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Effect of the Degree of Conversion of Composite Resins on Their Color Stability, Hardness, Flexural Strength and Flexural Modulus at Different Storage Time in Artificial Saliva

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Introduction

Good esthetics and the ability of establishing a bond to enamel and dentin makes composites a common restorative material.[1] Composites employing adhesive technologies allow for minimal invasive interventions and sometimes do not require tooth preparation.[2]

Resin based composites are basically composed of an organic matrix i.e. dimethacrylate monomers – such as bisphenol-A-glycidyldimethacrylate (Bis-GMA), urethane dimethacrylate (UDMA), bisphenol-A-ethoxydimethacrylate (Bis-EMA) or triethyleneglycoldimethacrylate (TEGDMA).[3] and inorganic fillers. Inorganic fillers in resin composites have been progressed from macrofills to microfills and from hybrids to microhybrids. Materials such as packable and nanofilled composites also have been introduced to the dentistry.[4]

Microfilled composites with smaller particle fillers allowed better polish retention which results in enhanced esthetics, but provide less strength. Hybrid composites combines larger particle fillers to improve strength but were less polishable. Particles size of 1-4 µm responsible for optimal wear resistance and adequate mechanical properties of composites.[5]

The newer generation of nanohybrid composites creates durable restorations which maintain a gloss shine which reduces plaque accumulation and provide maximum esthetics. Nanofilled resin materials consist of nanomeric particles blended with nanoclusters in a conventional resin matrix, having filler particle size ranges from 0.1 to 100 nanometers[6].

Higher amount of filler content results in reduced polymerization shrinkage and improved mechanical behavior, like diametral tensile strength, flexural strength, flexural modulus, compressive strength and microhardness, which is very critical in areas with high functional stress in oral cavity.[7-9]

Polymerization stage is the most critical aspects of a composite resin restoration. Complete polymerization may result in a resin having low porosity, more hardness, high polishing and low staining capacity and no detrimental effects on pulp caused by the free monomers[10]. The polymerization process during setting reaction of composite materials is never complete. The average value of the degree of conversion of the composites is 43-75%.[11] The number of double carbon links (C=C) present in the monomers, which are converted into single links (C-C) to form the polymeric chain during the polymerization process, is called *degree of conversion* (DC).[12] Physical properties of composites are dependent on the degree of conversion of the resin matrix.[13] A previously published study showed a correlation between the degree of conversion, color stability, hardness, modulus of elasticity and flexural strength of restorative resin material.[1, 14]

Mechanical properties of composites are influenced by their chemical composition and the environment to which they are exposed.[15] It ultimately leads to decrease the mechanical properties of the material and affects the longevity of resin composite restorations. Color stability of light cured composite resin remains a concern after long term intraoral exposure.[16]

Hence this study was conducted to evaluate and compare the effect of degree of conversion of microhybrid, nanohybrid and nanofilled composite resins on their color stability, hardness, flexural strength and flexural modulus at different storage time in artificial saliva.

Aim and Objectives

To evaluate and compare the effect of the degree of conversion of three different types of composite resins (microhybrid, nanohybrid and nanofilled) on their color stability, hardness, flexural strength and flexural modulus stored in artificial saliva for 1 day and 30 days duration.

Material

Three different composite resins were chosen in accordance with their types of filler particles. They were divided into three groups.

Group A: Microhybrid composite resin (Denfil™, VERICOM)

Group B: Nanohybrid composite resin (HerculitePrecis, Kerr)

Group C: Nanofilled composite resin (Filtek™ Z350 XT, 3M ESPE)

The shade selected for each material was A2, and the manufacturer's instructions concerning the use of each material were carefully followed.

Sample Preparation

All the samples were prepared by same operator using same technique to avoid manipulative error.

A total of 90 rectangular samples, 30 from each type of composite resin measuring 23mm length, 5mm width and 2mm thickness were prepared in the customized mold. The mold was placed over the Mylar strip supported with a glass slide. After placing the materials into the mold, another Mylar strip was placed over the material and pressed against other glass slide. Light pressure was applied to expel the excess material and trapped air. Then glass slide was removed.

Degree of Conversion of Non-Polymerized Sample

The degrees of conversion of all the non-polymerized samples of each group (n=90) were measured using Fourier transform infrared (FTIR) Spectrometer. For each sample, a spectrum was collected in the range of 3000 cm^{-1} to 500 cm^{-1}, starting with 24 scans that had a resolution of 4 cm^{-1} in absorbance mode. A spectrum of the non-polymerized composite was used as a reference for calculation of the conversion degree.

Afterwards all the samples of each group (n=90) were polymerized using LED light curing unit with standard polymerization cycle on top and bottom of the sample to ensure complete polymerization. Exposure time intervals of 20s were used for each site, throughout the study. The light was first applied at the center of the surface, being both ends cured one at time (60s per surface) After light polymerization, the flash was removed.

Further each group was randomly subdivided into two; I and II having 15 samples in each subgroup. The samples of first subgroup were stored for 1day and of second subgroup for 30 days duration at 37^0C and 100% humidity in light-proof dark colored containers containing an artificial saliva solution to avoid further exposure to light irradiation.

Evaluation of Parameters after 1 Day

All the samples of first subgroups i.e. subgroups A I, B I and C I were tested to measure the degree of conversion, color, hardness, flexural strength and flexural modulus.

1. Evaluation of the DC

Spectra for all the samples were collected as before using FTIR Spectrometer.

Considering the peak intensity of absorbency of the C=C aliphatic connections (1637 cm^{-1}) and, considering the internal pattern of the aromatic connections C-C (1607 cm^{-1}), the degree of conversion was established according to the following formula:

$$\text{Residual double Connections (\%)} = \left(\frac{a/b\ \text{(Polymerized)}}{a/b\text{(Non-Polymerized)}} \right) \times 100$$

Degree of conversion (%) = 100–Residual double connections (%)

Where, a= absorbance in 1637 cm^{-1}

b= absorbance in 1607 cm^{-1}

2. Evaluation of the color

Color values were recorded using digital spectrophotometer (Ocean Optics HR4000). Color measurements were performed by positioning the sample on a white background to prevent potential absorption effects on any of color parameter.

The color was evaluated according to the color system CIE La*b*, in which L indicates color luminosity (ranging from 0 to 100, that means black to white); a* determines the amount of red (positive values) and green (negative values); b* determines the amount of yellow (positive values) and blue (negative values).

3. valuation of hardness

The Vickers hardness (VK) of the surface was determined with a microhardness tester (Mitutoyo, HM – 100) using a Vickers diamond indenter and a 200 g load applied for 15 sec. Five VK readings were recorded for each sample. For a given sample, the five hardness values for each sample were averaged and reported as a single value.

4. Evaluation of flexural strength

Samples were placed on a 25 mm-length supporting base and assembled in a universal testing machine (UNITEST 10, Acme Engineers, India). A customized device was adapted to the upper holder to allow vertical loading of the samples according to a three-point bending test design. Axial load was applied until failure at a crosshead speed of 0.5 mm/min. Flexural strength data were obtained in N and transformed in MPa using the formula:

$s=3FL/2bh2$

Where s is the flexural strength (MPa), F is the recorded force (N), L is the length between the supporting points (20 mm), b is the width of the sample (5 mm), and h is the thickness of the sample (2 mm). The load-deflection curves were recorded with computer software (UNITEST).

5. Evaluation of Flexural modulus

Based on flexural strength data, flexural modulus was calculated using computer software.

Evaluation of Parameters After 30 Days

After 30 days of storage period in artificial saliva, all the samples of second subgroups i.e. subgroups A II, B II and C II were tested again to measure the degree of conversion, color, hardness, flexural strength and flexural modulus as before.

Change of color

Color change between the samples of subgroups I and II was obtained through Hunter equation

$\Delta E_{ab}{}^{*}= [(\Delta L^{*})^2 +(\Delta a^{*})^2 +(\Delta b^{*})^2]^{1/2}$

While luminosity values (ΔL^{*}), were reached using $\Delta L^{*}=L_{30}-L_1$, where L_{30} represents L value at immersion time of 30 days and L_1 represents L value at immersion time of 1 day.

The adopted classification of ΔE values was determined by the National Bureau of Standards (NBS) that considers: 0.0 to 0.5 values: extremely slight change; 0.5 to 1.5: slight change; 1.5 to 3.0: perceivable change; 3.0 to 6.0: marked change; 6.0 to 12.0: extremely marked change; 12.0 or more: change to another color.[17]

The data for the degree of conversion, color, hardness, flexural strength and flexural modulus of the composites tested are shown in master chart. The obtained data was statistically analyzed by using ANOVA and Student's *t*-test using Statistical Package IBM SPSS-20.

Results

Degree of Conversion

The degree of conversion of tested composites was found to range from 47.78-60%. Mean DC values for group A I, group B I and group C I were 47.95%, 51.04% and 53.45% respectively. Mean DC values for group A II, group B II and group C II were significantly increased (P= 0.000) to 55.77%, 56.99% and 59.68% respectively (Table 1,2 and 3). DC was found highest for group C followed by group B and group A at both the storage period.

Color

Regarding color change of all tested composites, ΔE was recorded 2.416, 2.286 and 2.029 for group A, group B and group C respectively. According to National Bureau of Standards (NBS) ΔE= 1.5 to 3.0 is considered as perceivable change.[62]As color was measured using CIE La*b* system, change of L values for group A (P= 0.338), group B (P=0.357) and group C (P= 0.390) composite resin was found to be statically insignificant at 1 day and 30 days storage duration. Similarly values of a and b parameter changed insignificantly for tested resin composites (Table 1,2 and 3).

Hardness

Mean hardness values of three composite resins was recoreded as group A I (53.38), group B I (65.01) and group C I (82.15). After storage of 30 days in artificial saliva, mean hardness value was highest for group C II (79.83) followed by group B II (62.53) and group A II (51.35). Hardness values were decreased for all tested composite resins after 30 days. There was stastistically significant decrease in hardness values of group A (P=0.012), group B (P= 0.013) and group C (P= 0.026) shown in Table 1,2 and 3.

Flexural Strength and Flexural Modulus

Mean Flexural strength and flexural modulus was highest for group C I (103.06 MPa; 6.70) followed by group B I (96.31 MPa; 6.27) and group A I (74.04 MPa; 4.79). After 30 days, mean flexural strength and flexural modulus values were 66.14 MPa; 5.8 for group A II, 82.98 MPa; 6.98 for group B II and 90.58 MPa ; 7.2 for group C II. There was significant decrease in flexural strength value of all three types of composite resins (P=0.000). Flexural modulus of three composites was found to be increased significantly after storage of 30 days (P=0.000) as shown in Table 1,2 and 3.

Table 1: Comparison Of Parameters Between Group A I And Group A II

Parameter	t value	P value
DC_1 and DC_{30}	159.88	0.000
L_1 and L_{30}	0.975	0.338
a_1 and a_{30}	0.958	0.347
b_1 and b_{30}	0.991	0.330
Hardness	2.362	0.012
Flexural strength	17.55	0.000

Flexural modulus	108.4	0.000

($P < 0.05$ = significant)

Table 2:Comparison Of Parameters Between Group B I And Group B II

Parameter	**t value**	**P value**
DC_1 and DC_{30}	94.23	0.000
L_1 and L_{30}	0.936	0.357
a_1 and a_{30}	0.957	0.347
b_1 and b_{30}	0.432	0.669
Hardness	2.637	0.013
Flexural strength	18.04	0.000
Flexural modulus	53.16	0.000

Table 3:Comparison Of Parameters Between Group C I And Group C II

Parameter	**t value**	**P value**
DC_1 and DC_{30}	105.25	0.000
L_1 and L_{30}	0.873	0.390
a_1 and a_{30}	0.826	0.416
b_1 and b_{30}	0.926	0.362
H hardness	2.35	0.026
Flexural strength	17.62	0.000
Flexural modulus	54.18	0.000

Discussion

Degree of conversion is defined as the percentage of reacted aliphatic C=C bonds from the dimethacrylate monomers present in their polymeric matrices.[12] Physical and mechanical properties of resin composites can be affected by their degree of cure.[18] There are various studies carried out which showed correlation between DC and physical properties of composite resins,[3,16,19,20] hence this study was conducted to evaluate the effect of DC on color, hardness, flexural strength and flexural modulus.

The filler particle size in resin composites directly affects their radiopacity, properties, wear resistance and elastic modulus.[21]The decreasing filler particle size allows an increased interfacial area between the matrix and the fillers leading to better dispersion.[8,22] This in turn will increase flexural strength and surface microhardness. Three different types of composites were chosen in this study. Group A (microhybrid; Denfil), group B (nanohybrid; HerculitePrecis) and group C (nanofilled; Filtek Z350 XT).

Restorations are constantly interacting with saliva in the mouth, water based artificial saliva with neutral pH was chosen as storage media in this study since it simulate the natural saliva that usually exist in the mouth.[23]

Ferracane et al studied the effects of normal- cured and heat-cured composites after aging in water for 1 to 180 days. The aging had little effect after 30 days; the storage period of 30 days was selected for this study. Depending upon storage time, three experimental groups i.e. group A, group B and group C were randomly divided into two subgroups I and II. First subgroup of each group was stored for 1 day and other subgroup for 30 days.

Degree of Conversion

Degree of conversion of tested composite resins was found to range from 47.78-60%. The calculated degree of conversion was in accordance with the results reported by ***Silva et a l***[24] and ***Taher NM*** .[3] Calculated DC in our study for group A I, B I and C I were 47.7-48.1%, 50-51% and 53.2-58.6% respectively. Degree of conversion significantly changed to 55.52-56% of group A II, 56.76-57.24% of group B II and 59.58-60% of group C II when stored for 30 days in artificial saliva.

Degree of conversion was highest for group C followed by group B and group A among the tested materials at both storage periods in artificial saliva.[3] ***da Silva EM et al***[24] showed that the DC of nanofilled composite was significantly lower than that of the hybrid composite. In the present study Filtek Z 350 XT (nanofilled composite resin) has been used, which contains new opacifiers to improve the opacity of the resin composites. Opacity is a factor that can significantly affect light transmission which could be the explanation of the obtained results.[24]

Matrix composition plays important role in the DC, as the methacrylate functional group is capable of copolymerization with monomer and may increase the DC.[3] It had been suggested that amines having methacrylate functionality may be favorable in increasing the DC in commercial systems, with an attendant increase in physical properties. With high molecular weight monomers such as Bis-GMA there is notable amount of un-reacted c=c remaining within the composites when it is polymerized with visible light at mouth temperature. In addition to the unreacted monomer, additional un-reacted c=c structures may be present from diluents such as TEGDMA monomer or similar substances. This could be the reason behind the higher DC readings of the tested nanofilled and nanohybrid resins.[19] All tested composites contain similar resin matrix compositions. But, the addition of UDMA to the resin matrix of nanofilled and nanohybrid may have resulted in a higher DC than microhybrid resin composites.[25]

Degree of conversion of all three resin composites increased significantly after 30 days of storage in artificial saliva (p<0.05). This is because polymerization reaction of light-activated composites continues to progress over a time even after the end of light irradiation and the DC showed a slow increase after light exposure. ***Sabah Ismail et al*** and ***LFJ Schneider et al*** showed that after one month of water storage, DC was significantly increased with resin composite.[11,26]

Color

Interpretation of color is a psychological issue and is affected by the skill of observer and may be reported differently on different occasions. To overcome such type of errors, color evaluating devices were utilized and the data were recorded in the CIE L*a*b* system. The CIE system uses three-dimensional colorimetric measurements: L* values correspond to the brightness of the color, a* values to the red–green content, and b* values to the yellow–blue content. The color changes (ΔE) are calculated from the ΔL, Δa, and Δ b values for each type resin composite. $\Delta L = L_{30} – L_1$, where L_{30} represents L value at immersion time of 30 days and L_1 represents L value at immersion time of 1 day. Similarly Δa, and Δ b values were calculated.[25,27]

ΔE value of group A, group B and group C was 2.416, 2.286 and 2.029. These results are in accordance with a study conducted by ***Sabatini et al***[28] and ***Malekipour MR et al***.[25] According to National Bureau of Standards (NBS); 1.5 to 3.0 is considered as perceivable change.[17] As composite resin samples were stored in artificial saliva for 30 days, all groups produced color change under the threshold of 3.0. The observed color change could be due to water sorption over the period of 1 month. This color change is clinically acceptable. It indicates that water itself may not plays a major role in the color change of composite resins. It acts as a carrier for staining agents.[28]

ΔE value was highest for group C followed by group B and group A. As color stability is related to degree of cure, group C composite resin with high DC were more color stable than other two. All tested light activated composite contains BIS-GMA and TEGDMA as the resin matrix and filler 66%-78% wt. silica / zirconia. Different studies have shown that the presence of TEG DMA in materials cause a high amount of hydrophilic capacity and it makes Bis-GMA prone to tonality and water absorption in comparison to UDMA. UDMA is highly resistant to stain than Bis-GMA and at the same time leads to lower.[25,29,30]

Moreover, it has been noted that a composite resin with large filler particles are more prone to water aging discoloration than a composite with small filler particles. Thus, there was more color change observed with microhybrid composite resin. These results were in accordance with the study conducted by ***Malekipour MR***

et al.[25] The filler particle size and distribution seem to be directly related to optical properties and that nanofiller particles provide low visual opacity in non-pigmentedental composites.[30] A smaller filler size might contribute to decrease staining and enhance esthetic appearance.[31] Analysis of the individual parameter ΔL, Δa, and Δ b was performed. There was no significant difference in L_{30} and L_1; a_{30} and a_1 and b_{30} and b_1 for all three composite resins.

Hardness, Flexural Strength and Flexural Modulus

It is generally accepted that an increase in the filler concentration of resin composites is related to an increase in certain properties such as elastic modulus, flexural strength, hardness and compressive strength.[22] In this study mean Vickers hardness number (VHN) value was found to be highest for group C I (82.15) followed by group B I (65.01) and group A I (53.08) composite resin. The different filler content of material accounts for these results.[32] Filler loading of nanofilled composite is about 78.5 wt% whereas that of nanohybrid and microhybrid is 77 wt% and 66 wt% respectively. Although filler content of nanofilled and nanohybrid composites was nearly equal but filler particle size differs in both.[33]

Calculated mean flexural strength and modulus was highest for group C I (103.06 MPa; 6.70) followed by group B I (96.31 MPa; 6.27) and group A I (74.04 MPa; 4.79). One of the reasons for these types of results is the filler content of material. ***Braem M et al*** showed that as the filler load increased, flexural strength of composite also increased.[22] Another important possibility could be higher DC of nanofilled resin material.

After storage period of 30 days, hardness and flexural strength in all the groups decreased significantly. The reduction in these properties is predominantly related to uptake of water by polymer. Artificial saliva contains major portion of distilled water in its composition. Water swells the resin polymer matrix and occupies space between the main chain and cross links, as well as fills microvoid created during the process of polymerization. At the same time water causes hydrolysis of the filler-matrix interface which would contribute to reduction in properties.[2,15,24]

All tested composites contains silica or silicate glass fillers, they have irregularly distributed Si-O-Si bonds. After immersion in artificial saliva, when resin matrix swells, radial tensile stresses are introduced at filler interface, which makes filler prone to stress corrosion attack. It results in partial or complete debonding of filler matrix interface. ***AUJ Yap et al*** and ***Ferracane JL et al*** showed that matrix shrinkage during polymerization develops hoop stresses around the filler particle.[34, 35] These stresses increases the frictional forces among filler and resin matrix, that decreases the pull out tendency of the filler. After aging in water, the swelled resin matrix reduces hoop stresses and facilitates filler pull out.[36] These mentioned mechanisms might contribute to decreased hardness and flexural strength after storage in artificial saliva for 30 days.

Regarding flexural modulus, in all the three subgroups i.e. group A II, B II and C II values were increased significantly after 30 days of storage period. Continuous degree of conversion of composites could be the reason.[37] Apparently, the water uptake by composite resin might not have the same deleterious effect on flexural modulus as on microhardness and flexural strength. These results were in accordance with ***AUJ Yap et al***.[35] The effect of aging on flexural properties was material dependent.[38]After aging, flexural properties obtained is dependent on the balance between composite post-cure and the degradation by water.[35] For all composites, an increase in stiffness was observed with aging. The increase in stiffness that can be attributed to post-cure was significant for all composites.

As the effect of DC on color, hardness, flexural strength and flexural modulus of composites was evaluated in-vitro condition at two time period interval i.e. at 1 day and 30 days, further studies are required to evaluate effect of DC on physical properties of composites in the in-vivo condition.

Conclusion

The present *in-vitro* study focuses on the effect of DC and storage time on color, hardness, flexural strength and flexural modulus in artificial saliva.

According to the results obtained in the present in-vitro study, following conclusions can be drawn:

1. Composite resins with high DC exhibit superior physical properties at the storage period of 1 day in artificial saliva.
2. Nanofilled composite resin possesses superior physical properties as compared to nanohybrid and microhybrid composites.
3. All tested composites showed significantly reduced values of hardness and flexural strength when stored in artificial saliva for the period of 30 days.
4. There was no adverse effect of storage media and time on flexural modulus of tested material.

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