Comparison of Bond Strength, Flexural Strength, and Hardness of Conventional Composites and Self-adhesive Composites: An *in vitro* Study

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ABSTRACT

Aims and objectives: To evaluate and compare the bond strength, flexural strength, and hardness of conventional composite resin and self-adhesive composite resin.

Materials and methods: The materials, conventional composites (Herculite Precis by Kerr—nanohybrid composite material) and self-adhesive composites (Dyad Flow by Kerr), were compared based on their bond strength, flexural strength, and hardness. The test for bond strength was carried out on extracted premolars embedded in acrylic blocks. About 20 samples (10 of each group) were prepared by building up composite on these tooth-embedded acrylic blocks. For determination of flexural strength and microhardness of the material, 20 specimens of a specific size were made using aluminum molds, and the material was cured according to manufacturer's instructions. Bond strength and flexural strength were determined with the help of a universal testing machine, and microhardness was determined with the help of Vickers hardness test.

Results: The results of the tests were obtained by two independent sample t tests. Bond strength of conventional composite resin and self-adhesive composite resin did not significantly differ in their values (p = 0.354). Similarly, the flexural strength of these two materials did not differ significantly (p = 0.213). However, the microhardness of conventional composite resin and self-adhesive composite resin differed significantly (p = 0.012).

Conclusion: Self-adhesive composite resins can replace conventional composite resins in terms of their bond strength and flexural strength. Since the values of microhardness differed significantly for these two materials, self-adhesive composite resins cannot be a substitute for conventional composite resins in terms of their microhardness.

Keywords: Bond strength, Conventional composites, Flexural strength, Hardness, Self-adhesive composites.

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INTRODUCTION

Composite is a system composed of two or more macromolecules, which are essentially insoluble in each other and differ in form.¹

The conventional etch-bond protocol of composite increases the technique sensitivity and clinical time. Self-adhesive composites have been marketed to simplify the clinical procedures and overcome their technique sensitivity.² These self-adhesive composite resins are flowable composites with excellent handling properties, low viscosity, and superior injectibility, and require less chair-side time, which is advisable in treatment of young children.

Self-adhesive composite combines the merits of adhesive and restorative material technologies in one product, bringing novel horizons to restorative techniques.

The literature with respect to self-adhesive composites does not show studies related to mechanical properties of flexural strength, bond strength, and microhardness, and their comparison with conventional composites.

Hence, the aim of this study is to determine the bonding efficacy, flexural strength, and microhardness of these resins to tooth structure; also, their comparison with the conventional composite resins. The assumed null hypothesis states that there is no difference between conventional composites and self-adhesive composites with respect to the above-mentioned properties.

MATERIALS AND METHODS

It is an *in vitro* study carried out using the following materials:

- Group I—conventional composite resins (Herculite Precis by Kerr—nanohybrid composite material)
- Group II—self-adhesive composite resins (Dyad Flow by Kerr) (Fig. 1).



Fig. 1: Armamentarium: (A) Conventional composite resin; (B) gel etchant; (C) bonding agent; (D) self-adhesive composite resin; and (E) light cure unit

MATERIALS

Material	Brand name	Composition
Conventional composite resin	Herculite Precis by Kerr – nanohybrid composite material	7,7,9 (or 7,9,9)-trimethyl-4,13- dioxo-3,14-dioxa-5,12- diazahexadecane-1,16-diyl bismethacrylate 2,2-bis(acryloyloxymethyl)butyl acrylate 3-trimethoxysilylpropyl methacrylate
Acid etchant	Gel etchant by Kerr	37.5% phosphoric acid Cobalt aluminate blue spinel
Bonding agent	Optibond S	Ethanol 2-hydroxyethyl methacrylate 2-hydroxy-1,3-propanediyl bis methacrylate alkali fluorosilicates (Na)
Self-adhesive composite resin	Dyad Flow by Kerr	glycerophosphoric acid dimethacrylate (GPDM) adhesive monomer Fillers: • Prepolymerized filler • 1-µm barium glass filler • Nano-sized colloidal silica • Nano sized vtterbium fluorido

METHODS

Sample Size Determination

The sample size for the tests was determined based on previous studies on composite resins as well as on the following formula:

$$n = \begin{bmatrix} z_{\frac{\alpha}{2}} \sigma \\ \frac{z}{E} \end{bmatrix}^2$$

where E is the margin of error;

 ${\sf Z}_{\frac{\alpha}{2}}$ is known as the critical value, the positive z value, i.e., at

the vertical boundary for the area of $\frac{\alpha}{2}$ in the right tail of the standard normal distribution;

 σ is the population standard deviation; n is the sample size.



Fig. 2: Tooth embedded in an acrylic block for determination of bond strength

Determination of Bond Strength

For determination of bond strength, 20 extracted premolars were selected with the following inclusion criteria:

- Intact crown structure of the tooth
- Nonrestored tooth
- Noncarious tooth

All the selected premolars were stored in chloramine-T solution at room temperature. Acrylic blocks were prepared by cold-cured acrylic resin material. The selected premolars were embedded into the acrylic resin up to their occlusal surfaces. The blocks were then put in water to avoid expansion of the material (Fig. 2).

The occlusal surface of the samples was then flattened as a prerequisite for the determination of shear bond strength. These surfaces were then bur-abraded with the help of straight-fissured diamond bur. The samples were then subjected to thermocycling of 5,000 thermocycles in water from 5°C to 55°C with a dwell time of 30 seconds at each temperature and a transfer time from one water bath to other of 5 seconds (Fig. 3).



Fig. 3: Flattened occlusal surface of the sample for determination of bond strength

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Figs 4A to C: Determination of bond strength: (A and B) Composite build-up done on the dentinoenamel junction; and (C) sample loaded in the universal testing machine for determination of bond strength

For the determination of bond strength of selected samples, 10 samples (n = 10) each of group I (conventional composite resin) and group II (self-adhesive composite resin) were prepared.

Group I (conventional composite resin) samples were subjected to acid etching and application of bonding agent was done according to the manufacturer's instructions. The samples for group II (self-adhesive composite resin) were just washed and air dried.

With the help of a 4-mm diameter straw, already cut into 3 mm pieces lengthwise, 10 cylinder-shaped composite build-ups were made for each group. The incremental build-up was done at the dentino-enamel junction (Figs 4A to C).

Bond strength of the samples was determined on Star Testing System's, universal testing machine (computerized, software based), model number STS 248. The area of the composite build-up was calculated for each sample. Shear load was measured for each sample, keeping the crosshead speed at 3 mm/minute.

Shear bond strength (MPa) = shear load (N)/area of composite build-up (2 πr^2)

Determination of Flexural Strength

With the help of an aluminum mold of size 40 mm × 5 mm × 3 mm, 20 samples, 10 each of group I (conventional composite resin) and group II (self-adhesive composite resin), were prepared. The mold was first coated with separating medium for the ease of composite sample retrieval from the mold. Any excess material was trimmed away with the sand paper (Figs 5A to D).

Flexural strength was determined on Star Testing System's, universal testing machine (computerized, software-based), model number. STS 248, with the crossheadspeed of 3 mm/minute and the span length being 20 mm. The area of individual samples was calculated and the flexural load was measured in Newton.

> Flexural strength (MPa) = $3 PL/2 bd^2$ where P = fracture load L = span between supports d = thickness of the sample b = width of the sample

Determination of Microhardness

For the determination of microhardness, 20 samples, 10 each of group I (conventional composite resin) and group II (self-adhesive composite resin), were prepared with the help of an aluminum mold and embedded in acrylic blocks. The blocks were polished and excess material was trimmed away with the help of sand paper (Figs 6A to C).

Vickers hardness test was performed on these samples with the help of Vickers microhardness tester. The test specimens were placed on the stage of the tester and stabilized. The test load of 50 gm was applied with dwell time of 20 seconds. The load and the penetration depth of the indenter were continuously measured during the load. Then, the area to be indented was selected by focusing with a 600× objective lens. Vickers hardness was calculated from standard chart given in the International Organization for Standardization standard.



Figs 5A to D: Determination of flexural strength: (A) Aluminum mold; (B) samples of conventional composite resins; (C) samples of self-adhesive composite resins; and (D) sample loaded on the universal testing machine for determination of flexural strength



Figs 6A to C: Determination of microhardness: (A) Samples for determination of microhardness; (B) sample loaded on the microhardness tester; and (C) vickers microhardness testing machine

RESULTS AND STATISTICAL ANALYSIS

Bond Strength

Statistical Analysis of Bond Strength

Bond strengths of groups I and II were compared by two independent sample t tests. The results showed that the difference in bond strength of conventional composite and self-adhesive composite is nonsignificant (Table 1).

Flexural Strength

Statistical Analysis of Flexural Strength

Flexural strengths of groups I and II were compared by two independent sample t tests. The results showed that the difference in flexural strength of conventional composite and self-adhesive composite is nonsignificant (Table 2). Table 1: Statistical analysis of bond strength

	Group I:	Group II:
	Conventional	Self-adhesive
	composite resin	composite resin
Minimum	5.92	3.66
Maximum	19.77	17.93
Average	12.33	10.307
Standard deviation	4.80	4.69
Median	11.42	10.2
Comparison	0.354	

No significant difference in bond strength of conventional and self-adhesive composites, p = 0.354 (p > 0.05)

Microhardness

Statistical Analysis of Microhardness

Hardness of groups I and II was compared by two independent sample t tests. The results showed that the difference in the hardness of conventional composite and self-adhesive composite is significant (Table 3).



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Table 2: Statistical analysis of flexural strength				
	Group I:	Group II:		
	Conventional	Self-adhesive		
	composite resin	composite resin		
Minimum	39.94	41.63		
Maximum	137.57	101.43		
Average	93.03	74.69		
Standard deviation	38.24	22.98		
Median	100.00	72.55		
Comparison	0.213			

No significant difference in flexural strength of conventional and self-adhesive composites, p = 0.213 (p>0.05)

Thus, bond strength and flexural strength of conventional composite resins and self-adhesive composite resins do not significantly differ from each other, while there is difference in their microhardness.

DISCUSSION

Introduction of composite resins has been a boon to the restorative and esthetic dentistry. Continuous advances in these resins in terms of strength as well as in esthetics have led these materials to become the pioneer in the field.

Further, the introduction of self-adhesive composites in dentistry in 2009 has challenged the original idea of micro-mechanical bonding of the material to the tooth structure.³ The original bonding of the composite resin to the tooth structure is micromechanical in nature. Resin tags are formed, which helps the material adhere to the tooth structure. Usage of the original etch-rinse technique, though immensely effective, is time consuming.

Further, etching leads to the collapse of dentinal tubules, and drying leads to dentinal fluid wash up leading to sensitivity.

According to previous studies, there is significant reduction in bond strength if the enamel, contaminated with saliva, is not washed off thoroughly. Saliva produces an organic film that can penetrate into the enamel microporosities created by acid etching and, thereby, interfere with the bonding of the material into the etched enamel.⁴

To overcome this problem, self-adhesive composite resins have come into the picture.

Self-adhesive composite resins are the flowable composite resins, which have modified the traditional method of bonding. The contents of self-adhesive resins are as follows:

- GPDM: Adhesive monomer
- Fillers: (1) Prepolymerized filler, (2) 1-µm barium glass filler, (3) nano-sized colloidal silica, (4) nano-sized ytterbium fluoride.

The main constituent, i.e., GPDM adhesive monomer has a functional phosphate group, which aids in chemical

Table 3: Statistical analysis of microhardness				
	Group I:	Group II:		
	Conventional	Self-adhesive		
	composite resin	composite resin		
Minimum	40.58	36.00		
Maximum	52.06	48.33		
Average	46.74	42.45		
Standard deviation	3.12	3.68		
Median	46.83	42.98		
Comparison	0.012			

Statistically significant difference in microhardness of conventional and self-adhesive composites, p = 0.012 (p < 0.05)

bonding of the material to the calcium ions in the tooth as well as it helps in etching the tooth structure.

The two methacrylate functional groups help for copolymerization with other methacrylate monomers to provide increased cross-linking density and enhanced mechanical strength for the polymerized adhesive.³

Thus, self-adhesive composite resins have a micromechanical as well as chemical bond with the tooth structure.

The study performed showed no significant difference (p = 0.354) in the bond strength of conventional (12.33 MPa) and self-adhesive composite resins (10.33 MPa).

This implies that the self-adhesive composite, without the use of any special bonding agent, has comparable bond strength to that of conventional composite resin.

A similar study performed using Vertise flow, Kerr: Bonding effectiveness of self-adhesive composites to dentin and enamel by Poitevin et al⁵ on pre-etched tooth surfaces; this showed that prior phosphoric-acid etching of dentin/enamel significantly ameliorated the bonding effectiveness of Vertise Flow (Kerr).

"Bonding performance of a self-adhering flowable composite to substrates used in direct technique" by Garcia et al⁶ showed that Dyad Flow showed lower bond strength to median dentin, however, higher bond strength to cut enamel and that the Dyad Flow can provide acceptable bond strength.

The study: "Bond strength of self-adhesive resin cements to tooth structure", concluded with the understanding that the performance of self-adhesive composite resins is far from being comparable to that of multistep conventional resin cements. These cements must be used with caution, in light of their limited bond performance.⁷

A similar study, "Comparative evaluation of shear bond strength and nano-leakage of conventional and self-adhering flowable composites to primary teeth dentin" was performed on primary teeth to compare the bond strengths of conventional flowable composites and self-adhesive composites. It was found out that the bond strength of conventional flowable composites was greater

than the self-adhesive composites. It was also found out that, in general, the bond strength of composites is lower in primary teeth than permanent teeth due to have relatively less intertubular dentin present in primary teeth after cavity preparation.⁸

Flexural strength or transverse strength is also known as the bending strength or modulus of rupture of the material.⁹ It is measured to determine bend ability of the material against forces/stresses of mastication in the oral cavity.

Flexural strength is a collective measurement of tensile, compressive, and shear stresses. It is calculated by the formula

Flexural strength (MPa) = 3 PL/2 bd² where P = fracture load L = Span between supports d = Thickness of the sample b = Width of the sample

In this study, it was found out that the flexural strength of conventional composite resins and self-adhesive composite resins do not differ significantly (p = 0.213) and thus, self-adhesive composite resins have the ability to replace conventional composite resins in the long run.

These results are in accordance to a study performed to evaluate the flexural strength of self-adhesive composite resins, conventional composite resins, and several types of glass ionomer cements (GICs). The results showed that flexural strength of self-adhesive composites and conventional composite is comparable and far greater than that of GICs.¹⁰

Traditional composite resins generally have mechanical properties that are superior to the newer so-called "universal" self-etching/self-adhesive resins. However, recent reports indicate that some properties of these new composite resins have been improved, approaching those of the traditional etch and rinse resins.¹¹

Hardness of the material is the resistance of the material to plastic deformation typically when measured under an indentation load.⁹

The relative hardness of a substance is based on its ability to resist scratching. In this study, microhardness was evaluated using Vickers hardness test.

A high Vickers hardness value combined with a relatively low surface roughness value would be an ideal characteristic of posterior composites.¹²

Microhardness of flowable composites, in general, is found to be less than that of hybrid composite resins. The hardness values for flowable composites were significantly lower than those for human enamel (408 kg/mm²) or dentin (60 kg/mm²) denoting that the materials were not appropriate for use in relatively high stress areas.¹²

This study also showed that the values of microhardness of conventional and self-adhesive composite resins differed significantly (p = 0.012) from each other, thus implying that self-adhesive composite resins cannot replace conventional composite resins in terms of their microhardness.

Lesser filler content in the material (self-adhesive composite resin) under study may be a reason contributing to the reduced Vickers hardness number.

This part of the study is left for further research, so that the microhardness of the material can be improved.

The condition of the tooth in the oral cavity differs from person-to-person. Though every possible effort was taken to simulate the oral cavity, this being an *in vitro* study; further *in vivo* studies have to be carried out, with a larger sample size to assess the materials more effectively.

CONCLUSION

Self-adhesive composites are the newer composite resins, which can replace the conventional composite resins in terms of their bond strength as well as their flexural strength.

These resins can be used in routine practice for pediatric patients as well as for uncooperative adult patients including patients requiring special care, since these resins have the ability to reduce the chair side time of the operator.

The microhardness of self-adhesive composite resins is significantly lower than that of the conventional composite resins. This leaves this part of the study for further research so that the hardness of self-adhesive resins can be improved and brought full fledgedly into routine practice in clinical dentistry.

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