A COMPARATIVE STUDY BETWEEN DEGREE OF CONVERSION AND FLEXURAL STRENGTH OF COMPOSITE RESINS

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Statement of problem. Although many studies have been carried out to investigate the correlation between the degree of conversion and the flexural strength of composite resins, there is minimal information in the literature attempting to compare degree of conversion, flexural strength and their correlation between restorative composite resins and flowable composite resins.

Purpose. The purposes of this study were to measure the degree of conversion and flexural strength of composite resins with different rheological behavior and to correlate the two properties.

Materials and methods. Four restorative (Vit-l-escence, Z-250, Tetric ceram, Esthet-X) and four flowable (Aeliteflo, Admiraflow, Permaflo, Revolution) light-curing composite resins were investigated. The degree of conversion(DC) was analyzed with Fourier transfer infra-red spectroscopy(FTIR) spectrum by a potassium bromide(KBr) pellet transmission method. The spectrum of the unpolymerized specimen had been measured before the specimen was irradiated for 60s with a visible light curing unit. The polymerized specimen was scanned for its FTIR spectrum. The flexural strength(FS) was measured with 3-point bending test according to ISO 4049 after storage in water at $37°C$ for 24 hours. The data were statistically analyzed by an independent sample t-test and one-way ANOVA at the significance level of 0.05. The dependence of flexural strength on the degree of conversion was also analyzed by regression analysis.

Results. Mean DC and FS values ranged from 43% to 61% and from 84.7MPa to 156.7MPa respectively. DC values of the flowable composite resins were significantly higher than those of restorative composite resins ($P < 0.05$). The FS values of restorative composite resins were greater than those of flowable composite resins. No statistically significant correlation was observed between the DC and the FS tested in any of the composites. The dependence of FS on DC in restorative or flowable composite resins was not significant.

Conclusion. It can be concluded that radical polymerization of the organic matrix is not a major factor in determining flexural strength of the commercially available composite resins.

Key Words

Degree of conversion, Flexural strength, Restorative composite resin, Flowable composite resin

The application of Bis-GMA monomer and dental composites to restorative dentistry was so successful that it was soon adopted and dental composite has been successfully used as an esthetic filling material. Dental composite resins essentially comprise of resin matrix (organic phase), inorganic filler particles (dispersed phase), filler-matrix coupling agent (interphase) and minor additions including polymerization initiators, stabilizers and coloring pigments.[1,2] A number of factors influence the properties and the clinical performance of the polymerized composite resins. Such factors are molecular weight and viscosity of the resin matrix, the size and distribution of filler particles, the intensity and duration of curing light and the temperature of the resin at the moment of curing.[3]

Research on composites is being directed towards the development of much stronger systems for better mechanical and physical characteristics.[4] Flexural strength (FS) is one of the important properties of composite resins when used in areas where heavy masticatory force occurs, especially in posterior regions. Thus, high flexural strength is desirable where stress is significant.[1] The other important characteristic of composite resins is the degree of conversion (DC). This is of great importance for the ultimate success of the restoration and clinical longevity of the restoration.[5] The results of microstructural analysis suggest that crack propagation in the experimental composite resins is mainly in the resin matrix. It is suggested that a possible approach to improvement of the strength of dental composite resins is to increase the strength of the resin matrix itself.[6] As physical and mechanical properties associated with dental composites are influenced by the level of conversion, it is desirable for all of its monomer to convert to polymer during the polymerization reac-

tion. However, with high molecular weight monomers, such as Bis-GMA or UDMA, there is always an incomplete and significant concentration of unreacted carbon-carbon double bonds remaining in the final product.[7]

There are two types of composite resins used in dentistry, restorative composite resins and flowable composite resins. The difference is dependent on their viscosity. The viscosity of composite resins is a critical factor for the success of adhesion and manipulation. Although many studies have been carried out to investigate the correlation between the degree of conversion and the flexural strength of composite resins, there is little information in the literature attempting to compare the degree of conversion, flexural strength and their correlation between restorative composite resins and flowable composite resins.

The purposes of this *in vitro* study were to investigate the degree of conversion and the flexural strength of several commercially available composite resins with different rheological behavior and to correlate between the two properties. The null hypothesis to be tested in this study was that there was no significant correlation between the degree of conversion and the flexural strength of the composite resins with different rheological behavior.

MATERIAL AND METHODS

Eight commercially available light-cured composite resins were investigated. The brand names and manufacturers are listed in Table I. Vit-l-escence, Z-250, Tetric ceram and Esthet-X were restorative composite resins, and Aelieflo, Admiraflow, Permaflo and Revolution were flowable composite resins. The intensity of curing light was verified during the test using a radiometer (Curing Radiometer Model 100, Demetron Research Corp., Danbury, CT, USA).

Table I. Resin systems investigated in this study

Product	Lot No.	Manufacturer	Characteristics
Vit-l-escence	1-800-552-5512	Ultradent, South Jordan, Utah, USA	Bis-GMA
Z-250	4LAJ	3M-ESPE, St. Paul, MN, USA	Bis-GMA,UDMA,Bis-EMA
Tetric ceram	F50402	Ivoclar-Vivadent AG, Schaan / Liechtenstein	Bis-GMA,UDMA,TEGDMA
Esthet-X	309052	Dentsply Caulk, Milford, DE, USA	Urethane modified Bis-GMA, TEGDMA
Aeliteflo	400004867	Bisco, Schaumburg, IL, USA	Ethoxylated Bisphenol-A DMA, TEGDMA
Admiraflow	360729	Voco, Cuxhaven, Germany	Ormocer (anorganic-organic co-polymers),
Permaflo	1-800-552-5512	Ultradent, South Jordan, Utah, USA	Methacrylate-based
Revolution	402380	Kerr, Orange, CA, USA	Bis-GMA

A. Degree of conversion

1. Preparation of the specimen

The resin paste was diluted with high purity KBr powder and mixed thoroughly in a pestle and mortar.(Fig. 1) The mixture was pressed with sufficient pressure (10 ton) to produce a transparent pellet,(Fig. 2) which dimension was 0.75 mm in thickness and 13mm in diameter. The pellet was placed in the transmission holder for analysis. First, the spectrum of the unpolymerized resin was measured, and then the resin specimen was irradiated for 60s with a visible light-curing unit (Optilux 360, Demetron Research Corp., Danbury, CT, USA). The polymerized specimen was scanned for its FTIR spectrum at 5 min after the start of irradiation.[8,9]

2. Fourier transfer infra-red spectroscopy (FTIR)

The DC in the bulk of the resin was analyzed by FTIR. The background spectra were collected through pure KBr pellet. For the measurement of DC, FTIR spectra of uncured and cured samples of composite resins were analyzed on a FTIR spectrometer (Fig. 3, MB100, ABB Bomem, Canada) operating with 16 scans at a resolution

of 4 cm^{-1} within a wave length of 400-2000 cm^{-1} and then the range from 1560-1670 cm^{-1} was expanded. From the FTIR spectra obtained, the ratio of peak intensities of aliphatic C=C to aromatic C=C (1640 and 1610 cm^{-1}, respectively) were evaluated before and after irradiation to determine the percentage of unsaturated aliphatic C=C bonds remaining in the test material. To determine the percentage of reacted double bonds, the spectra, recorded initially as the transmission mode, were converted to the absorbance mode. The intensities of the aliphatic C=C absorbance peak at 1640 cm^{-1} and the aromatic C=C reference peak at 1610 cm^{-1} were measured. The DC was calculated by using the baseline method.

3. Calculation of the degree of conversion.

The DC of each specimen was equal to 100 minus (% C=C) (Equation 1).

$$100 - (\% C=C) \cdots\cdots\cdots (Equation\ 1)$$

where,

$$(\% C=C) = \frac{(aliphatic\ C=C\,/\,aromatic\ C=C)\ polymcer}{(aliphatic\ C=C\,/\,aromatic\ C=C)\ monomer} \times 100$$

B. Flexural strength

1. Preparation of the specimen

For flexural strength testing, a stainless steel mold (Fig. 4) was used for preparation of the specimen (25 mm × 2 mm × 2 mm). Light-cured composite resin was condensed into the mold and the middle third of the specimen was first activated for 40s with 400 mW/cm², and then the remaining thirds were activated at the same intensity and curing time as the middle third. The illuminated areas overlapped. The light intensity of the lamp was tested regularly with a radiation meter and was at the same level during all tests. Following a possible post-cure, the specimens were placed in water at 37°C for 24 hours. After storage, the specimens were ground on a wet sand paper (grit 600) and measured using a digital vernier caliper. Measurements were taken from two locations for length, width and height. The average of the two values was taken to calculate the flexural strength.[10,11]

2. Calculation of flexural strength

The flexural strength of the polymerized resins was measured with the three point bending test according to the ISO 4049. Flexural strength testing was carried out with an Instron Universal testing machine (Model 4201, Norwood, MA, USA) at a crosshead speed of 0.5 mm/min until the specimen was fractured. The maximum load exerted on the specimen was recorded, and flexural strength (MPa) was calculated using the following equation (Equation 2);

$$FS = \frac{3FL}{2bh^2} \quad \text{(Equation 2)}$$

where F is the maximum applied load, L is the supporting span length (20 mm), b is the width of the test specimen, h is the height of the test specimen. Seven specimens were tested for each material.

C. Statistical analysis

A mean value and standard deviation were computed in each experimental group. Independent sample t-test, one-way ANOVA and multiple-comparison Scheffé test were performed at the significance of 0.05.(SPSS ver. 11.0, SPSS Inc., Chicago, IL, USA) Dependence of the flexural strength on the degree of conversion
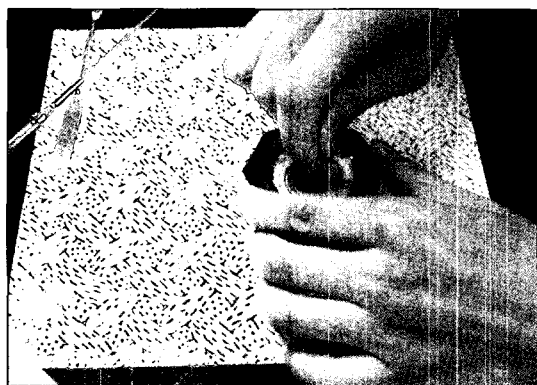

Fig. 1. Mix thoroughly in a pestle and mortar.
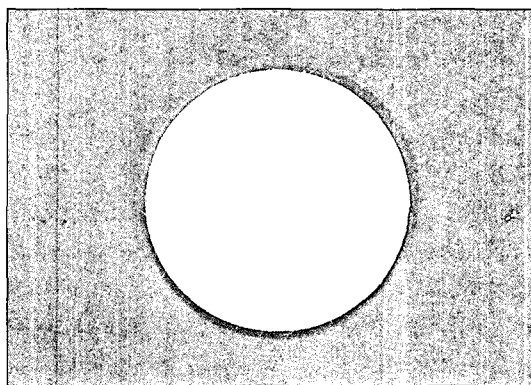

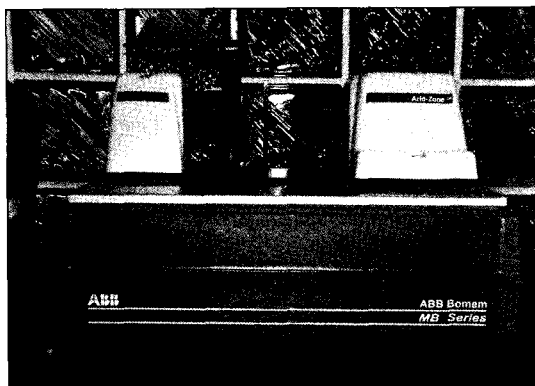**Fig. 2.** Transparent pellet.

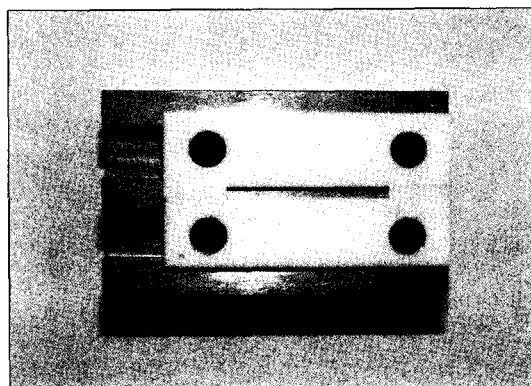Fig. 3. FTIR spectrometer.

Fig. 4. Stainless steel mold.

between materials was also analyzed by regression analysis.

RESULTS

A. Degree of conversion

Fig. 5 shows the spectra obtained for the pre- and post- irradiation of the Aeliteflo. The aromatic C=C stretching mode at 1610 cm^{-1} (internal standard) and the aliphatic C=C stretching mode at 1640 cm^{-1} are clearly detected. The ratio of aliphatic C=C after polymerization was decreased obviously as compared with the ratio of aromatic C=C. Since aromatic C=C, which is used as an internal standard, does not change during the polymerization, infrared absorption ratio of residual unpolymerized aliphatic C=C to aromatic C=C was compared by baseline method.

Table II and Fig. 6 present that the degree of conversion values ranged from 43 % to 61 %. Z-250 showed the lowest DC (43%) and Aeliteflo showed the highest DC (61%). Table II also shows that statistically significant differences between two experimental groups (P $<$ 0.05) were found. The DC values of the flowable composite resins are significantly greater than those of restorative com-
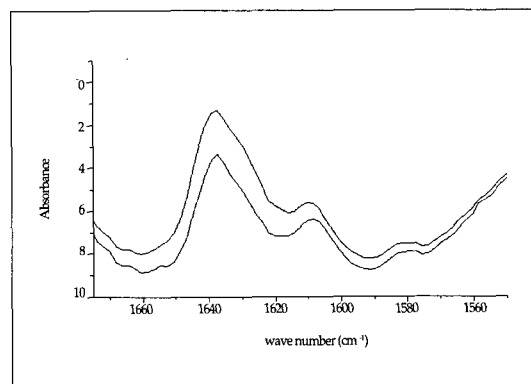

Fig. 5. FTIR spectrum of Aeliteflo. Before (red) and after (blue) polymerization.

posite resins. Inter-material comparisons of DC presented that the value of Z-250 was lower than that of Vit-l-escence, Aeliteflo and Permaflo and the value of Esthet-X was lower than that of Aeliteflo and Permaflo.(P $<$ 0.05) With the restorative composite, the value of Z-250 was lower than that of Vit-l-escence and Tetric ceram and the value of Vit-l-escence was higher than that of Esthet-X and Z-250. With the flowable composite resin, the value of Aeliteflo was significantly higher than that of Admiraflow.

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B. Flexural strength

Table III and Fig. 7 present that the mean flexural strength ranged from 84.7MPa to 156.7MPa. Z-250 showed the highest FS (156.7MPa) among the test materials but significantly lower DC than all the other brands. On the other hand, Revolution showed the lowest FS (84.7MPa). The comparison of FS between restorative and flowable composite resins is also presented in Table III. As expected, statistically significant differences ($P < 0.05$) were found between two experimental groups. Flowable composite resins showed lower FS than restorative composite resins. In the flexural strength test, through ANOVA multiple comparisons, the restorative composite resin presented the flexural strength from highest to lowest as follows; Z-250 > Vit-l-escence > Tetric ceram = Esthet-X. The flexural strength of the flowable composite resin presented from highest to lowest as follows; Permaflo > Aeliteflo = Admiraflow > Revolution. Significant difference was observed between the brands.
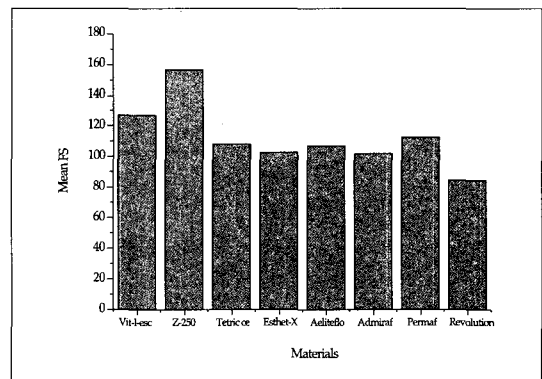
Table II. Mean, SD and Independent Samples t-Test of the DC value

Materials		Mean (SD)		Sig. (2-tailed)
Restorative	Vit-l-escence [b,c]	56.9 (6.3)		
	Z-250 [a]	43.0 (3.8)	49.41	
	Tetric ceram [a,b,c]	52.8 (3.7)	(0.07)	
	Esthet-X [a,b]	46.2 (3.4)		0.04
Flowable	Aeliteflo [c]	61.2 (3.9)	54.20	
	Admiraflow [a,b]	47.9 (5.5)	(0.08)	
	Permaflo [b,c]	57.9 (7.8)		
	Revolution [a,b,c]	50.6 (3.3)		

(Letters indicate statistically similar groups.)
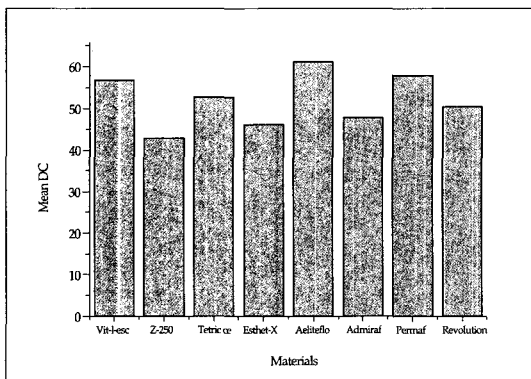
Fig. 6. Mean of the degree of conversion value.


Fig. 7. Mean of the flexural strength.

Table III. Mean, SD and Independent Samples t-Test of the flexural strength

| Materials		Mean (SD)		Sig. (2-tailed)
Restorative	Vit-l-escence [d]	127.2 (4.9)		
	Z-250 [e]	156.7 (5.0)	123.2	
	Tetric ceram [b,c]	108.5 (2.8)	(22.3)	
	Esthet-X [b,c]	102.3 (9.6)		.000
Flowable	Aeliteflo [b,c]	107.0 (2.4)		
	Admiraflow [b]	101.8 (4.4)	102.1	
	Permaflo [c]	112.7 (3.9)	(12.0)	
	Revolution [a]	84.7 (2.6)		

(Letters indicate statistically similar groups.)

Table IV. Regression analysis. Dependent Variable: FS (P < 0.05)

Model	Sig.		
	Total	Restorative	Flowable
(Constant)	067	268	601
DC	549	634	383
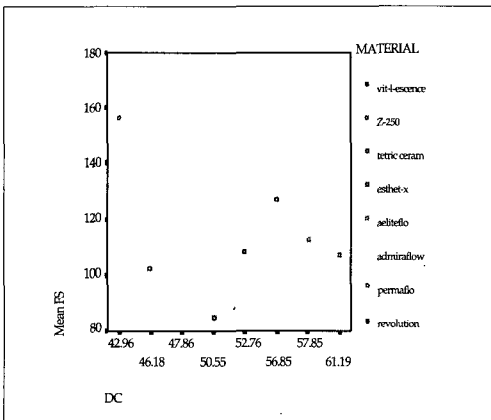
Fig. 8. The correlation between the DC and the FS of the investigated composite resins.
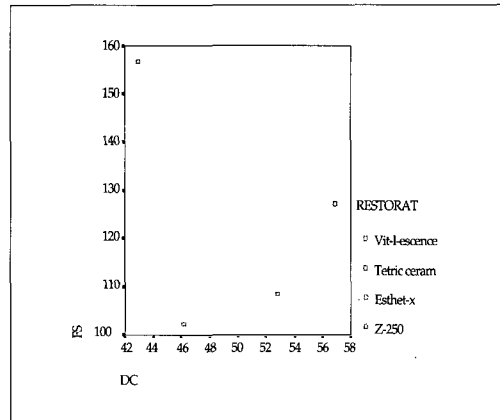
Fig. 9. The correlation between the DC and the FS of the restorative composite resins.

C. Regression analysis

Regression analyses were performed on the relationship between the DC and the FS. The plots in Fig. 8 to 10 show the correlation between the DC and the FS of the commercially available composite resins. In restorative and flowable composite resins respectively, direct linear relationship was not present between the DC and the FS (Table IV). In this study, no statistically significant correlation was observed between the DC and the FS tested in any of the resins.
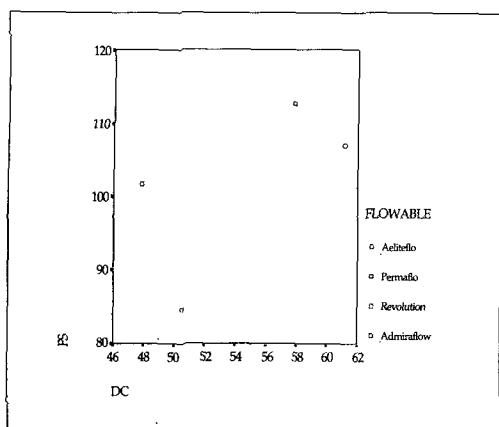
Fig. 10. The correlation between the DC and the FS of the flowable composite resins.

DISCUSSION

Among several methods[8,12-19] for the determination of DC of dental resins, FTIR was used in this study as a reliable method.[8,20] It has been widely used as it detects the C=C stretching vibrations directly before and after curing of composite resins. FTIR spectroscopy is based on the absorption of radiation in the infrared frequency range due to molecular vibrations of the functional groups contained in the polymeric chain.[13,16]

Ferracane and Greener studied the unfilled Bis-GMA-based resins and found that the degree of conversion for the experimental resins varied between 55 % and 72 %.[8] Chung and Greener reported that the degree of conversion of seven light-cured posterior composites ranged from 43.5 % to 73.8 %.[4] The DC of composite resins investigated in this study ranged from 43.0 to 61.2 %. This is very much in agreement with the results of previous studies.[4,8,14,21] The DC of the low viscosity materials, flowable resins, which ranged from 47.9 to 61.2 %, exhibited significantly higher than that of the high viscosity materials, restorative composite resins, which ranged from 43.0 to 56.8 %. It is suggested that the higher value of DC of flow-

able composite resins was related to the increased molecular movement due to increased rheological effect when the filler volume is reduced. And another possible explanation might be due to the difference of composition of the monomer in the resin matrix system. Flowable resins were added with more amount of diluent or thinner with low molecular size than restorative composite resins in order to reduce their viscosity. The monomers with lower molecular weight were freer to move than those with higher molecular weight. As a result, more three-dimensional network was formed in the flowable resins during polymerization.

Contrary to the results of DC, in this study the flexural strength of flowable composites was significantly lower than that of restorative composites. Flexural strength can be attributed to the filler content of the composite resins. Previous studies have reported a positive correlation between the mechanical properties and volumetric fraction of fillers.[22-24] Highly filled composite resin is expected to be stronger than that with lower filler loading. Dental clinicians should keep in mind this variety when they decide on cavity design and materials to use. Low FS would be impractical for clinical placement in load bearing posterior cavities.

Z-250, which has a high filler volume, exhibited highest flexural strength, even though its DC was the lowest. A blend of UDMA and Bis-EMA, which is diluent of Z-250, has large molecular weight, so their molecular movement may have been decreased and they have fewer double bonds per unit of weight than TEGDMA. High resin viscosity restricted the mobility of reactive radicals and reduced the frequency and probability of random encounters, which led to a decrease in polymerization propagation. Due to these reasons, the DC of Z-250 is low.[9]

DC of composite resin system significantly

affects its material properties, especially mechanical strength.[6,19,25] It is generally accepted that this strength is increased with the increase of DC. In the lower DC case, unreacted monomers remaining in the material may break the integrity of polymer structure. This jeopardizes the clinical serviceability of composite through accelerated wear and discoloration of the materials.[26] Thus, the higher the conversion of double bonds, the greater the mechanical strength.[25,27] However, a direct linear relationship was not present between the DC and FS of the tested materials. This no correlation was similar to the Ferracane's study,[24] which tried to make a correlation between hardness and degree of conversion. The reason why there was no correlation between the DC and FS of the composite resins investigated in this study was that each composite has different organic matrix compositions and different filler loadings. Direct comparison could not be used to predict the correlation of the DC and the FS when comparing different brands. Varying the relative amounts of UDMA, Bis-GMA and TEGDMA in the matrix could have a varying effect on the mechanical properties of the composites.[18,28] The lack of correlation between DC and FS can be explained by multi-factorial determinants of mechanical property, such as formulation of diluents, filler composition, morphology and concentration. Generally, these various factors do not individually provide the composite with all the desirable characteristics. They should always be improved together.

FS is related more to the amount of inorganic filler in highly-filled composite, so as to compare the DC and the FS, experimental composite resins have to contain the same amount of filler. But all the tested materials have different composition according to their manufacturers. The limitations of this study may be the small sample size and the usage of composites with different monomer composition and filler loading.

Polymerization shrinkage is a critical shortcoming of dental resins and may contribute to microleakage. An increase in the filler content of composite resin lessens the polymerization shrinkage. As the composite resin becomes more flowable, the restoration will shrink more. High DC brings about increased polymerization shrinkage, so to compensate it clinically, the clinician has to add composite with small amount using layering technique in the prepared cavity. The choice of composite resin depends upon various clinical situations. To improve the properties of the composite materials, further investigations are needed to search for the filler composition which best suits all clinical situations. Modifying the inorganic phase and the monomer composition of the composite resins to achieve higher degree of conversion should also be focused on in further research.

CONCLUSION

Within the limitations of this study, the following conclusions were drawn;
1. The degree of conversion of the restorative composite resins was significantly lower than that of the flowable composite resins.($P < 0.05$)
2. The flexural strength of the restorative composite resins was significantly higher than that of the flowable composite resins.($P < 0.05$) Flexural strength of the composite resins in the same class was significantly different between brands.
3. No significant correlation was found between the degree of conversion and the flexural strength of the materials investigated.

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