## EVALUATION OF DISCOLOURATION OF SOME COMPOSITE RESTORATIVE MATERIALS

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#### ABSTRACT

This in-Vitro study was conducted to investigate the stain resistance of direct composite resin restorative materials with different polishing systems (diamond finishing and polishing burs, aluminium oxide disks and abrasive impregnated disks) in comparison to the material cured against a mylar strip. The composite resin materials investigated included Clearfil AP-X (hybrid), Filtek Z-250 (hybrid), Definite (Ormocer), and Silux Plus (microfill).

Forty disk specimens (10 mm x 2 mm) of each composite resin were prepared. Ten mylar cured specimens were assigned for each polishing system and ten were used as controls with no further treatment after curing.

A spectrophotometer (Spectraflash 500) was used to determine the initial colour and the colour change after one week immersion. All specimens were stored in tea separately for one week at 39 °C. Post immersion colour measurements were undertaken to assess colour change ( $\Delta E$ ).

One way ANOVA showed that there was statistically significant difference between the different composites and the polish groups for discoloration at p < 0.05. All the composites tested showed significant discoloration when polished with Diamond burs. The least discoloration was found when specimens were polished with soflex except with Z-250 which discoloured less under mylar than soflex but the difference was not significant. Among the materials tested AP-X showed the least discoloration.

Key words: Composites, discolouration

## INTRODUCTION

Aesthetics plays a major role in the development of dentistry and dental research. The trend towards the natural look has paved the way for the development of tooth coloured restoratives that simulate the tooth as closely as possible.<sup>1</sup>

Composite resins are gaining popularity and drawing major attention as aesthetic anterior restoratives <sup>2</sup> and as a dominant alternative to amalgam for direct restoration of posterior teeth.<sup>3</sup> Composite resins being used are susceptible to discolour by taking up stains from normal diet and food dyes.<sup>4</sup> Discoloration is caused by distinct pigments that penetrate the composite resins.<sup>5,6</sup> Tannin is the component in tea, which contains tannic acid, which causes the discoloration in composite resins.<sup>7</sup> The extent of discoloration may be associated with individuals' dietary habits. As suggested the acid causes the swelling of the restoration influencing the susceptibility to surface staining. The presence of tannic acid may also induce staining by the same phenomenon.<sup>8</sup>

One of the most troublesome features of these aesthetic materials is the difficulty of finishing the

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restoration surface to decrease adherence of food debris onto the restoration that may lead to discoloration especially at the margins.<sup>9</sup> The rougher the finished surface is, the more chance there is for bacterial accumulation and discoloration of restoration along the restoration margin with secondary caries formation.<sup>10,11</sup>

To prevent the accumulation of plaque and stain pigmentation from food, a finished composite resin surface should be highly polished and very smooth.  $^{12, 13, 14, 15, 16, 17}$ 

Previously a lot of attention has been paid in the surface effects of surface roughness but not much work has been done on discoloration and stain control in newer materials.

### METHODOLOGY

Forty specimens of 10 mm diameter and 2 mm thickness were made from each composite resin and ormocer listed in Table 1 & shown in figure 1, to form four experimental groups. The composite resin was injected directly into a cylindrical polytetrafluoroethylene (PTFE) mould that was placed on a microscopic slide with a celluloid mylar strip over it. The open surface of the mould was covered with a mylar strip and a microscopic glass slide. Finger pressure was applied on top of the glass slide to remove excess material and to form a parallel plane surface. The glass slide was used to protect the composite resin from oxygen inhibition.

The resin was cured through the upper side of the glass slide for 40 seconds with a visible light activation unit (Coltolux 3 Coltené/ Whaledent Inc., USA). The plates were then reversed to cure the resin from the other side of the plate for 40 seconds. The tip of the light source was placed against the glass slide to ensure uniform distance from the light source to the composite resin. All procedures were carried out at room temperature (23°C  $\pm$ 2°C).

Each group of specimens was randomly divided into four subgroups of 10 specimens each and was randomly assigned as either the control group or to one of the three polishing systems listed in Table 2 and shown in Figure 2.

### Polishing of the samples

### 1 Polishing with aluminium oxide disks (Soflex®)

The specimens in this subgroup were polished immediately after curing. Sof-lex pop-on contouring and polishing disc system (3M Dental products, St Paul Minn USA) was used according to the manufacturer's instructions. Coarse grit was used at about 10,000 rpm for gross reduction. The surface of the filling material was then rinsed and dried. The medium grit was used at 10,000 rpm for 15 seconds followed by fine grit at 30,000 rpm for 15 seconds and superfine grit at 30,000 rpm for 15 seconds. Any debris or powder formed on the surface of the filling materials was washed away with water.

## 2 Diamond polishing burs (Komet® Germany)

The Komet diamond polishing burs were used at high speed in a sequential manner from coarse grit, medium grit, fine grit to ultra fine grit. The procedure was performed using water spray. Each bur was used for 30 seconds.

# 3 Abrasive impregnated polishing system (Enhance® Polishing System)

The Enhance polishing system (Dentsply/Caulk USA) finishing discs were used to finish the composite resin surface. Polishing was done at low speed using light pressure in a circular motion as per manufacturer's recommendation.

The surface was then polished with the Prisma gloss paste. A small amount of Prisma gloss was applied to the surface of the finishing cup. The cup worked on the surface of restoration with moderate speed and pressure, initially for 30 seconds. To increase lustre, water was added in small amounts (drop wise) to dilute the paste using a light buffering motion for 30 seconds. The paste was rinsed off from the restoration and cup. The other composite resin materials were further polished with Prisma gloss extrafine paste using the same procedure as that used with Prisma gloss. The cup was discarded after each use.

## Post polishing treatment

All specimens were observed under light microscope (Kyawa Optical Co. Ltd., Japan) under 23 x 10

Composite resin & ormocer material	Shade	Composition	Number of specimens	Manufacturer	Batch number
Silux-Plus	U	Heterogeneous microfilled	40	3M Dental products, St Paul Minn USA	Lot 19990222 Ref5702-U
Clearfil AP-X	A3	Midifilled Hybrid	40	Kuraray Co. Ltd Japan	0596AB
Filtek Z-250	A3	Minifilled Hybrid	40	3M Dental products, St Paul Minn USA	Lot 9BN Ref1370AB
Definite ormocer	A3	Minifilled Hybrid	40	Degussa AG, Geschäftsber-eich Dental Hanau	Lot 21 Ref4380003

### TABLE 1: COMPOSITE RESINS AND ORMOCER RESTORATIVE MATERIALS USED IN THE STUDY

## TABLE 2: POLISHING MATERIALS USED IN THIS STUDY

Polishing system	Manufacturer	Composition	
Sof-lex <sup>™</sup> Pop-on contouring and polishing disc system	3M Dental products, St Paul Minn USA	Aluminium oxide discs coarse, medium, fine and superfine	
Komet® Diamond polishing burs	Komet, Germany	Diamond finishing and polishing burs coarse, medium, fine and superfine	
Enhance® composite finishing and polishing system	Dentsply/Caulk USA	Abrasive impregnated silicon discs with 2 polishing pastes	
Ruwa mylar strips	Kem-Dent, UK	Polyester foil strips for uni versal use	

# TABLE 3: COMPARISON OF MEAN DISCOLORATION FOR SILUX PLUS

Polishing material	Mean discoloration $(\Delta E)$
Soflex	3.20
Enhance	3.50
Mylar	4.56
Diamond	4.90

## TABLE 5: COMPARISON OF MEAN DISCOLORATION FOR DEFINITE

Polishing material	Mean discoloration (ΔΕ)
Soflex	3.62
Mylar	4.11
Enhance	4.24
Diamond	5.29

# TABLE 4: COMPARISON OF MEAN DISCOLORATION FOR CLEARFIL AP-X

Polishing material	Mean discoloration (ΔΕ)
Soflex	2.63
Mylar	3.03
Enhance	3.06
Diamond	5.27

# TABLE 6: COMPARISON OF MEANDISCOLORATION FOR FILTEKZ-250

Polishing material	Mean discoloration (ΔE)
Soflex	3.79
Mylar	3.92
Enhance	4.55
Diamond	5.88



Fig 1: Left to right: Clearfil AP-X, Filtek Z-250, Silux Plus, Definite)

magnification to see the surface topography and the effect of polishing on the surface. Each specimen was placed in dark container filled with 20 ml distilled water individually for 24 hours.

Initial colorimetric evaluations were carried out using (Spectraflash) spectrophotometer as shown in Figure 3. The CIE (Comission Internationale de L'Eclairage) standard illuminant  $D_{65}$  was used in this study. Each sample was removed from the container, rinsed for 60 seconds and dried with an absorbent paper before placing it against the eyelet of the aperture of 6 mm diameter. The disc came in contact with the eyelet at a radius of 3 mm from the center. Five readings were taken per specimen. The average CIElab reading was computed by the software program and was recorded.

The samples were then immersed in the tea (Lipton Yellow label tea bags, Lipton Pvt Ltd, UK) solution for one week. The tea was prepared by placing two tea



Fig 2: Soflex, Enhance, Mylar, Diamond burs

a = spectrophotometer, b= calibrating box



Fig 3: Spectraflash spectrophotometer

bags in 250 ml boiling water for 5 minutes as per manufacturer's instructions. Each specimen was stored in one container at 37°C for seven days in an incubator to simulate the temperature of the oral cavity. Each container was separately marked.

On the 8<sup>th</sup> day, the samples were taken out of the tea solution, rinsed thoroughly and dried with an absorbent paper. Another colorimetric reading was performed.

The measurements of experimental group of samples were compared with regards to different surface treatments.

The difference was calculated as:

$$\Delta \mathbf{E} = [(\Delta \mathbf{L})^2 + (\Delta \mathbf{a})^2 + (\Delta \mathbf{b})^2]^{\frac{1}{2}}$$

whereby:

 $\Delta E$  = is the difference in colour





cates the post staining value and "L" = indicates the brightness (a value of 100 corresponds to perfect white and that of zero to black)

- $\Delta a = is the difference in the red green scale (a_2 a_1). a_1 indicates the pre staining value, a_2 indicates the post staining values and "a" = determines the amount of red (positive values) and green (negative values)$
- $\Delta b = determines the difference in yellow blue scale$ (b<sub>2</sub>-b<sub>1</sub>). b<sub>1</sub> indicates the pre staining value, b<sub>2</sub>indicates the post staining values and "b"=determines the amount of yellow (positivevalues) and blue (negative values).

The difference between the initial colorimetric measurement and the post immersion measurement was calculated for each specimen and used for statistical analysis.

#### RESULTS

Upon visual examination, the gloss of the polished specimens were found to be less than that of the Control (mylar cured) specimens. All specimens exhibited discoloration after one week of immersion. The light microscopy examination showed porosities on the surface of the specimens cured under the mylar strip. Superficially the extrinsic discoloration was seen along the air entrapment areas.

Statistical analysis was performed using One way ANOVA. Among the composite materials tested, Clearfil AP-X discoloured the least followed by Silux Plus, Definite and Filtek Z-250 except for mylar treated group where Filtek Z-250 discoloured less than Silux Plus and Definite. Post-hoc Scheffe's test showed that there was no significant difference (p < 0.05) found between the Clearfil AP-X, Silux Plus and Definite.

Among the polishing materials used the least discoloration was found when the specimens were polished with Soflex disks followed by mylar, Enhance and diamond burs except Clearfil AP-X (where the specimens polished with Enhance discoloured less than that of mylar) and Z-250 (where the least discoloration was found with mylar).

#### DISCUSSION

The methods currently available and used as a research methodology to measure discoloration in-

clude visual and instrumental techniques. Instrumental techniques, however, are most commonly used in research. The human eye is best in detecting colour difference by comparison.<sup>18</sup> Other detectors used in place of eye as an observer are photo-detectors like colorimeters and spectrophotometers<sup>19</sup> whose use make it possible to overcome basic difficulties of evaluation.<sup>20</sup> The National Bureau of Standards (NBS) established a sophisticated classification to describe colour differences by NBS units. A human eye can perceive a colour shift at  $\Delta E \ge 1$ . The acceptable limit for the colour shift set by NBS is  $\Delta E \le 3.3$ . Colour shift with corresponding E values less than 3.3 are acceptable.<sup>21</sup> Above this value is marked colour shift.<sup>22</sup>

Instrumental colorimetry is more authentic over visual colorimetry because descriptive visual comparisons are less reliable in making quantitative comparison of materials and treatments. Instrumental colorimetry can potentially eliminate the subjective errors of colour assessments<sup>23</sup>like observer's variability (physiological and psychological) and optical metamerism.<sup>24</sup> The advantage of spectrophotometer is that it measures the amount of light reflected by the surface within a full reflectance spectrum. The perceivable values set by NBS are DE between 1 and 3.3 NBS units. In the visual method, colour difference value greater than 2 NBS units can always be correctly judged. When the measured colour difference falls within 1 to 2 NBS units, incorrect judgements become frequent and when the measured colour difference becomes less than  $1\Delta E$ unit, the visual technique cannot distinguish the colour change.<sup>25</sup>

In this study, all  $\Delta E$  values were above 1 NBS unit. Only one observation had a reading below 2  $\Delta E$ . Majority of the readings were above 3.3 NBS units except for the Clearfil AP-X specimens polished with mylar, Soflex and Enhance and the Silux Plus specimens polished with Soflex. This indicates a severe colour change in the majority of the specimens.

It should also be noted that there were statistically significant differences (p < 0.05) in the initial colorimetric readings found between some of the restorative materials tested even though same shade A3 was used by different manufacturers. This suggests that there exist a significant difference between the CIElab value and the hue and chroma of these materials. This in turn suggests a need for improved shade standardisation.

The different polishing systems were chosen because of their popular use in clinical practice. The mylar subgroup acted as the control as it has been generally accepted that composite resin cured under a mylar strip would produce the smoothest surface. It has been found that the surface cured directly in contact with mylar discoloured more than the polished surface.<sup>26,27</sup> This was true only for Silux Plus specimens cured under the mylar strip which discoloured more than those in the Soflex and Enhance subgroups at a visually perceptible level. Filtek Z-250 specimens, on other hand cured under the mylar strip registered the lowest DE value among the subgroups. The difference in the type of composite resin may explain these findings. It should also be noted that the studies cited were made in the 1970's and 1980's where improvement on composite resin chemistry is not comparable to the materials that are available to us today. The strain that is caused on the surface of composite cured against the mylar strip increases the activity of the atoms on the surface and facilitates accumulation of stains. It has been previously found that when the resin rich layer was removed by polishing with Soflex disks the discoloration was reduced.

Every component of resin may be implicated in discoloration. The resin matrix plays major part in the colour stability of composites. The high viscosity of resin matrix is reduced by proper mixture of diluent, to help incorporate more filler loading. The variation in the water sorption rate between materials using Bis-GMA matrix may be due to the different proportions of diluent TEGDMA.<sup>28</sup> The addition of fillers reduces polymerisation shrinkage, coefficient of thermal expansion and water sorption. As for water sorption it has been shown that materials exhibiting high water sorption values are more easily stained by hydrophilic colourings in aqueous solutions, the water presumably acting as a penetrating vehicle. The hydrophilic composites allow water to penetrate the matrix or filler matrix interface.<sup>29,30, 31,32</sup> In instances where both adsorption and absorption are known to exist and it is not clear which process predominates, the whole process is called as sorption. The staining may result from adsorption of stain on the surface of the composite<sup>4</sup> following the absorption of water by the resin matrix.<sup>33</sup>

The least discoloration was found upon polishing with Soflex disks followed in ascending order by En-

hance, mylar and diamond burs system (Table 3). There was a greater amount of discoloration in specimens polished with diamond instruments and cured under the mylar strip as compared to specimens polished with Soflex and Enhance. The difference in the discoloration  $(\Delta E)$  in NBS units between the mylar and Soflex and mylar and Enhance were 1.36 and 1.06 respectively, which is discernible by the naked eye. It has been shown that the resin matrix plays a major part in the colour stability of composites and water sorption rate is of particular importance.<sup>34,35,36</sup> The Bis-GMA matrix is a very viscous and bulky difunctional monomer. The high viscosity of Bis-GMA is reduced by the admixture of trietylene glycol dimethacrylate (TEGDMA), which is more reactive. The lower the viscosity of the monomer mixture, the more filler may be incorporated into the mixture. An increased filler content will decrease water sorption rate.<sup>37</sup> As the surface area to volume ratio of the colloidal particles in Silux Plus is quite high (52% wt), it is difficult to attain higher level of loading in composite resins containing larger fillers. This reduced size of pyrolytic silica and the consequently filler matrix contact may also account for the increased water sorption and staining phenomena. 38,39

Silux Plus, a microfilled composite resin used as a control among the composites, performed as expected. It contains Bis-GMA as a matrix and TEGDMA as a diluent. It uses pyrogenic silica as fillers distributed at a concentration of 52% by weight and increased water sorption.

Clearfil AP-X is a midifilled hybrid type of composite resin. It contains Bis-GMA as a matrix and TEGDMA as a diluent. It uses barium glass as fillers distributed at a concentration of 85% by weight. The mean  $\Delta E$  value was found to be lowest when the surface was polished with Soflex disks followed in ascending order by the mylar strip, Enhance and diamond polishing system (Table 4). The difference in colour of the specimens in the Soflex and mylar subgroups against the diamond subgroup was visually discernible. The  $\Delta E$  of the specimens of the diamond subgroup (5.27) was more severe than those in the Enhance subgroup (3.06). The discoloration in the Enhance subgroup was not visually discernible to those specimens in the Soflex and mylar subgroups. The difference in the degree of discoloration is visually discernible. Seven out of 10 specimens polished with Enhance had  $\Delta E$  values below 3.3. The rest of the specimens in the Enhance subgroup had DE values ranging from 3.43 to 3.71. A possible explanation for this is the use of the polishing paste in the Enhance polishing system. A film may have formed on the surface of the composite resin that hindered the adsorption and absorption of the staining solution. This decreased amount of discoloration, however, was true only for the Clearfil AP-X Enhance subgroup. The chemistry of the composite resin may have played a role in the apparent reduced stainability of these specimens. The more severe discoloration in the diamond subgroup could be perceived visually.

### Definite

A definitive classification of ormocer has yet to be established. Ormocer is a composite material and its chemistry of this material appears to be that of a modified composite resin. It uses barium glass as fillers distributed at a concentration of 77% by weight. The material is said to have improved polymerisation shrinkage and water sorption properties. The matrix, which consists of ceramic polysiloxane, may have led to these improvements.

Specimens treated with Soflex had the least mean discoloration value followed by mylar, Enhance and diamond system (Table 5). There was, however, no significant difference (p < 0.05) between the Soflex, mylar, Enhance treated specimens. There was also no significant difference (p < 0.05) in the discoloration values between the mylar, Enhance and the diamond system.

These results are in agreement with the results of studies<sup>26</sup> that mylar treated surface discolours more than the polished surface. Enhance disks and diamond burs produced the roughest surface on Clearfil AP-X, Filtek Z-250 and Definite. This is in agreement with the findings of Stoddard and Johnson<sup>40</sup> that regardless of composite resin filler type, Enhance disks and diamond burs produced the roughest surface. This was not true for Silux Plus, which had more discoloration in the mylar subgroup than for Enhance.

### Filtek Z-250.

Filtek Z-250 specimens with the lowest mean discoloration value was in the mylar subgroup followed

in ascending order by those in the Soflex subgroup, Enhance subgroup and diamond system subgroup. There was no statistically significant difference (p < 0.05) between mylar, Soflex and Enhance subgroups. There was also no significant difference (p < 0.05) in the mean discoloration values between the Enhance and diamond subgroup (Table 6).

### Comparison between the groups

Clearfil AP-X discoloured the least followed by Silux Plus, Definite and Filtek Z-250 except for mylar treated group where Filtek Z-250 discoloured less than Silux Plus and Definite (Fig 1). There was no significant difference (p < 0.05) found between the  $\Delta E$ value of Clearfil AP-X, Silux Plus and Definite. There was also no significant difference (p < 0.05) in the  $\Delta E$  value between Silux Plus, Definite and Filtek Z-250. These results were expected based on the water sorption values given by the manufacturers. Our results are in agreement with other studies which suggest that hydrophilic composites allow water to penetrate the matrix or filler matrix interface.<sup>29,30,31,32</sup>

Overall, Clearfil AP-X showed the least discoloration among the composite materials tested and among the polishing materials tested Soflex produced the least discoloration.

### RECOMMENDATIONS

- 1 The Komet diamond burs system should not be regarded as a final step in the polishing procedure as it produces a rough surface and prone to discoloration.
- 2 Results of the study also suggest that Silux Plus need to be polished when cured under the mylar strip to reduce discoloration.

### CONCLUSION

The  $\Delta E$  (discoloration) values of the test materials cured against the mylar strip is consistently greater than those polished with Soflex disks. The difference, however, is not visually perceptible.

### DISCLAIMER

Authors state that they have not gained any material advantage from this study.

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