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## Comparative Study on The Structure, Physical Properties and Hardness Indentation of a Bulk Fill & An Incremental Composite Resin Restorative Materials

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### Abstract

Totally 80 disk-shaped specimens of Filtek™ Bulk-Fill, 3M ESPE, and Filtek™ Z250 XT, 3M ESPE, were prepared by using split-Teflon mold (4mm×10mm). Specimen characterization XRD, Surface roughness, degree of conversion, Surface microhardness, and depth of cure of the specimens were evaluated after the specimens were stored in distilled water in darkness at 37°C for 24 h. Statistical analysis was done using t-test with a significance level at  $p < 0.05$ . Filtek™ Z350 XT showed the highest mean value of Vickers microhardness either on top surface 94.94% or bottom surface 69.92% and recorded 76.51% while Filtek™ Bulk-Fill was 66.85% for depth of cure. Filtek bulk-fill recorded a lower mean value of 0.73 for Surface roughness than Filtek™ Z350 XT and the highest degree of conversion of 84.34%. Statistical analysis showed that there was a significant difference between the mean values for all tested results. Increasing the filler content proved to have ability to enhance some properties, specially the surface roughness. Incorporation of some structurally rigid monomers like Bis-DMA, UDMA, TEGDMA within the resin matrix have a great influence on the measured properties, including degree of conversion reached to 84.34%, depth of cure to a level of 76.51% and Vickers microhardness to 94.94%.

**Keywords:** Bulk-Fill Composite Resin, Degree of Conversion, Depth of Cure, Vickers Microhardness, Surface Roughness.

### 1. Introductions

Resin-based composites were successfully introduced to dentistry for the last few years to replace amalgam as a posterior restoration and overcome the esthetic problem rather than the functional problem of dental amalgam [1]. Composite resin is "a mixture or combination of two or more micro or macro constituents which differ in form and chemical composition and is essentially insoluble in each other" [2], and the resulted material (composite) combine the properties of the constituents included. It can be used as restorative materials and also to treat pits and fissure sealants. During the last few years, there have been great physical and mechanical improvements for developing more stable composite materials, which led to a wide variety of composite material selection based on the clinical situation [3]. We will use two types of composite resin materials. The first one is called incremental composite resin material. One of the important advantages of the incremental filling technique is the reduced thickness of the resin, which would provide enough light transmission and, therefore, polymerization, which leads to enhance physical properties. As a result of inadequate curing for incremental placement technique with increments greater than 2 mm thickness or for large cavities restoration [4], the use of incremental placement technique of resin composite is time-consuming for the patient and the operator because of increasing the time which is needed to place and cure each layer [5]. There may be also chances of moisture contamination, the possibility of voids incorporation or air bubble entrapment, bond failures between each incremental layer of the resin composite [6]. It also has a rough surface and a high tendency to discoloration. For these reasons, the introduction of the bulk-fill composites has aimed to solve the problems faced by incremental placement techniques of the conventional type. The advantage of the so-called "bulk fill" composite material is that it can be placed in a single layer of



4mm thickness as a bulk and cured in one step unlike the incremental fill technique [7, 8]. Using this type of composite resin is easier and faster than the incremental type, decreased moisture contamination and air bubbles. Therefore, it is expected to have superior physical and mechanical properties. Therefore, the aim of this study was to evaluate the structure, degree of conversion (DC %), Vickers microhardness (HV), depth of cure and surface roughness of bulk-fill resin-based composite and compare these measurements to those of incremental resin-based composite.

## 2. Materials and Methods

One of the newly introduced bulk-fill composites, Filtek™ Bulk-Fill (3M ESPE, USA) and one of an incremental composite resin, Filtek™ Z350 XT (3M ESPE, USA) are the materials used in the study as shown in Table 1. A total of 80 specimens (40 for each resin composite material used in this study) prepared by using a split-Teflon mold of 4mm thickness and 10 mm diameter. The mold was positioned over a microscopic glass plate and a celluloid strip, the mold was packed with a 4mm single layer in case of Filtek™ Bulk-Fill composite and with two successive layers each of 2mm thickness in case of Filtek™ Z350 XT composite. The packed material covered with another celluloid strip and compressed with a load of 0.5 kg over a glass plate to remove air bubbles and excess material. Each one of specimen was light cured by using a visible blue light source (LED source) that has an intensity of  $1000 \text{ mw.cm}^{-2}$  for 20 sec. After curing, specimens were removed gently out of the mold and polished from the top surface and their edges by Silicon carbide (SiC) papers with decreasing size of the abrasive particles (coarse, medium, fine, ultrafine) then they stored in distilled water at  $37^\circ\text{C}$  for 24hrs.

**Table1: Materials, manufacturer, the chemical composition of the matrix and inorganic filler content by weight and volume %.**

Name	Shade	Composition	Manufacture
Filtek™ Bulk-Fill (Bulk-fill)	A3	Resin matrix: UDMA <sup>a</sup> , Aromatic UDMA and 1, 12-dodecane-DMA <sup>b</sup> Inorganic filler: non-agglomerated/non-aggregated 20nm silica filler, non-agglomerated/non-aggregated (4:10)nm zirconia filler, aggregated zirconia/silica cluster filler, ytterbium trifluoride filler (76.5 wt%, 58.4 vol%)	3M ESPE, Dental Products Division, St. Paul, MN, USA
Filtek™ Z350 XT (Incremental)	A3	Resin matrix: Bis-GMA <sup>c</sup> , UDMA, TEG-DMA <sup>d</sup> , PEG-DMA <sup>e</sup> , Bis-EMA <sup>f</sup> Inorganic filler: non-agglomerated/non-aggregated 20nm silica filler, non-agglomerated/non-aggregated (4:10)nm zirconia filler, aggregated zirconia/silica cluster filler (72.5 wt%, 55.6 vol%)	3M ESPE, Dental Products Division, St. Paul, MN, USA

a: urethane dimethacrylate, b: dimethacrylate, c: bisphenol-A-glycidyl methacrylate, d: triethylene glycol dimethacrylate, e: polyethylene glycol dimethacrylate, f: ethoxylated bisphenol-A-methacrylate, wt%: weight percentage, vol%: volume percentage.

Characterization of one specimen of each type of the used material was obtained by using a Shimadzu x-ray diffractometer (Dx-30) XRD with a CuK  $\alpha$  radiation with a wavelength  $\lambda=1.54056\text{\AA}$ . From Bragg's law, states that: if we bombard a crystal lattice with X-rays of a known wavelength ( $\lambda$ ) and at a known angle ( $\theta$ ), then we can detect the diffracted X-rays of various intensities which represent a specific interplanar spacing ( $d$ ) in the lattice.

$$n\lambda = 2d \sin \theta \dots\dots\dots (1)$$

For measuring the surface roughness, disk-shaped specimens of each composite material were polished from the top surface and their edges by Silicon carbide (SiC) papers with decreasing size of the abrasive particles (coarse, medium, fine, ultrafine) to get a smooth surface. The measurements were done in the air using a profilometer (SJ210 Mitutoyo Surface Roughness Tester). Totally, six areas (2 in right, 2 in left, and 2 in the center) were scanned on the top surface for evaluating the surface roughness in ( $\mu\text{m}$ ). Fourier transform infrared spectroscopy (Thermo Scientific Nicolet<sup>TM</sup>iS<sup>TM</sup>10FTIR spectrometer, Madison, WI USA) was preferably used to assess the degree of monomer conversion (DC %). Uncured specimen of each composite resin type was smeared onto a thin potassium bromide (KBr) discs and placed into a cell holder in the spectrophotometer. Therefore, a spectrum was obtained with the same parameters as for the cured specimen. After photopolymerization, each cured specimen of each composite type was pulverized into a very fine powder with a certain mortar and pestle. 5mg of the pulverized powder were mixed with 100 mg of potassium bromide (KBr) powder and then pressed in a press by using a load of 10 tons over 1min to get a thin disc, then positioned in a specimen holder and moved to the spectrophotometer. By using the diffuse-reflection mode of the FTIR technique, the absorbance peaks were recorded under the following conditions: 32 scans, over a used wavelength of  $(400 - 4000) \text{ cm}^{-1}$  and a resolution of  $4 \text{ cm}^{-1}$ . The degree of resin monomer conversion of the composite was evaluated by estimating the changes in the peak absorbance intensities of the aliphatic (C=C) peak at  $1638 \text{ cm}^{-1}$  and that of an internal standard absorbance peak of aromatic (C=C) at  $1608 \text{ cm}^{-1}$  of the cured and uncured material. The degree of monomer conversion (DC %) for each specimen was measured by using the following equation:

$$\text{DC}\% = 1 - \left[ \frac{[1638 \text{ cm}^{-1}/1608 \text{ cm}^{-1}]_{\text{cured}}}{[1638 \text{ cm}^{-1}/1608 \text{ cm}^{-1}]_{\text{uncured}}} \right] \times 100 \dots\dots\dots (2)$$

The depth of cure of the investigated samples can be evaluated by using digital display Vickers microhardness tester (Model, FM.7 Future-Tech. Corp. Tokyo Japan) by measuring the mean value of hardness ratio of each sample of each composite type. It would be measured by dividing the Vickers hardness number (VHN) mean value of the bottom surface by the (VHN) mean value of the top surface for each used sample by using the following equation:

$$\text{Depth of cure} = \frac{\text{VHN (bottom surface)}}{\text{VHN (top surface)}} \times 100 \dots\dots\dots (3)$$

The microhardness of the specimens of each type on the top and bottom surface was measured in ( $\text{gf}/\text{mm}^2$ ) by using digital display Vickers microhardness tester (Model, FM.7 Future-Tech. Corp. Tokyo Japan) equipped with a certain diamond pyramidal indenter and an objective lens with a magnification of 20X. A load of 200 grams was applied for 10 seconds. Five random indentations were made either on the top and the bottom surface of each specimen. The length of the diagonals ( $d_1$  and  $d_2$ ) left by the indenter was digitally measured under the light of the microscope that connected with the Vickers microhardness technique. The surface Vickers hardness number (VHN) can be determined by dividing the applied load to the indentation area as following:

$$\text{VHN} = 1.8544 \times \frac{F}{d^2} \dots\dots\dots (4)$$

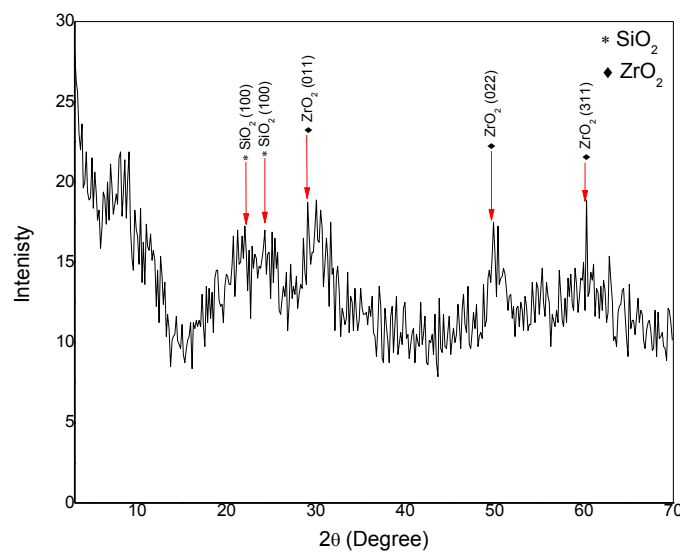


Where  $F$  is the applied load in gram of force (gf), 1.854 is a constant related to geometric factor for the diamond pyramid and  $d$  is the average length of diagonal in (mm).

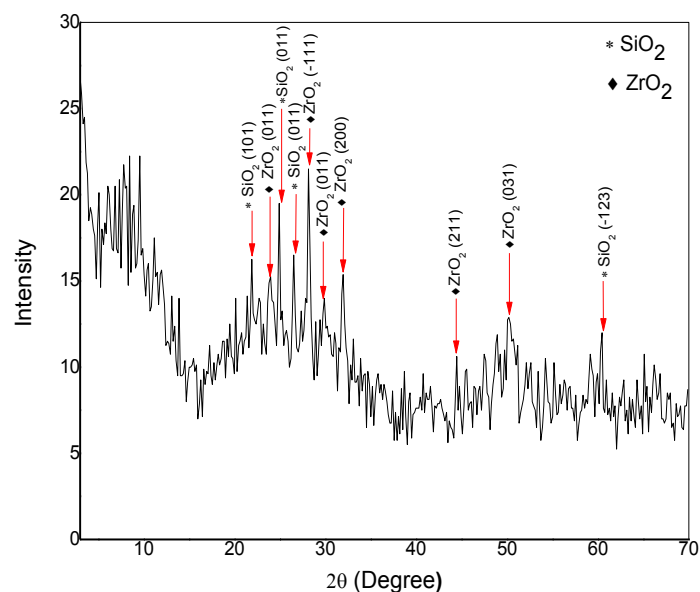
### 3. Results and Discussions

#### 3.1. XRD analysis

The structure characterization of each type of our sample can be tested using XRD technique because it has some advantages such as easy sample preparation, effective and accurate technique and low cost which Saves time and money. The XRD pattern for the incremental composite resin Filtek™ Z350 XT and the bulk-fill composites Filtek™ Bulk-Fill and are shown in Fig.1a and Fig.1b respectively.



**Fig. 1a: XRD pattern of Filtek™ Z350 XT.**



**Fig.1b: XRD pattern of Filtek™ Bulk-Fill.**

The XRD pattern of the two composite materials proved that they have a polycrystalline structure and showed that there are two inter-metallic components appeared in both samples, Silicon Oxide ( $\text{SiO}_2$ ) and Zirconium Oxide ( $\text{ZrO}_2$ ) but presented in different phases. For Filtek™ Bulk-Fill pattern, the existing  $\text{SiO}_2$  inter-metallic compound represented in tetragonal phase named Cristobalite- $\text{SiO}_2$  [101] (JCPDS card no.: 04-037) at  $2\theta=21.9$ , in monoclinic phase named Silicon Oxide- $\text{SiO}_2$  [011] (JCPDS card no.: 82-1574) at  $2\theta=24.7$ , in hexagonal phase of Quartz Low- $\text{SiO}_2$  [011] (JCPDS card no.: 85-0335) at  $2\theta=26.5$  and in monoclinic phase of Silicon Oxide- $\text{SiO}_2$  [-123] (JCPDS card no.: 82-1574) at  $2\theta=60.47$ . The other inter-metallic  $\text{ZrO}_2$  appeared in monoclinic phase named Baddeleyiet- $\text{ZrO}_2$  [011] (JCPDS card no.: 24-1165) at  $2\theta=24.07$  and the same in [-111] at  $2\theta=28.2$ , in orthorhombic phase of Zirconium Oxide- $\text{ZrO}_2$  [011] (JCPDS card no.: 83-0809) at  $2\theta=30.12$ , [200] at  $2\theta=31.9$ , [211] at  $2\theta=44.5$  and [031] at  $2\theta=50.27$ . For the Filtek™ Z350 XT pattern, the inter-metallic compound  $\text{SiO}_2$  presented only in the monoclinic phase of Silicon Oxide- $\text{SiO}_2$  [100] (JCPDS card no.: 82-1575) at  $2\theta=21.7$  and  $2\theta=24.2$ . The  $\text{ZrO}_2$  inter-metallic compound appeared in orthorhombic phase of Zirconium Oxide- $\text{ZrO}_2$  [011] (JCPDS card no.: 79-1796) at  $2\theta=30.1$ , [022] at  $2\theta=49.8$  and [311] at  $2\theta=60.31$ . The broad halos in the XRD pattern are the organic binder in the composites such as bisphenol-A-glycidyl methacrylate (Bis-GMA) and the crystalline phase which is the inorganic materials that give the peaks in the XRD pattern. It was seen that, the broad peaks in the XRD pattern are related to formation of the nano-sized grains and the change of the peaks is related the change of the inorganic filler in the composites [9]. From the XRD analysis of the materials used in this study we found that the polycrystalline structures of their patterns mean that using this types of material provide a regular and full packing also increase the longevity of the filling and reducing the possibility of caries. Therefore, they are a good choice to be used for teeth filling and restorations specially, when they used for the upper part of the teeth.

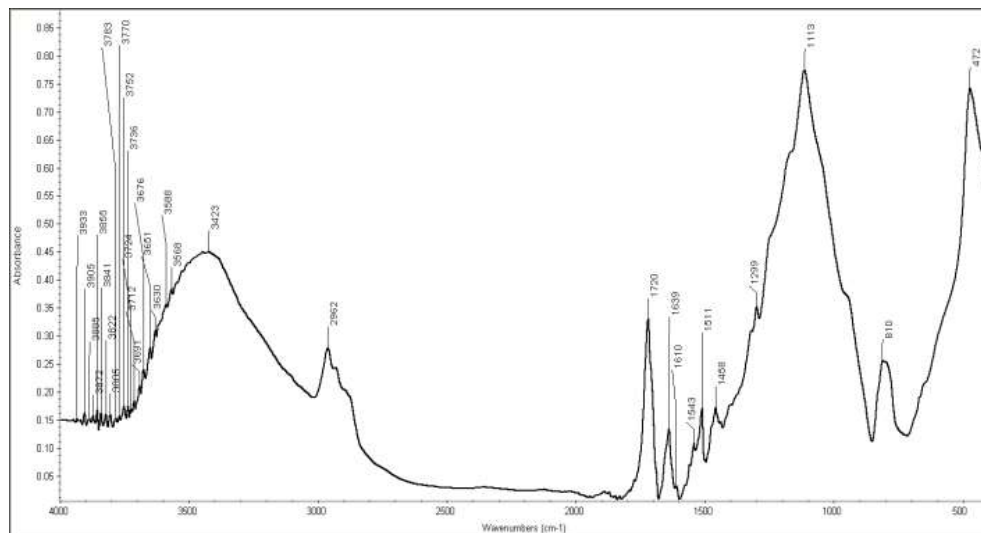
### 3.2. Surface roughness

Mean and standard deviation values of the surface roughness of the tested groups in ( $\mu\text{m}$ ) were listed in Table 2. For surface roughness measurement, the obtained results showed that Filtek™ Bulk-Fill group has a mean value ( $0.73\pm 0.34$ ) lower than that we recorded from the Filtek™ Z350 XT incremental group ( $1.70\pm 0.69$ ). This can be explained as the lower mean value of the surface roughness, the smoother the surface of the sample. Surface roughness strongly affects the esthetic appearance and restorations discoloration, the possibility of plaque accumulation, risk of secondary caries, inflammation of periodontal and tooth wear of opposing or adjacent teeth [10, 11]. As the mean value of the surface roughness of a material increases, the ability of the plaque accumulation, risk of secondary caries, inflammation of periodontal and tooth wear of opposing or adjacent teeth problems increase [12]. From the recorded results, we found that the risk of the previous problems to occur for Filtek™ Z350 XT (incremental resin composites) is higher than the Filtek™ Bulk-Fill resin composite. In our study, the possible reason for high surface roughness recorded for Filtek™ Z350 XT may be attribute to lower filler loading (72.5 wt%) compared with that of Filtek Bulk-Fill (76.5 wt%) [13].

### 3.3. Degree of Conversion

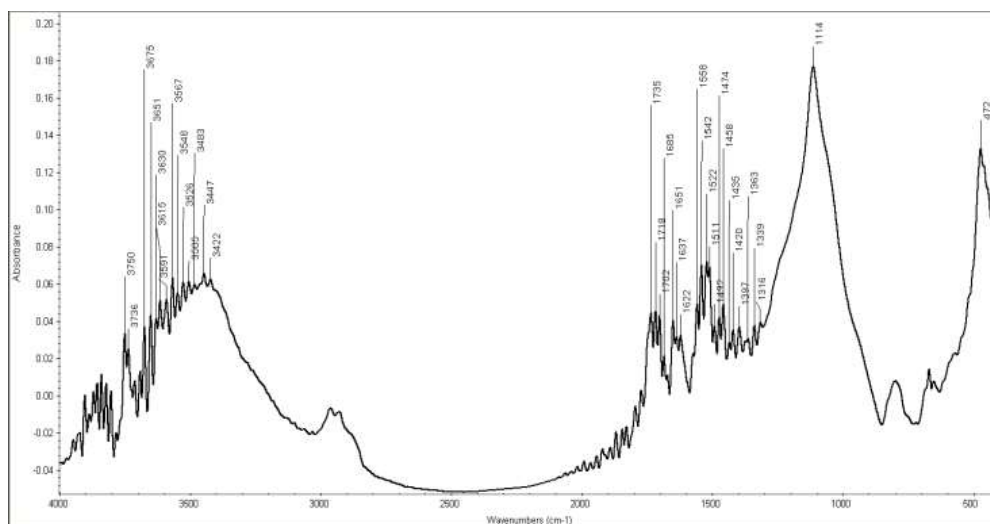
The results showed that the Filtek™ Bulk-Fill composite resin group recorded a higher mean DC% value ( $84.34\pm 0.19$ ) while the Filtek™ Z350 XT group recorded ( $76.76\pm 1.29$ ) as shown in Table 2. For the degree of conversion, the reported absorbance peaks intensities for the uncured and the cured specimens by the visible blue light source (LED) of the Filtek Bulk-Fill resin composite were graphically presented in Fig. 2 and Fig. 3 respectively. The noted absorbance peaks intensities of the infrared (IR) rays by FTIR of the aliphatic bond ( $\text{C}=\text{C}$ ) of the monomer were ( $1637\text{ cm}^{-1}$ ) and also ( $1637\text{ cm}^{-1}$ ) for both the uncured and cured resins respectively. Also, the noted absorbance peaks intensities of the IR rays by FTIR of the aromatic bond ( $\text{C}-\text{C}$ ) of the monomer for both the uncured and cured by (LED) resins were ( $1604\text{ cm}^{-1}$ ) and ( $1602\text{ cm}^{-1}$ ) respectively.





**Fig. 4: FTIR data analysis for the uncured Filtek™ Z350 XT composite resin specimen.**

(The absorbance peak reported for aliphatic bond C=C at the wavelength of  $1639\text{ cm}^{-1}$  was 0.131, while the absorbance peak of the aromatic C—C at the wavelength of  $1610\text{ cm}^{-1}$  was 0.0297).



**Fig. 5: Analyzed data by FTIR for the cured Filtek™ Z350 XT composite resin specimen.**

(The absorbance peak reported for the aliphatic bond C=C at the wavelength of  $1637\text{ cm}^{-1}$  was 0.0310 while the absorbance peak of the aromatic C—C at the wavelength of  $1622\text{ cm}^{-1}$  was 0.0318).

A lower degree of monomer conversion could greatly affect the longevity of the composite resin restoration because an inadequate conversion might result in unreacted monomers, which can dissolve in the wet environment of the mouth [14, 15], that may lead to an increase the amount of released unreacted monomer, which leads to a decrease in the biocompatibility of the restorations [16]. Moreover, the unreacted functional groups which did not cured by LED would act as plasticizers and then restorations with decreasing mechanical properties were produced [17, 18]. The main effective features of a monomer that have a strong effect on reactivity and DC are; the initial monomer viscosity, flexibility, and concentration of its chemical composition [19]. Filtek™ Bulk-Fill showed a significant higher DC% than Filtek™ Z350 XT as recorded, this might be as a result of the difference in their chemical structure of organic matrix. Filtek™ Bulk-Fill organic matrix contains; Aromatic UDMA, UDMA and 1, 12-dodecane-DMA while Filtek™ Z350 XT contains Bis-GMA, UDMA, TEG-DMA, PEG-DMA, and Bis-EMA. It was found that the degree of conversion of different monomer systems

increases in the following order: Bis-GMA < Bis-EMA < UDMA < TEGDMA. The amine group in the urethane structure of the UDMA monomer is responsible for the effective continuous chain transfer reactions that allow continuity of polymerization, these reactions enhance the polymerization and monomer conversion, resulting in an improved monomer resin conversion [20]. So, this can explain the significant higher DC and reactivity of the Filtek Bulk-Fill organic resin matrix. Although the Filtek™ Z350 XT organic matrix containing UDMA monomer similar to that of the Filtek™ Bulk-Fill it recorded a lower value of DC. This might be possible because of polymerization characteristics that significantly affected by the difference in the chemical composition of the organic matrix [21] and the concentration of each included monomer in the matrix [22]. This can explain the increase monomers reactivity and higher DC% of the UDMA including an organic matrix of the Filtek™ Bulk-Fill composite compared to the Bis-GMA including an organic matrix of incremental FiltekZ350 XT composite [23]. Such findings could be also explained by the translucency difference between the used bulk-fill and the incrementally filled resin composite materials. With higher degree of translucency, the Filtek™ Bulk-Fill resin composite could allow much more of the photo-polymerization light to be transmitted deeply through the resin of the composite material, therefore caused more polymerization and conversion of the resin composite monomers [24].

### 3.4. Depth of cure

The Filtek™ Bulk-Fill group recorded a mean value of depth of cure ( $66.85 \pm 14.52$ ) and the other group recorded ( $76.51 \pm 10.47$ ) as listed in Table 2. The results of the t-test showed that there were significant differences ( $p < 0.05$ ) among all mean values of the tested composite material groups as shown by Levene's test for equality of variances. The depth of cure of the used bulk-fill composite was significantly lower than that of the incremental composite that probably because of the reduced increment thickness of the Filtek™ Z350 XT type which could provide enough light penetration and subsequent polymerization. Our result also was in agreement with the explanation that could clarify the higher depth of cure of Filtek™ Z350 XT is that; when the incremental insertion method was applied, a high significant depth of cure measurement was obtained compared to that of the bulk insertion. That results could be confirmed by a former study which could be connected to the fact that, the prepared specimens with the incremental packing technique were able to receive much more total energy than those that inserted with bulk technique [25].

**Table 2: Means and standard deviations (SD) value of DC%, depth of cure and surface roughness of the two tested groups.**

Group	Degree of conversion (Mean $\pm$ SD)	Depth of cure (Mean $\pm$ SD)	Surface roughness ( $\mu\text{m}$ ) (Mean $\pm$ SD)
Filtek™ Bulk-Fill	$84.34 \pm 0.19$	$66.85 \pm 14.52$	$0.73 \pm 0.34$
Filtek™ Z350 XT	$76.76 \pm 1.29$	$76.51 \pm 10.47$	$1.70 \pm 0.69$

### 3.5. Surface Microhardness

Means and standard deviations value for Vickers microhardness of the investigated groups were scheduled in table 3. The results show that the tested upper surface of the Filtek™ Z350 XT group has the highest mean value of Vickers microhardness ( $94.94 \pm 15.51$ ), while the lowest value was for the lower surface of the Filtek™ Bulk-Fill group ( $57.79 \pm 4.03$ ) as shown in table 3. Also, we found a significant difference between the results recorded from each group ( $p < 0.05$ ).



**Table 3: Means and standard deviations (SD) value of Vickers microhardness (Hv) of the tested groups.**

Group	Upper surface	Lower surface
	Hv (gf/mm <sup>2</sup> )	Hv (gf/mm <sup>2</sup> )
	(Mean ±SD)	(Mean ±SD)
Filtek™ Bulk-Fill	89.09±16.18	57.79±4.03
Filtek™ Z350 XT	94.94±15.51	69.92±5.01

The surface hardness of our tested materials in this study was evaluated using one of the most common and accurate techniques called Vickers's microhardness tester which is a suitable method for measuring the surface hardness of brittle material [26]. with the highest VHN value was recorded for the incrementally Filtek™ Z350 XT composite in comparison to that of bulk-filled composite Filtek™ Bulk-Fill, these results are supported with previous findings of Leporine et al.. In that study, it was found that Grandio (the incremental type) had a significant higher VHN value than that of several high viscosity bulk-filled composites, namely x-tra fil, Sonic Fill, Tetric EvoCeram Bulk Fill, and Xenius (GC,Europe). Many factors related to composition were noted to influence the surface hardness of composite resin restorative material [27]. It was reported that the mass fractions [28, 29, 30], size and distribution of filler's particles have highly significant effects on some physical and mechanical properties, including the hardness of the material's surface [31, 32]. It was also mentioned that there are other factors such as; particle shape, density, monomer type and ratio, degree of polymers cross-linking and photo-initiators seem to have a significant impact on the material surface hardness. The increased in VHN value of the incrementally filled composite compared to that of the bulk-filled composite either for top or bottom surface, could also be related to the more total energy delivered to the incrementally filled composite [33]. For each material, The VHN on bottom surfaces of Filtek™ Bulk-Fill and Filtek™ Z350 XT were significantly decreased compared to that of their top surfaces. For any resin-based composite increment, the VHN on the top surface would be expected to differ obviously with that on the bottom surface because of monomer reactivity and filler-matrix refractive index mismatch, since the bottom surface is more critically affected by the light intensity during the polymerization process [34].

## Conclusions

Based on the results evaluated from this study, we noted that the Filtek™ Bulk-Fill resin composite exhibits a higher degree of conversion, lower depth of cure and a lower mean value of surface roughness than the incremental resin composite. However, incrementally filled resin composite (Filtek™ Z350 XT) showed higher VHN especially on the top surface than the Filtek™ Bulk-Fill composite. It can be noted that the degree of conversion of the composite material increased with increasing the concentration of the UDMA monomer in the resin matrix of the material which responsible for the chain transfer reactions that allow continuity of polymerization and the increment thickness which influence the light penetration into the material and subsequent the polymerization process. Also, the depth of cure is highly affected by the amount of filler loaded in the material and the thickness of the incremental layer. In addition, the surface microhardness can be affected by the composition of the resin matrix of the material and the total energy delivered to the incrementally filled composite. So we can say that, the difference in organic matrix chemistry or composition and filler features (mass fraction, density, and particle size, shape, and distribution) contribute to significant differences in surface roughness, DC, depth of cure and microhardness values between tested materials.

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