



## Colour Changes of Tooth-Coloured Restorative Dental Materials Immersed in Food Simulating Solution

Razak A<sup>1</sup>, Long A<sup>1</sup>, Sivananda S<sup>1</sup>, Bhatia S<sup>2</sup>, Kohli S<sup>3</sup>, Subramaniam R<sup>4</sup>

<sup>1</sup> Year 5 Student, Faculty of Dentistry, MAHSA University, Malaysia

<sup>2</sup> Lecturer, Department of Conservative Dentistry, Faculty of Dentistry, MAHSA University, Malaysia

<sup>3</sup> Lecturer, Department of Prosthetics Dentistry, Faculty of Dentistry, MAHSA University, Malaysia

<sup>4</sup> Associate Professor, Department of Family Dentistry, Faculty of Dentistry, MAHSA University, Malaysia

### ABSTRACT

The study examines the surface staining of two different types of photopolymerised composite by tea. Thirty samples each from two generations of composite with different filler particle sizes, Filtek Z250 (3M ESPE) microhybrid and Filtek Z350 XT (3M ESPE) nanohybrid of A2 shade were subjected to an experimental 7-day staining period with each of them immersed in a solution containing 75gm of Lipton tea and 15gm of sugar and kept in 37° Celsius to simulate the temperature of the oral cavity.

After 1,3,5 and 7 days, visual examination of all samples were done to detect colour difference of each sample by comparison, using the Lobene Stain Index, which uses a grading system ranging from 0 to 3.

It was found that both restorative material used in this study were susceptible to staining by tea. However, Filtek Z350 XT showed more resistance to staining, while Filtek Z250 produced more stained samples. The colour changes of both groups of samples were found to be statistically significant only for day one of the study ( $p < 0.05$ ). However, the results showed insignificant findings on the 3rd, 5th and 7th day ( $p > 0.05$ ).

In conclusion, composite restorative materials are susceptible to staining regardless of the composition and particle size, but the degree in which it affects the aesthetic value is dependent upon specific composition of said material and other external conditions.

**Key Words:** Colour, Composite, Tea solution, Staining

Please cite this article as: Razak A et al. Colour changes of tooth-coloured restorative dental materials immersed in food simulating solution. Malaysian Dental Journal 2013; 35(Suppl. APDC): 12-18.

### INTRODUCTION

Aesthetic failure is one of the most common reasons for the replacement of restorations.<sup>1</sup> Currently, resin-based composite (RBC) materials are the most prominent esthetic restorative materials because of their universal usage, minimal loss of tooth structure, and ability to be directly placed without laboratory procedures.<sup>2</sup>

A good combination of the tooth colour and the initial colour of the material before curing is an important clinical factor for a successful outcome. More importantly, this combination must remain after the material is completely cured and throughout the life time of the restoration. Even though manufacturers found a breakthrough in light-cured formulations of composite materials,

which have considerably better aesthetic quality due to the exclusion of benzoyl peroxide in its composition, most users note discolouration of restorative material over a period of years. This still pose as a major problem for aesthetic restorations.<sup>3-5</sup>

Discolouration of tooth-coloured, resin-based materials may be caused by intrinsic or extrinsic factors. The intrinsic factors in the deeper layers of the materials involve a variety of factors related to their composition.<sup>1,3</sup> More specifically, the type of photoinitiator, nature of resin matrix, filler content, filler particle size distribution are related to colour stability.

Colour stability is also influenced by the intensity and duration of polymerization and, consequently, by the degree of conversion. Staining

of composite materials could be a consequence of (1) alteration of resin matrix, (b) alteration in the interface between matrix and fillers, (c) change in tertiary amines in composite under heat and light, (d) oxidation in structures of polymer matrix, and (e) oxidation of unreacted pendant methacrylate group.

In addition to that, water accumulation has also been reported to be responsible for internal color change. Water plays an important role in chemical degradation process such as oxidation and hydrolysis and the subsequent change of the optical properties of the provisional restorative materials.

The extrinsic factor that contributes to staining of composite are : (a) production of coloured components in plaque by chromogenic bacteria, (b) retention of coloured substances from dietary constituents passing through the oral cavity, and (c) formation of coloured products from the chemical transformation of pellicle components. Factors such as diet (coffee, tea, and red wine), smoking, exposure to antimicrobial agents, and

daily teeth cleaning affect extrinsic stain development.<sup>6-9</sup>

This study focuses on evaluating the colour changes of two different tooth-coloured restorative dental materials immersed in food simulating solutions (i.e. tea) that have been subjected to accelerated aging, and if possible, to relate staining capability of said materials to the difference in their composition.

### AIMS AND OBJECTIVES

There are two main aims and objectives of this research project are as follows:

- To evaluate the degree of staining of two different composites (3M Filtek Z 250 & 3M Filtek Z 350 XT ) in a food simulating solution ( Tea ).
- To evaluate the effect of duration on the degree of staining in the composites immersed in a food simulating solution.

**Table 1. Composites used in the study**

Product Name	Material Type	Composition
FILTEK Z350 XT	Nano	Resin: BIS-GMA, BIS-EMA (6), UDMA with small amounts of TEGDMA. Filler: a combination of non-agglomerated/non-aggregated 20nm silica filler, non agglomerated/ non aggregated 4 to 11 nm zirconia filler, and aggregated zirconia/silica cluster fillers (comprised of 20nm silica and 0.004µm to 0.011µm zirconia filler particles).
FILTEK Z250	Micro Hybrid	Z250 resin is based on the Z100 system which consists of BisGMA (Bisphenol A diglycidyl ether dimethacrylate) and a low viscosity resin called TEGDMA (tri[ethylene glycol] dimethacrylate). Contains a blend of UDMA (urethane dimethacrylate) and Bis-EMA (Bisphenol A polyethylene glycol dietherdimethacrylate). The cluster particle size range is 0.01µm to 3.5µm.

### MATERIALS AND METHODS

#### Materials

1. Restorative material (Table 1)
  - Filtek Z250 ( 3M ESPE )
  - Filtek Z350 XT ( 3M ESPE )
2. Drinking straws
3. Light curing unit ( Motion Blue Life LED – 320 )
4. Carborandom disc ( 25 x 0.3 mm Separating Disc Secudisk )
5. Opti Discs ( OptiDisc – Kerr – Course/ Medium, Fine, Extra – Fine )
6. Deionized Distilled Water (DDW)
7. Tea Powder / Tea Bags ( Lipton )

8. Incubator
9. Sugar ( Prai – Granulated Sugar )
10. Electronic Digital Caliper ( Kern Germany )
11. Measuring Cylinder – 100 ml
12. Electronic Balance ( A & D Company Limited ) – Fx – 300i
13. Hand Instruments : Plugger, Ball burnisher, Metal ruler

#### Methodology

This is an in vitro study that has been carried out at MAHSA University. The restorative materials tested in this study are Filtek Z250 (3M ESPE ) and Filtek Z350 XT (3M ESPE). 60 samples are prepared with

30 samples for each group. The restorative material is then packed into drinking straws with a diameter of 13 mm and thickness of 1 mm. These specimens were then light – cured for 40 seconds with a light curing unit (Motion Blue Life LED – 320) and then sectioned with a carborandom disc (25 x 0.3 mm Separating Disc Secudisk) according to 1 mm thickness to fabricate a total of 60 individual discs.

The samples were then polished with Opti Discs (OptiDisc – Kerr) from coarse or medium to fine and then finally extra - fine. This is done by the same operator to reduce variability.

All the 60 specimens are placed in a container containing deionized distilled water (DDW) at 37°C for 24 hours as a baseline measurement. After 24 hours, the specimens were taken out from the deionized distilled water and a baseline colour measurement will be made after blot – drying the specimens.

All the 30 samples from both groups ( 60 samples ) will be immersed into tea solution (Lipton Brand) where each solution is prepared using 15gm of tea powder, 10 gm sugar (PRAI Brand – Granulated Sugar) and 100ml of hot water. All the discs will be immersed into the solutions after the solution becomes cold.

The specimens are then kept in each solution for a period of 1 day, 2 days, 3 days, 4 days, 5 days, 6 days and 1 week. This time period is done to stimulate the period of continuous exposure and the specimens will be stored in an incubator at 37°C.

After each period, the discs from each sub group were removed and irrigate under running water for 20 seconds and blotted dry with tissue paper before colour measurement. Stains were assessed visually and recorded using Lobene Stain Index10 (which is based on these four scores):

- 0 = no stain
- 1 = light stain (yellow to light brown or gray)
- 2 = moderate stain (medium brown)
- 3 = heavy stain (dark brown to black)

For this assessment, each sample is measured by two investigators which are by 2 researchers. Firstly, one researcher will assess the results and then the second researcher will assess the results. These results are then combined and an average result of both will be taken.

## RESULTS

### Statistical Analysis

The statistical analysis was analyzed using Independent t-Test.

Mean value was calculated separately for Filtek Z250 and Filetk Z350 XT for day 1, 3, 5 and 7. Generally, Filtek Z250 marked a higher mean value compared to Filetk Z350 XT for all the days.

The formula for the sample mean is as follows:

$$x = \frac{\sum_{i=1}^n xi}{n}$$

The mean value obtained for the composites where then used to calculate standard deviation.

$$S = \sqrt{\frac{\sum (x - \bar{x})^2}{n - 1}}$$

Standard error was calculated,

$$\sigma = \sqrt{\left(\frac{1}{n} + \frac{1}{n}\right)}$$

Pooled SD were calculated,

$$S_p = \sqrt{\frac{(n - 1)(S) + (n - 2)(S)}{n + n - 2}}$$

And all the values obtained were used to calculate t-Test:

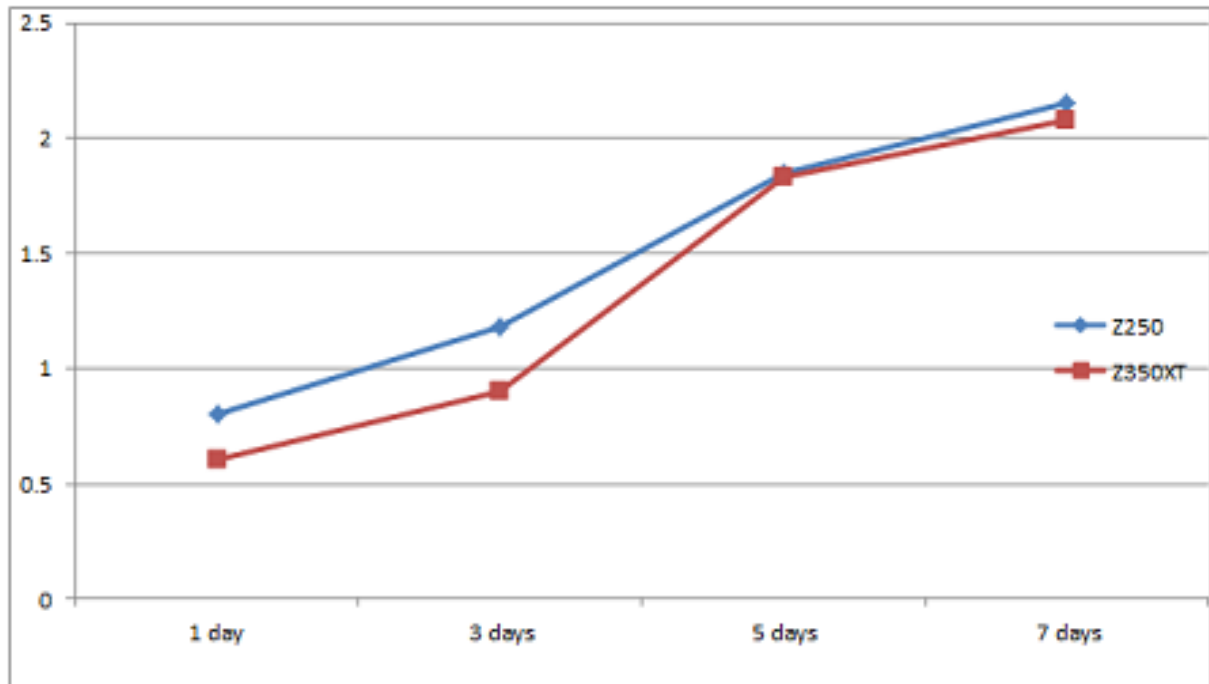
$$t = \frac{\bar{x}_1 - \bar{x}_2}{\sqrt{\frac{S_1^2 + S_2^2}{\chi}}}$$

The values obtained were summarized in Table 2 and Graph 1 explains the degree of staining based on the mean value obtained for Filtek Z250 and Filtek Z350 XT for 1 day, 3 days, 5 days and 7 days.

**Table 2. Comparing the colour changes between 3M Filtek Z250 and 3M Filtek Z350 in the solution**

Time	Composite Resin	Mean (SD)	T-value	P-value
1 day	Z250	0.80 (0.363)	2.0513	< 0.05 (Significant)
	Z350XT	0.60 (0.392)		
3 days	Z250	1.18 (0.737)	1.657	> 0.05 (Not Significant)
	Z350XT	0.9 (0.563)		
5 days	Z250	1.85 (0.577)	0.2389	> 0.05 (Not Significant)
	Z350XT	1.83 (0.562)		
7 days	Z250	2.15 (0.645)	0.412	> 0.05 (Not Significant)
	Z350XT	2.08 (0.671)		

**Graph 1. Colour changes for 3M Filtek 250 and 3M Filtek 350XT in tea solution**



## DISCUSSION

Tea has been proven to play a significant role in causing discolouration of composite resins through the act of adsorption or absorption. Staining solutions and immersion time were significant factors that affected colour stability of the composite resins after polishing. 3M ESPE Filtek Z350 XT, which is a nanohybrid, was chosen because it is currently the choice of composite restorative material used in many dental clinics. The other composite resin studied was 3M ESPE Filtek Z250 Universal Restoration, a microhybrid composite from the same manufacturer.

For Filtek Z250, which is the predecessor to Filtek Z350XT, the 3 major components of composite restorative material are retained from the previous make of composite from the same brand (Filtek Z100). Here, the majority of TEGDMA

has been replaced with a blend of UDMA and Bis-EMA. Both these compounds are of high molecular weight, and therefore have lesser double bonds per unit of weight. The high molecular weight results in less shrinkage, decreased aging, and a slightly softer matrix. These resins impart a greater hydrophobicity and are less sensitive to changes in atmospheric pressure.<sup>5</sup> Filler particle size for this make is also a breakthrough in the microhybrid region, with particle zirconia/silica particle distribution ranging from 0.01µm to 3.5 µm. The average size is around 0.6 µm.

Filtek Z350 XT Universal Restorative is a visible light-activated composite designed for use in anterior and posterior restorations. This material consists of three primary ingredients: an organic matrix, inorganic filler particles, and a coupling agent. Other ingredients include colour stabilizers, pigments, and an activation system. The organic

resin matrix contains mainly UDMA ((urethane dimethacrylate) and Bis-EMA (Bisphenol A polyethethyleneglycol dietherdimethacrylate). UDMA and Bis-EMA resins are of higher molecular weight than TEGDMA and therefore have fewer double bonds per unit of weight. The higher molecular weight of the resin results in less shrinkage, improved aging and a slightly softer resin. TEGDMA and PEGDMA are used in minor amounts to adjust the viscosity. PEGDMA (hydrophobic) was used to replace part of the TEGDMA (hydrophilic) component to moderate shrinkage in Filtek Z350 XT restorative.<sup>11</sup>

As for the filler particles, the manufacturer used a combination of non-agglomerated/ non aggregated 0.020  $\mu\text{m}$  silica filler, non agglomerated/ non aggregated 0.004 to 0.011  $\mu\text{m}$  zirconia filler, and aggregated zirconia/silica cluster fillers (comprised of 20nm silica and 4 to 11 nm zirconia filler particles).

In this study, tea was chosen as this beverage is a staple drink in Malaysians' daily dietary habits. Tea contains Tannin (tannic acid), a substance which gives it its yellowish-brown appearance. Tannin has been found to be the source of discolouration in composite resins. The presence of tannic acid causes swelling of the restoration, thereby manipulating the susceptibility of the composite surface to staining.<sup>12</sup>

Sugar was added because the presence of sugar in coffee and tea increased the colour difference compared to coffee or tea without sugar for light-polymerised composite material and microhybrid composites.<sup>8,10</sup> Also, most Malaysians generally consume tea with sugar as accompaniment.

The omission of milk in this study is due to established laboratory studies showing the protective nature of milk against tooth erosion. Milk contains a high concentration of calcium, which, along with phosphate, may have a protective effect on tooth enamel and consequently resist deterioration of tooth surface. Cow's milk has shown that it can strengthen tooth enamel by remineralisation after exposure to acidic drinks.<sup>13</sup> Therefore, even though tea is acidic and milk is alkaline in nature, it is thought that the presence of milk in the solution would offset, if not mar the actual extent of discolouration tea would have on the samples.

Low consumption of milk was also a reason behind the exclusion of milk from this study, which is concurrent with a data from the national survey among adults in Malaysia in 2006. The survey reports that the average consumption of milk among adults was 0.14 serving per day (1 serving = 250 mL; 0.14 serving= 35 mL) compared to the recommendation of one to two servings per day. Only 17.1% of the adult population consumed milk with the frequency of 1.4 times daily.<sup>8</sup>

Samples were kept at 37° Celcius constantly in incubator throughout the entire study to simulate the average temperature in the oral cavity. Although this contradicts the fact that most people prefer hot tea, it was presumed that the temperature of the beverage drops soon after consumption, therefore, the samples remain at normal human body temperature for most of the period. Furthermore, previous studies on this topic also warranted the selection of the temperature above.<sup>8,10</sup>

The duration of the study considered the cumulative effect of months or years of tea consumption, which elucidates the constant exposure of samples to the staining solution. It purely deliberated the extent of staining on composite materials, excluding the effect normal act of oral hygiene routine such as tooth brushing and the use of mouth rinses.

For the purpose of synchronizing and standardizing the finished products, polishing was done by a sole operator, using polishing discs (OptiDisc by Kerr) ranging from coarse/medium, fine, to extra fine. A high-gloss surface is generally considered lesssusceptible to staining.<sup>10</sup> Rough surfaces mechanically retain surface stains more than smooth surfaces. It can be concluded that the rougher the finished surface, the more vulnerable the restoration is to bacterial and plaque accumulation.<sup>11</sup> Also, a poorly-finished restoration leads to increased surface area for adsorption of pigments from food, allowing discolouration along marginal lines of the restoration to occur.<sup>6,11</sup>

Discolouration of composite resins can be evaluated via various instruments. In this study, Lobene (1968)<sup>14</sup> Stain Index was used for assessment of the extent of staining by this beverage. The score was given accordingly from the lightest to the darkest based on four scores from 0 to 3.<sup>10</sup> This method was chosen due to the

limitations of the research team to utilise more accurate equipment such as a spectrophotometer. It has been found that the human eye is best in detecting colour difference by comparison. The National Bureau of Standards (NBS) established a sophisticated classification to describe colour change difference by NBS units. They say that the acceptable limit for the colour shift is  $\Delta E < 3.3$ . Any value below this mark is considered to be of acceptable colour shift. The human eye can perceive a colour shift at  $\Delta E < 1$ . Therefore, the use of Lobene Stain Index, although permitting discrepancies, is an acceptable method to gauge colour difference between the two samples.<sup>6</sup>

Staining is highly influenced by each composite monomer and filler composition. In general, the hydrophobic materials showed greater colour stability and stain resistance than the hydrophilic materials. The difference of colour stability in this study might be due to the smaller nanoparticles in Z350XT which resulted in less staining absorption into this composite resin.<sup>12,15</sup>

In addition to matrix content and composition, filler size and distribution may also influence water discolouration, presumably through hydrolytic degradation of the filler-matrix interface and modification of the way light is scattered by the particles. The addition of fillers reduces polymerization shrinkage, coefficient of thermal expansion, and water sorption.<sup>12,16</sup>

Water can penetrate the matrix or matrix-filler interface, and, in the case of this study, brings along the tea molecules with it to bind to the composite surfaces. The variation in the water sorption rate between materials using Bis-GMA matrix may be due to the different proportions of TEGDMA. Materials exhibiting high water sorption values are more easily stained by hydrophilic colourings in aqueous solutions, as the water will act as a penetrating vehicle. Composites which are mostly hydrophilic will then, allow water molecules to penetrate the matrix or filler-matrix interface.

Another possible cause of staining is the presence of residual camphorquinone, which is added to the composite resin materials as a photo-initiator. Colour stability is influenced by the intensity and duration of polymerization and the degree of conversion. Prolonged polymerization duration (prolonged light-curing) may increase the degree of conversion and thus, decrease

discolouration of composite resins containing TEGDMA.<sup>1</sup>

## CONCLUSION

The colour stability of two composites restorative materials (Filtek Z250 and Filtek Z350 XT) were evaluated after 1 day, 3 days, 5 days and 7 days of immersion in staining solution (tea in this case). Within the limits of this study, the following conclusions were drawn:

1. Filtek Z250 (Universal resin composite) exhibited increased staining as compared to Filtek Z350 XT, due to its filler particle size.
2. In Filtek Z350 XT the addition of PEGDMA with TEGDMA reduces shrinkage thus increasing the resistance towards staining.
3. For both resin composite restorative materials tested, it is noteworthy that both material contained TEGDMA showed discoloration, meaning that TEGDMA was responsible for the discolouration due to its hydrophilic character. In clinical practice, patients should be aware of the staining effects of the drinks tested in this study, while practitioners should take into consideration the staining susceptibility of the resin composite. Further studies need to be undertaken to unravel the mechanisms of staining, whereby investigations will require more complex but more informative methods to measure colour change.

## REFERENCE

1. Barutçigil C and Yıldız M. Intrinsic and extrinsic discolouration of dimethacrylate composites and silorane." *Journal of Dentistry* (January 2012).
2. Dietschi D, Campanile G, Holz J and Meyer JM (1994). Comparison of the color stability of ten new-generation composites: an in vitro study. *Dent Mater*, 10(6):353-62.
3. Ertaş E, Güler AU, Yücel AC, Köprülü H and Güler E (2006). Color stability of resin composites after immersion in different drinks. *Dent Mater J*, 25(2): 371-76.
4. Ghahramanloo A, Madani A S, Sohrabi K and Sabzevari S (2008). An Evaluation of Color Stability of Reinforced Composite Resin Compared With Dental Porcelain in

- Commonly Consumed Beverages. *CDA Journal*, 36(9): 673-80.
5. Gupta G and Gupta T (2011). Evaluation of the effect of various beverages and food material on the color stability of provisional materials – An in vitro study. *J Conserv Dent*, 14(3): 287–92.
  6. Omata Y, Uno S, Nakaoki Y, Tanaka T, Sano H, Yoshida S, Sidhu SK (2006). Staining of hybrid composites with coffee, oolong tea, or red wine. *Dental Material Journal*, 25(1):125-31.
  7. Gross MD and Moser JB (1977). A colorimetric study of coffee and tea staining of four composite resins. *J Oral Rehabilitation*, 4:311-22.
  8. Guler AU, Yilmaz F, Kulunk T, Guler E and Kurt S (2005). Effect of different drinks on stainability of resin composite provisional restorative materials. *J Prosthet Dent*, 94(2):118-24.
  9. Mohan M, Vaidyanathan J, Janal M, Munisamy S, Vaidyanathan TK, Shey Z (2008). Color changes of restorative materials exposed invitro to cola beverages. *Pediatr Dent*, 30: 309-16.
  10. Ibrahim MA, Wan Bakar WZ and Husein A (2009). A comparison of staining resistant of twocomposite resins. *Archives of Orofacial Sciences*, 4(1): 13-16.
  11. Khokhar NK, Qureshi R and Ali SM (2009). Evaluation of discolouration of some composite restorative materials. *Pakistan Oral & Dental Journal* , 29(1):123-30.
  12. Topcu FT , Sahinkesen G, Yamanel K, Erdemir U, Oktay EA and Ersahan S (2009). Influence of Different Drinks on the Colour Stability of Dental Resin Composites. *European Journal of Dentistry*, 3(1) : 50-6.
  13. Gedalia I, Dakuar A, Shapira L, Lewinstein I, Goultshin J and Rahamim E (1991). Enamel softening with coca-cola and rehardening with milk or saliva. *Am J Dent*, 4(3):120-2.
  14. Lobene RR (1968). Effect of dentifrices on tooth stains with controlled brushing. *J Am Dent Assoc*, 77(4): 849-55.
  15. Villalta P, Lu H, Okte Z, Godoy FG, Powers JM (2006). Effects of staining and bleaching on color change of dental composite resins. *J Prosthet Dent*, 95:137-42.
  16. W M Johnston, M H Reisbick “Color and translucency changes during and after curing of esthetic restorative materials” *Academy of Dental Materials* 1997; 13(2): 89-97.