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필러의 유형이 유동성 레진의 미세경도와 굴곡성질에 미치는 영향

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Effect of filler type on microhardness and flexural properties of flowable resins

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ABSTRACT

본 연구는 필러의 유형이 유동성레진의 경도와 굴곡성질에 미치는 영향을 평가한 것이다. 이를 위하여 필러 유형이 각기 다른 세 부류의 유동성레진 (nanohybrid, microhybrid, and minifil)을 사용하였다. 경도측정을 위하여 4×2×3 mm의 시료를 만들고 24시간 경과 후에 시료의 윗면과 바닥면에서 경도를 각각 측정하였다. 굴곡성질을 평가하기 위하여 ISO 4049 규정에 따라 25×2×2 mm의 시료를 만들고 굴곡강도와 modulus를 만능시험기를 이용하여 측정하였다. 측정값은 ANOVA test로 분석하였다. 그 결과 nanohybrid 제품들의 미세경도는 다른 제품들에 비하여 높은 (20.2-34.2 Hv vs 8.7-30.5 Hv) 값을 보였다. 굴곡강도는 필러 유형에 유의한 연관성이 없었지만 modulus의 경우 nanohybrid 제품이 다른 제품들에 비하여 높은 값의 보였다.

KEY WORDS: 유동성 레진; 필러 유형; 미세경도; 굴곡성질

INTRODUCTION

Methacrylate-based resin composites are widely in use as a dental material in dentistry. The advantages of resin composites as a restorative material are their excellent mechanical, physical, and aesthetic properties, easy handling and caries prevention features (Wiegand et al., 2007; Ferracane, 2011). Also, with minimum expense, the damaged part in tooth can be swiftly and timely restored. Resin composites are a commixture of different monomers and inorganic fillers. Among the monomers, Bis-GMA is of high viscosity due to high molecular weight, and it works as a backbone monomer in most resin composites. To reduce viscosity for easy handling, diluents of low molecular weight (MW) such as TEGDMA (triethyleneglycol dimethacrylate) and UDMA (urethane dimethacrylate) are mixed (Geurtsen & Leyhausen, 2001; Floyd & Dickens, 2006). Since resin products have different combinations and ratios of monomers and inorganic fillers depending on the purpose of usage, each resin products have slightly different viscosity (Ilie & Hickel, 2009; Papadogiannis et al., 2011).

Inorganic fillers are important in that they improve strength, hardness, and wear resistance of resin composites. Polymerization shrinkage that causes many unwanted problems, such as restoration fractures, marginal leakage, and recurrence of caries after restoration, can be reduced by increasing filler content (Kemp-Scholte & Davidson, 1998; Lai & Johnson, 1993). In many resin composites, microhybrid-type composites

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have achieved excellent roles. However, recently, with an aid of nanotechnology to further reduce polymerization shrinkage and improve esthetics of resin composites, containing of nanofillers in resin products becomes general (Chen, 2010; Sideridou et al., 2011).

For lining or basement procedure, flowable resin became the first choice (Ivanovas et al., 2011; Beun et al., 2012). The remarkable advantage of flowable resins is their easy-to-handle feature compared to conventional resin composites. High flowability was achieved by reducing the filler content and increasing diluents in the composition of flowable resins. Flowable nature due to the low viscosity is good in placing the resins into the cavity with excellent wetting and marginal adaptation (Abedian & Millstein, 2006; Lee et al., 2010). Reduction of restoration procedure and treatment time can be expected. However, the decrease of filler content had significantly reduced hardness and also much increased polymerization shrinkage compared to many other resin composites (Kleverlaan & Feilzer, 2005; Pick & Pelka et al., 2011). To improve these shortcomings, many recent flowable resins contain nanofillers in their resin matrix to increase filler weight and volume. Under these circumstances, evaluation of composite resins which have different filler types may provide valuable results such as mechanical properties.

The purpose of the present study was to test how the fillers of different types affect the mechanical properties of flowable resins.

MATERIALS AND METHODS

Flowable resins

For the study, nine flowable resins in three different filler types (all with A3 shade) were selected as outlined in Table1: nanohybrid [Premise flow (PF), Synergy D6 flow (SF), Tetric N flow (TF), Filtek Z350 flow (ZF)], microhybrid [AeliteFlo (AF), Esthet X flow (EF), Palfique Estelite LV (PL), Revolution Formula 2 (R2)], and minifil [Heliomolar flow (HF)]. For light curing, a quartz-tungsten-halogen (QTH) light-curing unit (LCU) [Optilux 501, Kerr, Orange, CA, USA] was used.

Table 1. Materials tested in the present study

Type	Code	Composition	Filler content vol%/wt%	Manufacturer
Nanohybrid	PF	Bis-EMA, TEGDMA, Barium glass, silica fillers	54.6/72.5	Kerr Orange, CA, USA
	SF	Bis-GMA, TEGDMA, Barium glass, amorphous silica	42/63	Coltene/Whaledent Cuyaho Falls, OH, USA
	TF	Bis-EMA, UDMA, TEGDMA, Barium glass, YbF_3, SiO_2	39/63	Ivoclar Vivadent Schann, Liechtenstein
	ZF	Bis-GMA, TEGDMA, Zirconia/silica cluster fillers	55/65	3M ESPE St. Paul, MN, USA
Microhybrid	AF	Bis-EMA, TEGDMA, Barium glass, glass fillers	42/60	Bisco Inc. Schaumburg, IL, USA
	EF	Bis-GMA adduct, TEGDMA, Ba-F-B-Al silicate glass, silica	53/61	Dentsply Caulk Milford, DE, USA
	PL	Bis-EMA, Bis-GMA, TEGDMA, Silica-zirconia, silica-titania filler	50/65	Tokuyama Tokyo, Japan
	R2	TEGDMA, Barium glass	41/60	Kerr Orange, CA, USA
Minifil	HF	Bis-GMA, UDMA, TEGDMA, YbF_3, SiO_2	30/51	Ivoclar Vivadent, Schann, Liechtenstein

PF: Premise Flow; SF: Synergy D6 Flow; TF: Tetric N Flow; ZF:Filtek Z350 Flow;
AF: AeliteFlo; EF: Esthet X Flow; PL: Palfique Estelite LV; R2: Revolution Formula 2
HF: Heliomolar Flow

Microhardness

To measure the surface microhardness (Hv) of the specimens, resin was filled into a metal mold (4×2×3 mm), both top and bottom surfaces were covered with glass slides, and light cured for 40 s using the LCU. The cured specimen was removed from the mold and aged for 24 h in a 37°C dry and dark chamber. The microhardness of the top (z=0) and bottom (z=3 mm) surfaces was measured using a Vickers hardness tester (MVK-H1, Akashi, Tokyo, Japan) by evaluating the size of microindentations (n=12 for each test condition). To make the micro-indentation, a 200 gf load and 10 s dwell time conditions were applied.

Flexural properties

The three-point bending test was performed to determine the flexural properties [flexural strength (FS) and modulus (FM)]. To make specimens, a metal mold (25×2×2 mm) was filled with resin according to the ISO 4049 guidelines. After filling the mold, both top and bottom surfaces were covered with glass slides to make a flat surface. The specimen was irradiated for 40 s using the LCU. Since the specimen was much wider (25 mm) than the tip size (7 mm), five light exposures were performed on each side by overlapping the curing light. After light curing, specimen was removed from the mold and aged for 24 h in a 37°C dry and dark chamber. After aging, the specimens (n=5 for each test condition) were loaded to a universal test machine (Instron 3345, Grove City, PA, USA) at a crosshead speed of 1 mm/min. FS (σ_f in MPa) was determined using the following formula:

$$\sigma_f = 3DP/(2WH^2)$$

where D is the distance between the supports (20 mm), P is the maximum failure load (N), W is the width (2 mm), and H is the height (2 mm) of the tested specimen. FM (E in GPa) was obtained using the following formula:

$$E = (P/D) \cdot (D^3/(4WH^3))$$

where P/D is the slope in the linear portion of the load-displacement curve.

Statistical analysis

The obtained results were analyzed using the ANOVA. A post-hoc Tukey's test was followed at α=0.05 level.

RESULTS

The microhardness of the specimens both on the top and bottom surfaces is shown in Table 2. Among the specimens, ZF and PL showed the highest and lowest microhardness, respectively. Also, interestingly, these two products showed slightly higher microhardness on their bottom surface compared to their top surface

Table 2. Microhardness (Hv) of the tested specimens

Type	Code	Top	Bottom	Type
Nanohybrid^{A}	PF	33.0 ± 1.3^{a}	32.6 ± 1.3^{a}	Nanohybrid^{A}
	SF	27.0 ± 0.7^{b}	19.3 ± 2.5^{b}	
	TF	20.2 ± 1.2^{c}	19.3 ± 0.7^{b}	
	ZF	34.2 ± 2.1^{a}	35.4 ± 2.2^{c}	
Microhybrid^{B}	AF	25.1 ± 0.7^{d}	22.1 ± 0.6^{d}	Microhybrid^{B}
	EF	30.5 ± 0.9^{e}	26.0 ± 1.4^{e}	
	PL	8.7 ± 0.6^{f}	9.8 ± 1.0^{f}	
	R2	24.9 ± 0.6^{d}	20.5 ± 0.9^{bd}	
Minifil^{B}	HF	18.8 ± 0.4^{c}	11.0 ± 0.7^{f}	Minifil^{C}

* Statistically significant difference on type and resin product are shown by superscript letters^{A,B,C} and letters$^{1,2,\cdots}$, respectively. Same letters or numbers are not significantly different (p〉0.05).

Table 3. Flexural properties of the tested specimens

Type	Code	Strength (FS, MPa)	Modulus (FM, GPa)	Type
Nanohybrid[A]	PF	136.5 ± 17.1[abc]	8.82 ± 0.20[a]	Nanohybrid[A]
	SF	123.9 ± 6.1[ab]	7.86 ± 0.66[ab]	
	TF	122.8 ± 5.7[ab]	7.33 ± 0.17[bc]	
	ZF	162.1 ± 12.8[de]	8.41 ± 0.17[ab]	
Microhybrid[A]	AF	141.4 ± 3.1[ad]	5.81 ± 0.26[d]	Microhybrid[B]
	EF	152.1 ± 14.9[cd]	8.40 ± 0.54[ab]	
	PL	114.4 ± 10.6[b]	5.39 ± 0.66[d]	
	R2	121.1 ±10.6[ab]	6.46 ± 0.80[cd]	
Minifil[A]	HF	126.3 ± 9.6[ab]	5.55 ± 0.18[d]	Minifil[B]

* Statistically significant difference on type and resin product are shown by superscript letters[A, B] and letters[a,b,..], respectively. Same letters or numbers are not significantly different (p〉0.05).

(34.2, 35.4 Hv; 8.7, 9.8 Hv). A significantly different microhardness was observed between the nanohybrid and microhybrid products (α 〈0.05).

The flexural strength (FS) and modulus (FM) are shown in Table 3. FS of the tested specimens ranged from 114.4 to 162.1 MPa. Among the specimens, ZF and PL showed the highest (162.1 MPa) and lowest (114.4 MPa) FS, respectively. For FS, the filler type had no statistically significant influence. However, the FS of nanohybrid products was slightly greater (122.8-162.1 MPa) than that of microhybrid products (114.4-152.1 MPa). FM ranged from 7.33 to 8.82 GPa for nanohybrid products and 5.39 to 8.40 GPa for microhydrid products. The FM of nanohybrid products was significantly different to that of microhybrid products (α 〈0.05).

DISCUSSION

Microhardness is the measure of how material resists to an external force that causes permanent deformation on the material. Unlike resin composites of high filler-loading, most flowable resins contain less filler than that of most other resin composites to increase flowability (Ilie & Hickel, 2009; Hadis et al., 2011). Both on the top and bottom surfaces, specimens of nanohybrid showed slightly higher microhardness than that of microhybrid and minifil specimens, it is due to the slightly greater filler content. The microhardness of the top and bottom surfaces was linearly correlated with the filler content (wt%) at the range of 0.84-0.93 (according to the curve fit) depending on the surface and if TF and PL are excluded. In the case of TF and PL, they showed exceptionally low microhardness even though their filler content was not low. Slightly higher microhardness on the bottom surface of ZF and PL compared to their top surface can be deduced from the condensation of the bottom surface. The condensed surface becomes more dense and stiff due to the shrinkage and subsequently may achieve increased hardness. Nevertheless, such microhardness increase in the bottom surface was not general in all products, but was a product-dependent outcome.

Flowable resins are usually used as a liner or base material beneath the resin composites. For the optimal and durable restoration, consistent mechanical properties among tooth, resin composite, and flowable resin are important. In the present study, flexural and compressive properties were tested. The flexural properties [strength (FS) and modulus (FM)] are the measure of material resistance against transverse stress. The FS of the tested specimens was approximately 114-162 MPa. The range of these values is similar to the most methacrylate-based proprietary products (63-161 MPa) (Ilie & Hickel, 2009). The correlation with the filler weight was negligibly low (R〈0.2) and the FS difference for filler type was statistically insignificant (α 〉0.05). Low correlation between FS and filler content is probably due to the possibility that FS depends more on the

internal defects which were formed during manufacturing process (Zeng & Odén, 1996, Della Bona & Anusavice, 2003). FM was approximately 5.6-8.8 GPa, lower than that of dentin (17-25 GPa) (Xu, 1998; Kinney & Marshall, 2003). In the load-displacement curve of the bending test, FM is the slope of the linear portion (elastic range) and is the measure of material's stiffness, so high FM implies a great stiffness (Ferracane, 2001). Since hardness of the material in the elastic range is the level of stiffness in that range, a high correlation between microhardness and FM for the top and bottom surfaces (R=0.83) seems natural. Unlike the case of FS, FM showed a high correlation with filler content (wt%) (R=0.61 for all products; 0.82 if PL is excluded).

CONCLUSION

Within the limitations of the present study, the following conclusions could be reached:

1. Flowable resins can be diversely classified according to the filler type, however, the differences in microhardness and flexural properties for nanohybrid, microhybrid, and minifil products were minor.
2. Except one or two products, filler weight was highly correlated with microhardness and flexural modulus.
3. Additionally, microhardness and flexural modulus were strongly correlated to each other.

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