restoration (Fig. 4), while in BC Filtek One Bulk Fill Restorative the thickness increase does not cause considerable change in the hardness at the bottom surface, confirming the results of Son S.A. et al. [14]. The difference in the ratio values is due to the different curing modes used. FC G-aenial Universal Flo is characterized with intermediate position between the other two composites. At low intensity and short polymerization time the hardness at the bottom of the material decreases significantly with increasing thickness, and at high intensity and increased polymerization time the hardness remains relatively unchanged.

5. Conclusions

Micro-hardness of three types of resin-based composites – universal, bulk fill and flowable was investigated in the present paper. The micro-hardness was evaluated in different polymerization modes varying with the light intensity, curing time and composite thickness. It was established that:

a) The highest is the micro-hardness of the bulk fill composite, followed by the conventional and the lowest is that of the flowable one.
b) Increasing the light intensity and curing time leads to increase of the micro-hardness on both surfaces of the three composites. In time increase the micro-hardness increase of the bottom surface is more clearly pronounced. The layer thickness influences mostly on the micro-hardness of the bottom surface - it is decreased with the layer thickness increasing.
c) The influence of the three variables on the micro-hardness is more clearly pronounced for the universal and flowable composites than the bulk fill.

The investigation of the micro-hardness of the three types of composites in different modes would be very helpful for clinicians to obtain successful polymerization of composite restorations in their everyday practice.

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