Optical and Surface Properties of Different Bulk-Fill Resin Composites after Storage in Different Media

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Abstract: Aim: to evaluate and compare surface properties (wettability, surface roughness and color) of paste bulk fill resin composite materials on baseline and after incubation in RedBull or Black coffee storage media and to find out if there is any change in these surface properties after 1 week and 1 month incubation. **Materials and Methods:** Three bulk-fill resin composites (Filtek bulk fill- 3M, X-trafil- Voco, and SonicFill bulk-fill- Kerr) were used. For the contact angle and surface roughness testing, standardized disc-shaped resin composite samples (6 mm X 4 mm) were prepared. For the color testing, standardized Teflon molds (7 mm X 4 mm) with predetermined color as a standard background were used. Five samples were prepared from each material for each test and were light cured following the manufacturers' instructions. Samples were tested at baseline, after 1 week and 1 month incubation in RedBull for contact angle and surface roughness tests and in black coffee for the color test. One-way ANOVA test statistics was done to compare between the mean values for the 3 materials with p value less than 0.05. Least significance test was followed to compare between each two materials in case there were statistical significant results. Paired t test was used to compare between baseline Vs 1 week and baseline Vs one month in each group. **Results:** there was a statistically significant decrease in the contact angle measurement for all 3 materials at 1 week and 1 monthhand no statistical significance between the 3 materials at baseline and 1 month. There was no significant difference in the Ra (surface roughness) values between all the 3 materials at each incubation times. Each material had no significant difference in the Ra (surface roughness) values between baseline, 1 week and 1 month incubation in RedBull. There was a just noticeable color difference between baseline and 1 week for 3M and SonicFill. There was also a noticeable color difference between baseline and 1 month for all the 3 materials. **Conclusion:** Sports drink (RedBull) increased the wettability of the materials surface, which was directly proportional to the incubation time but did not have a direct effect on surface roughness of the materials. The use of black coffee as a storage media showed noticeable significant staining of the materials. Testing in a more oral simulation environment is needed to reach clinically relevant conclusion.

1. Introduction:

Resin composite materials witnessed a great evolution over the past years. Improvements in mechanical, surface and optical properties, clinical application and performance introduced a wide variety of resin composite restorative materials. This necessitates a comprehensive knowledge of the materials characteristics and performance by the dentist for better evidence-based selection of restorative materials.

Lately, bulk fill resin composite materials are being introduced into dentistry with an increasing number and different applications. All of them share the same claim of being able to be properly cured as on bulk up to 4 mm thickness (Alrahlah, 2014). This has the advantage of reducing the chairtime and making the resin composite restoration procedure simple compared to incremental placement (Margeas, 2015).

Changing materials composition can change its surface properties. Of the important surface properties

to dental restorative materials are wettability, surface roughness and optical properties. Wettability is an important property to give indication about the susceptibility to bacterial adherence and plaque accumulation (Quirynen, 1989). Wettability is measured by measuring the contact angle of a drop of a liquid on the surface of the material through the liquid using contact angle goniometer (Della Volpe, 2001). A smooth surface restoration is crucial for esthetic and biological success of the restoration. Rough surfaces have unpleasant optical properties and are more susceptible to stain, plaque and bacterial retention (Yildiz, 2015). Sports drinks can affect the surface hardness (Wongkhantee, 2006) and might affect other surface properties of the resin composite materials such as surface roughness and wettability.

Color change of the resin composite restorations is one of the most common problems that result in esthetic failure of the anterior restoration and may necessitate its replacement. Color change of resin composite restoration can result from extrinsic

pigmentation, which is present in variety of foods and drinks and one of the most common staining beverages is black coffee (Yildiz, 2015).

No previous study has compared the wettability or color stability of bulk fill resin composite restorative materials. One study evaluated the effect of the bulk fill resin thickness on its optical properties (Kim, 2015). Very limited number of studies has evaluated the surface roughness of bulk fill resin composite materials (Piskin, 201, Awad, 2015).The aim of this study is to evaluate and compare surface properties (wettability, surface roughness and color) of paste bulk fill resin composite materials on baseline and after incubation in RedBull or Black coffee storage media and to find out if there is any change in these surface properties after 1 week and 1 month incubation.

2. Materials and Methods:
Materials Used:
Three bulk-fill resin composites were used in this study and all of them of paste-consistency:
1.   Filtek bulk fill- 3M.
2.   X-trafil- Voco.
3.   SonicFill bulk-fill- Kerr.

The materials names, specifications, and manufacturers are described in details in table (1).

Table (1): Materials specifications, manufacturers and compositions.

Material	Composition	Shade	Manufacturer
Filtek Bulk Fill, Posterior restorative	**The resin matrix:** AUDMA, UDMA, and 1, 12-dodecane-DMA. **The filler:** Non-agglomerated/non-aggregated 20nm silica filler, a Non-agglomerated/non-aggregated 4 to 11 nm zirconia filler, an aggregated zirconia/silica cluster filler (20nm silica and 4 to 11 nm zirconia particles), and a ytterbium trifluoride filler consisting of agglomerate 100 nm particles. (Khalil Yousef, 2015)	A2	3M ESPE, St. Paul, USA
SonicFill, nanohybrid composite restorative	**The resin matrix:** (1-methylethylidene) bis (4, 1-phenyleneoxy-2, 1-ethanediloxy-2, 1-ethanediyl) bismethacrylate. (1-methylethylidene) bis [4, 1-phenyleneoxy (2-hydroxy-3, 1-propanediyl)] bismethacrylate. 2, 2'-rthylenedioxydiethyl dimethacrylate. **The filler:** Glass, oxide, and Silicon dioxide. (Khalil Yousef, 2015)	A2	Kerr Corporation, Orange, CA, USA
X-trafil light-curing posterior filling material	**The resin matrix:** Bis-GMA, UDMA, TEGDMA **The filler:** Barium-boron-alumino-silicate glass (2-3 μm) 86% by weight. (Abed, 2015)	Universal	Voco GmbH, Cuxhaven, Germany

AUDMA: Aromatic urethane dimethacrylate; Bis-GMA: bisphenol A glycidyaldimethacrylate; UDMA: urethane dimethacrylate; TEGDMA: triethyleneglycoldimethacrylate.

Samples Preparation:
For the contact angle and surface roughness testing, standardized disc-shaped resin composite samples were prepared using a Teflon mold with an internal diameter of 6 mm and thickness of 4 mm. The mold can split into two parts and an outer metallic ring to hold the two parts together. A celluloid Mylar strip overlying a glass slide was placed under the mold and another celluloid strip and glass slide over the mold after packing the composite material inside the mold in one increment and hand pressure was applied to get a flat sample surface and to remove excess material. Samples were light cured using high intensity Elipar[TM] LED light curing unit (3M ESPE) and the curing time applied according to the manufacturers' instructions. The none-testing surface (the surface not facing the light-curing unit) was marked with a small dot using a permanent pen.

For the color testing, standardized Teflon molds with predetermined color as a standard background were used. The mold has an internal diameter of 7 mm and thickness of 4 mm. Each material was applied into its own mold with which it will be. A celluloid Mylar strip overlying a glass slide was placed under the mold and another celluloid strip and glass slide over the mold after packing the composite material inside the mold in one increment and hand pressure was applied to get a flat sample surface and to remove excess material. Samples were light cured using high intensity Elipar[TM] LED light curing unit (3M ESPE) and the curing time applied according to the manufacturers' instructions. The testing surface (the surface facing the light-curing unit) was marked with a number engraved on the mold using rotary round bur. The samples were not removed from the molds and the tests were done with the molds as a standard background.

An LED radiometer by Demetron was used to measure the output of the light-curing unit and ensure a constant output of 1,375 mw/cm[2] throughout the samples preparation procedure. Five samples were prepared from each material for each test with a total of 15 samples/ test and a total number of 45 samples were prepared. For each resin composite material, the

samples testing time was divided into 3 groups: Baseline, 1-week and 1-month incubation groups. The same samples tested at baseline were retested after incubation in storage media for 1-week and for 1-month.

Incubation in Storage Media:

For the contact angle and surface roughness tests, the samples were incubated in RedBull storage media. For the color test, the samples were incubated in black coffee storage media. The samples were placed in an incubator at 37°C to simulate body temperature for 1 week then readings were taken for the 3 tests and the samples were incubated again to complete 1-month. The solutions were replaced with fresh ones 3 times/week. The pH of the fresh solutions was measured using pH meter (Ultra Basic Ph/mv meter; Denver Instrument, U.S.A) and the pH was found to be 5.04 for coffee and 3.56 for RedBull.

Samples Testing:

The contact angle measurements were taken using drop shape analyzer (DSA 100 Drop shape analyzer; KRUSS, Germany) using distilled water drop on the unmarked surface at 18°C room temperature. One sessile drop was applied on each sample and a built in digital protractor was set to automatically measures the right and left contact angles after 5 seconds from applying the drop. The mean of the right and left angle was also automatically calculated. The contact angle measures were taken for the samples at baseline, after 1-week, and after 1-month immersion and incubation in RedBull. The mean contact angle of the 5 samples for each group and each material were calculated.

The surface roughness test was done using a none-contact-optical profilometer (Contour GT profilometer; BRUKER, Germany). The testing surfaces of the samples were polished before testing at baseline using a polishing wheel machine (Grinder polisher Metasery 250; BUEHLER, U.S.A.). The Ra (surface roughness) values were measured for the 5 samples for each material and the mean Ra value was calculated for each material at each testing time (baseline, 1-week and 1-month incubation in storage media). The incubated samples were washed for 5 seconds with tab water and dried using gauze before testing.

The colors of all specimens were measured with a spectrophotometer (Minolta Chromameter, Kanica, Japan) according to the Commission Internationaled' Eclairage (CIE) L*a*b* relative to the standard illuminant D65. Before measuring each group, the spectrophotometer was calibrated with standard calibratingblocks according to the manufacturer's recommendation against white background.

Color change with coffee is measured using the color differences (ΔE) according to the immersion time in coffee. ΔEbwmeans ΔE between the baseline and 1-week immersion specimens, and ΔEbmmeans ΔE between the baseline and 1-month immersion specimens. ΔEbwand ΔEbmwere calculated using the following formula:

$$\Delta Ebw = \{(L*w - L*b)^2 + (a*w - a*b)^2 + (b*w - b*b)^2\}^{1/2}$$
$$\Delta Ebm = \{(L*m - L*b)^2 + (a*m - a*b)^2 + (b*m - b*b)^2\}^{1/2}$$

L* represents the degree of gray corresponding to a lightness, a* is the red (+)-green (-) axis, and b* is the yellow (+)-blue (- b*) axis (Kim, 2015).

Statistical Analysis:

One-way ANOVA test statistics was done to compare between the mean values for the 3 materials with p value less than 0.05. Least significance test was followed to compare between each two materials in case there were statistical significant results. Paired t test was used to compare between baseline Vs 1 week and baseline Vs one month in each group.

3. Results:

The contact angle results of the 3 materials at baseline and after 1 week and 1 month incubation in RedBull storage media were calculated and data were expressed as mean and standard deviation (table 2). One-way ANOVA test statistics was done to compare between the mean values for the 3 materials with p value less than 0.05. Least significance test was followed to compare between each two materials. Paired t test was used to compare between baseline Vs 1 week and baseline Vs one month in each group.

There was no significant difference in the contact angle measurement between the 3 bulk fill resin composite materials at baseline and after 1 month incubation in RedBull storage media. There was a low significant difference between 3M and Voco and also between SonicFill and Voco after 1 week incubation; Voco had lower contact angle measurement. For all 3 materials, there was a low significant difference in the contact angle measurement between baseline and 1 week; there was a decrease in the contact angle measurement for all 3 materials at 1 week. There was an intermediate significant difference in the contact angle measurement between baseline and 1 week for all the 3 materials; the contact angle measurement decreased significantly after 1 month incubation in RedBull storage media.

Surface roughness results for the 3 materials at baseline and after 1 week and 1 month incubation in RedBull were recorded.Data are expressed as mean and standard deviation(table 3).One-way ANNOVA test was done to compare between the three groups if p value was less than 0.05. Least significance test was followed to compare between each two groups. Paired t test was used to compare between the baseline Vs 1 week and between baseline Vs 1 month for each group.

Table (2): The mean and standard deviation data for the contact angle results of the 3 materials at baseline and after 1 week and 1 month incubation in RedBull storage media.

	3M X ± SD	Voco X ± SD	SonicFil X± SD	*P* value
Baseline	78.89±4.04ᵃ	78.72±5.58ᵃ	80.55±4.87ᵃ	*P* =0.86 NS
1 week	**d=0.038**∞ 68.46±7.14ᵃ	**d=0.036**∞ 57.12±11.55ᵇ	**d=0.044**∞ 70.34±3.77ᵃ	*P* =0.039∞ **Significant**
1 month	**e‹0.001**∞∞ 48.91±8.56ᵃ	**e‹0.001**∞∞ 49.52±24.13ᵃ	**e‹0.001**∞∞ 54.02±4.43ᵃ	*P* =0.36 NS

NS = not significant. ᵃ,ᵇmaterials having different superscripted letters are significantly different from each other.d (baseline Vs 1 week), e (baseline Vs 1 month). ∞,∞∞and ∞ represent low, intermediate and high significance.

Table (3):The mean and standard deviation data for the surface roughness (Ra) results of the 3 materials at baseline and after 1 week and 1 month incubation in RedBull storage media. NS = not significant.

	3M X ± SD	Voco X± SD	SonicFill X± SD	*P* value
Baseline	279.1±36.69	436.2±118.4	345.91±134.71	*P* =0.175 NS
1 week	334.1±87.6	477.4±112.62	504.91±434.61	*P* =0.53 NS
1 month	276.9±46.11	463.3±105.62	400.4±205.2	*P* =0.130 NS

NS = not significant.

There was no significant difference in the Ra (surface roughness) values between all the 3 materials at each incubation times. Each material had no significant difference in the Ra (surface roughness) values between baseline, 1 week and 1 month incubation in RedBull.

Table (4) lists the CIE L*a*b* color coordinates of the resin composites at baseline, after immersion in coffee for 1 week and for 1 month. Data were expressed as mean and standard deviation. One way ANNOVA test was done to compare between the three groups if p value was less than 0.05. least significance test was followed to compare between each two groups.Paired t test was used to compare between baseline Vs 1 week and baseline Vs 1 month in each group. Table (5) lists the mean ΔE (ΔEbw and ΔEbm) values.

Table (4): The CIE L*a*b* color coordinates of the 3 resin composites at baselineand after immersion in coffee for 1 week and for 1 month.

		3M X ± SD	Voco X± SD	SonicFill X± SD	*P* value
Baseline	a*	-0.77 ±0.33	-0.092 ±0.46	-1.01 ±0.102	*P* =0.53
	b*	5.99 ±2.37	6.29 ±3.04	8.73 ±0.23	*P* =0.147
	L*	82.75 ±1.34	82.92 ±1.06	*b=0.026∞* *c=0.013∞* 81.09 ±0.39	*P* =0.025∞
1 week	a*	*a=0.002∞∞* *b=0.041∞* 0.62 ±0.151	-0.45 ±0.201	-0.205 ±0.28	*P=0.006∞∞*
	b*	*d‹0.001∞∞* 7.64 ±1.61	*d<0.001∞∞* 6.87 ±1.79	*d‹0.001∞∞* 8.78 ±0.129	*P* =0.137
	L*	79.55 ±1.79	80.93 ±1.25	78.89 ±0.46	*P* =0.0.77
1 month	a*	0.402 ±0.25	0.18 ±0.22	0.41 ±0.18	*P* =0.24
	b*	6.5 ±1.53	6.90 ±1.04	7.83 ±0.37	*P* =0.18
	L*	*e‹0.001∞∞∞* 78.06 ±2.2	*e‹0.001∞∞∞* 78.36 ±2.14	*e‹0.001∞∞∞* 75.83 ±0.57	*P* =0.092

∞,∞∞, and ∞∞∞ represent low, intermediate and high significant difference respectively. a is significant difference between 3M and Voco, b is significant difference between 3M and SonicFill, c is significant difference between Voco and SonicFill. d is significant difference between baseline and 1 week, and e is significant difference between baseline and 1 month.

At baseline, there was a low significant difference in the L* value between SonicFill and both 3M and Voco. There was no significant difference in the L* value between 3M and Voco and no

significant difference in the a* or b* values between the 3 materials at baseline.

At 1 week, there was an intermediate significant difference in the a* value between 3M and both Voco and SonicFill. There was no significant difference in the a* value between Voco and SonicFill and no significant difference in the L* or b* values between the 3 materials at 1 week.

At 1 month, there was no significant difference in the L*a*b* values between the 3 materials.

There was an intermediate significant difference in the b* value between baseline and 1 week for all the 3 materials. There was no significant difference in the L* or a* values between baseline and 1 week for all the 3 materials.

There was a high significant difference in the L* value between baseline and 1 month for all the 3 materials. There was no significant difference in the a* or b* values between baseline and 1 month for all the 3 materials.

Table (5):Color difference (ΔE) values for the different immersion periods in coffee.

	3M	Voco	SonicFill
ΔEbw	3.86	2.10	2.34
ΔEbm	4.86	4.61	5.47

ΔEbw is ΔE between the baseline and 1-week immersion specimens, and ΔEbm is ΔE between the baseline and 1-month immersion specimens.

The formula used to calculate the ΔE is the CIE76 formula in which a ΔE of about 2.3 corresponds to a JND (just noticeable difference) (Sharma, 2003). From table (5), there was a just noticeable color difference between baseline and 1 week for 3M and SonicFill. There was also a noticeable color difference between baseline and 1 month for all the 3 materials.

4. Discussion:

The contact angle results showed that the contact angle decreases after incubation in RedBull sports drink and this decrease is directly proportional to the incubation time period. This means that the acidic RedBull solution changed the surface characteristics of the resin composite material and thus its surface energy with an increase in the wettability of the surface. This will lead to restoration surfaces with more susceptibility to plaque and bacterial retention with increase consumption of sports drinks. Voco had significantly lower contact angle measurement compared to 3M and SonicFill at 1 week but there was no significant difference between the 3 materials at 1 month and all the 3 bulk fill resin composite materials had significantly lower contact angle measurements. This means that Voco had earlier surface changes to acidic environment but this change is slower so that at 1 month there was no significant difference compared to the other materials.

Ra (surface roughness) values were similar for all the 3 bulk fill resin composite materials at all measuring times. There was no significant change in the Ra values after incubation in RedBull for 1 week or 1 month. RedBull sports drink did not affect the surface roughness of these restorative materials. It is important to consider the limitation of this study, as it does not simulate the oral environment where the restoration surfaces are subjected to chewing occlusal

forces and friction with food. A study by Wongkhantee et al showed that there was a significant decrease in surface hardness of resin composite restorative materials with acidic drinks such as sports drinks, which has a significant effect on the erosive potential of these restorations (Wongkhantee, 2006). From Wongkhantee study, there might be an indirect effect of the acidic sports drinks on the surface roughness by decreasing the surface hardness making the surface more prone to abrasion and scratches by occlusal and frictional forces thus increasing the surface roughness.

CIE L*a*b* color coordinates of the 3 bulk fill resin composites at baseline showed that SonicFill had lower L* value which corresponds to darker shade, but this difference was of low significance. At 1 week, 3M had significantly higher (+) a* value which means the a* color parameter was more towards the red axis. Also at 1 week, the b* value for all the 3 materials was significantly higher compared to the baseline, which means that the b* color parameter for all 3 materials was more toward the yellow axis after 1 week incubation in coffee. At 1 month, there was no significant difference in all color parameters for all 3 materials. Also at 1 month, the L* value for all 3 materials was significantly lower compared to the baseline, which means all 3 materials became darker after 1 month and all 3 bulk fill resin materials were stained similarly after 1 month incubation in coffee.

From the data of the color difference (ΔE), the color changes between baseline and 1 week (ΔE_{bw}) for 3M and SonicFill were higher 2.3, whichcorrespond to the JND value. The color changes between baseline and 1 month (ΔE_{bm}) for all 3 materials were higher than the JND value. This means that all 3 bulk fill resin composite restorative materials change color after incubation in coffee to a noticeable degree, which increases with increasing the incubation period.

In the oral environment, these beverages are not continuously in contact with the restorative materials surfaces and the washing effect of saliva and food was not tested in this study. Also the effect of mechanical forces placed on the restoration might amplify the effects of these beverages on the restoration surfaces. Considering a more oral simulation environment under which these materials can be tested is important to reach more clinically relevant values and a significant clinical recommendation.

Conclusion:

Within the limitation of this study, the tested 3 bulk fill resin composite restorative materials showed changes in the surface and optical properties with different storage media. Sports drink (RedBull) increased the wettability of the materials surface, which was directly proportional to the incubation time but did not have a direct effect on surface roughness of the materials. The use of black coffee as a storage media showed noticeable significant staining of the materials. Testing in a more oral simulation environment is needed to reach clinically relevant conclusion.

Acknowledgment:

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6/25/2015