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Comparative evaluation of micro-leakage and shear bond strength between a self adhesive flowable composite resin and self etch based flowable composite restorative material: An in-vitro study

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***Abstract*---**Background: An in vitro evaluation and comparing the microleakage and shear bond strength between a self- adhesive flowable composite resin (Dyad Flow) and self-etch based flowable composite restorative material (Nano Filtek Z 350XT) Materials and methods: A total of sixty specimens were prepared from extracted teeth, which were divided into two groups. For microleakage analysis, Group I consisting of thirty teeth which were further divided into two subgroups of fifteen teeth each that is Group IA and Group IB, on which standard class V cavity was prepared and restored with Dyad Flow and Nano Filtek Z 350XT respectively. The prepared specimens were immersed in 0.6% aqueous Rhodamine B dye for 24 hours. The sectioned samples were examined under a Confocal Microscope at 10 X magnification and microleakage scores were analyzed statistically using Mann-Whitney U test. For shear bond strength evaluation, Group II consisting of thirty teeth, which were subdivided into two subgroups of fifteen teeth each that is Group IIA and Group IIB. The specimens were abraded to expose flat dentinal surface and then materials was packed into cylindrical polyethylene tube which were held on dentin surface of specimens and restored with Dyad Flow and Nano Filtek Z 350XT respectively and subjected to shear bond strength by using Universal testing machine. Statistical analysis was

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done using Unpaired t test. All the specimens were stored in distilled water at 370C for 24 hours and then subjected to 500 thermal cycles at 50C and 550C with a dwell time of 10 seconds and a transfer time of 30seconds between each bath. Results: The Shear bond strength between self-etch based flowable composite that is Nano Filtek Z 350XT (25.4 ± 13.5) and self-adhesive flowable composite that is Dyad Flow (10.4 ± 4.7) was found to be statistically significant (P<0.01). Whereas microleakage between Dyad Flow (1.60 ±0.7) and Nano Filtek Z 350XT (1.9 ± 1.2) was found to be statistically insignificant (P=0.801). Conclusion: there is an advantage of Nano Filtek Z 350XT a self-etch based flowable composite resin on the property of Shear bond strength while restoring with Flowable Composite.

***Keywords*---**confocal microscopy, microleakage, self-adhesive flowable composite, shear bond strength.

Introduction

Bond strength is one of the significant factors that play a major role for the long term clinical success of restoration. When selecting an adhesive system, micro-mechanical retention is important mechanism for bonding resin to dentin. Newer materials such as self-etch systems have been introduced for better bonding to tooth surface and to provide adequate strength for the restorative materials (1) these do not require separate acid etch step and they contain no rinse acidic monomers that simultaneously condition and prime dentin and enamel (2). which reduces technique sensitivity and the risk of making application errors during composite restorations 3. The first generation of flowable composites was introduced in 1996, with their major indication of use in class V restorations. They contain a lower filler content which leads to enhanced flow and reduced elastic modulus. This allows the material to adapt closely to the microstructural and macro-structural floors and walls of the cavity (4,5).

Self-adhering and self-etching flowable composite resin [Dyad Flow] is the newer development in adhesive dentistry (6, 7). It is a direct composite resin restorative material in which no separate bonding protocol is required (8). Dyad flow is based on the bonding technology that uses GPDM (glycerol phosphate di methacrylate) adhesive monomer, which acts like a coupling agent, ensure a tenacious bond to both enamel and dentin and protects against microleakage. On one hand, it has an acidic phosphate group for etching the tooth structure and also for chemically bonding to the calcium ions within the tooth structure. On the other hand, it has two methacrylate functional groups for co-polymerization with other methacrylate monomers to provide increased cross linking density and enhanced mechanical strength for the polymerized adhesive.(9) Its bond strength is comparable to that of other self-etch adhesives for both dentin and enamel. However very few studies have been conducted to test self-adhering flowable composite resin. Two studies showed that self adhering flowable composite resin has the highest hygroscopic dimensional expansion and water sorption after 150 days of water immersion when compared to conventional flowable and composite resins. (10)

Further work is required to evaluate the performance of this resin. Hence the present study compared and evaluated the micro-leakage between Dyad Flow™ (Kerr, Orange, CA, USA) and self-etch based flowable composite Filtek Z350 XT (3M ESPE, St Paul, MN, USA) in class V cavities. And also compared and evaluated shear bond strength between self-adhesive flowable composite resin and self-etch based flowable composite restorative material to dentin.

Materials and Methods

Sample size

Study was carried out after ethical approval from institutional ethical committee number5 HDCH/2016-17/ 1681---it was a collaborative effort from in association with Central Manufacturing Technology Institute Bangalore. The sample size for the present study was obtained as 60; n = 15 per group. A total of 60 freshly extracted teeth (molars and premolars) for orthodontic and periodontal therapy, based on the inclusion and exclusion criteria, (Teeth with dental caries, restoration, developmental defects, non-carious lesions, and micro-cracks were excluded), were selected, and were stored in saline solution at room temperature for 30days. Specimens were prepared for a specified dimension and were divided into two groups containing 30 teeth each (n=30). In order to compare and evaluate two flowable composite for micro-leakage and shear bond strength analysis, each group were subdivided into four subgroups containing 15 teeth each. Group I– Micro-leakage analysis: thirty premolars –subdivided into two groups fifteen teeth each. Microleakage between group IA and IB was assessed. Group II- Shear Bond strength assessment: Thirty molars - subdivided into two groups of fifteen teeth each as group IIA and group IIB. And shear bond was assessed.

Group I– Micro-leakage analysis

Specimen preparation:
A class V cavity with standard dimensions (3mm mesiodistal width, 3mm occlusogingival height, and 1.5mm axial depth) was prepared at the cemento-enamel junction on facial surface of each tooth. The specimens were rinsed and air dried with three-way syringe. In Group IA, self- adhesive flowable composite resin Dyad flow (Kerr, U.S.A] was injected incrementally into the cavity. Each increment was cured according to manufacturer's instructions. In Group IB, a single coat of Single Bond Universal (3M ESPE) was applied on the walls of the cavity and light cured as per the manufacturer's instructions. NanoFiltek Z350 XT (3M ESPE) was injected incrementally into the cavity and light cured according to manufacturer's instructions. Specimens were then stored in distilled water at 37^0C for 24 hours and then subjected to 500 thermal cycles at 5^0C and 55^0C with a dwell time of 10 seconds and a transfer time of 30seconds between each bath. No further finishing and polishing were done as flowable resin itself imparts a well glazed surface characteristics to the restoration. Thereafter, the external surface of all the prepared samples were covered with two layers of nail varnish except for 1 mm around the restoration margins.

The prepared samples were then immersed in Rhodamine B dye solution for period of 24 hours. After 24 hours, the samples were washed under running water, dried and then sectioned facio-lingually using slow speed diamond saw (Struers, Denmark). Micro-leakage of the sectioned samples was measured using confocal microscopy at 10×magnification (Confocal Imaging Microscope, Leica TCS-SP5, DM6000-CFS) in the fluorescent mode. With a digital scale, the width of interface between restoration and tooth surface was calculated. The microleakage score (TABLE 1) was recorded using the scoring method, as per Alavi and Kianimanesh (11). (table 1) and percentage of micro-leakage evaluated with in two sub groups. (table 2)

Group II- Shear Bond strength assessment

Thirty molars - subdivided into two groups of fifteen teeth each as group IIA and group IIB.

Specimen preparation:
The specimens were abraded to expose flat dentinal surface using diamond disc and then mounted in acrylic blocks. A cylindrical polyethylene tube with an internal diameter of 4mm and 5mm height were held on dentin surface of the specimens.

Group II A: Subgroup containing 15 specimens, which were packed with self - adhesive flowable composite resin Dyad flow (Kerr, U.S.A) was incrementally injected into the polyethylene tube and light cured according to manufacturer's instructions.
Group II B: Subgroup containing 15 specimens, a single coat of Single Bond Universal (3M ESPE) was applied on the flat dentin surface and light cured. NanoFiltek Z350 XT flowable (3M ESPE) composite resin was injected in increments into the polyethylene tube positioned on the adhesive applied region. Each increment was light cured according to manufacturer's instructions. The specimens were then stored in distilled water at 37^0C for 24 hours, Polyethylene tubes were removed with a BP blade. The specimens of both the groups were then subjected to thermocycling between 5^0C and 55^0C for 500 cycles using a dwell time of 10 seconds and a transfer time of 30 seconds between each bath.
Following thermocycling, specimens were secured in the holder of a universal testing machine and sheared with a knife-edge blade at a cross head speed of 1.0mm/minute. Shear bond strength was calculated in Mega pascals by dividing the peak load at failure with the specimen surface area. (table 5)

Results

In the present study statistical analysis was done using SPSS version 20 software and Microsoft excel version 2010. The results were subjected to Descriptive statistics, Un-paired t test and Mann-Whitney U test respectively. Man-Whitney U test was carried out to evaluate and compare the Mean microleakage (table 3) and Percentage microleakage. (table 4) According to the results of present study the Microleakage between Dyad Flow (1.60 ±0.7) and Nano Filtek Z 350XT (1.9 ± 1.2) was found to be statistically insignificant. (P=0.801) (graph no.1 and 2) and there was no statistical difference between the Percentage microleakage between

Dyad Flow (32±14.8) and Nano Filtek Z 350XT(37 ± 24.9). P=0.801 Assessment of Shear bond strength was carried out using Un-paired t test. According to results of present study the Shear bond strength between Dyad Flow (10.4 ± 4.7) and Nano Filtek Z 350XT (25.4 ± 13.5) was found to be statistically significant. (P<0.01) (Table 6; graph 3)

Table 1: microleakage scores

Table no.2: Microleakage scores master chart

MASTER CHART - MICROLEAKAGE				
	MICROLEAKAGE		PERCENTAGE MICROLEAKAGE	
SL NO	DYAD	FILTEK	DYAD	FILTEK
1	1	5	20	100
2	1	3	20	60
3	2	1	40	20
4	1	2	20	40
5	2	4	40	80
6	1	1	20	20
7	2	1	40	20
8	1	2	20	40
9	3	1	60	20
10	1	1	20	20
11	3	1	60	20
12	2	2	40	40
13	1	1	20	20
14	1	1	20	20
15	2	2	40	40
MEAN	1.6	1.866667	32	37.33333
STDEV	0.736788	1.245946	14.73577	24.91892

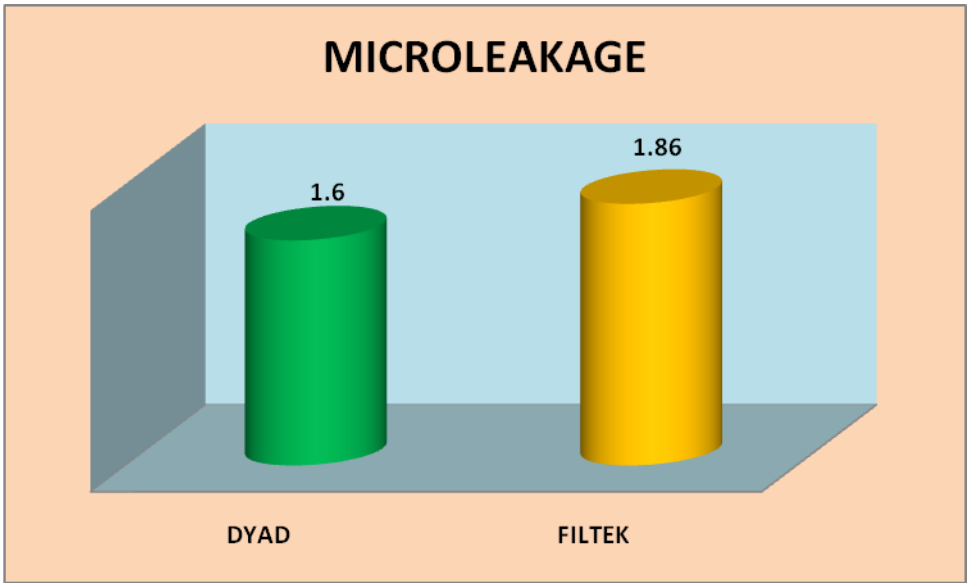
Table no.3: Comparative evaluation of Microleakage between Dyad Flow and Nano Filtek Z 350XT

Mann-Whitney U Test					
	GROUP	**n**	**Mean**	**Std. Deviation**	**P**
MICROLEAKAG	DYAD	15	1.60	0.73	0.801

E	FILTEK	15	1.86	1.24	

According to the results of present study the Microleakage between Dyad Flow (1.60 ±0.7) and Nano Filtek Z 350XT (1.9 ± 1.2) was found to be statistically insignificant. (P=0.801)

Graph 1: Bar graph showing Microleakage among tested groups



Comparative evaluation of Percentage microleakage between Dyad Flow and Nano Filtek Z 350XT

Table 4

MANN-WHITNEY U TEST					
	GROUP	**n**	**Mean**	**Std. Deviation**	**P**
PERCENTAGE MICRO LEAKAGE	DYAD	15	32.00	14.73	0.801
	FILTEK	15	37.33	24.91	

According to the results of present study, there was no statistical difference between the Percentage microleakage between Dyad Flow (32±14.8) and Nano Filtek Z 350XT(37 ± 24.9). P=0.801

Graph 2: Bar graph showing Percentage microleakage among tested groups

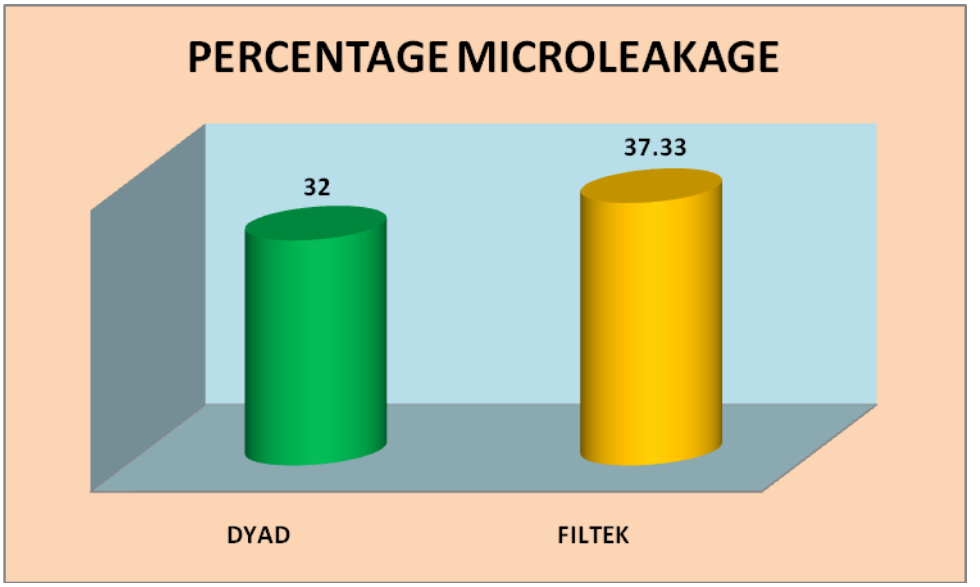


Table no.5: Shear bond strength master chart

MASTER CHART - SBS		
	SHEAR BOND STRENGTH	
SL NO	**DYAD**	**FILTEK**
1	17.01	38.11
2	6.11	35.71
3	6.2	20.04
4	5.34	35.14
5	23.2	33.94
6	10.48	5.33
7	5.33	7.03
8	11.11	26.61
9	10.61	33.03
10	6.86	26.91
11	9.61	25.39
12	10.25	10.31
13	11.47	14.05
14	12.62	53.91
15	9.82	15.37
MEAN	**10.40133**	**25.392**
STDEV	**4.743621**	**13.46037**

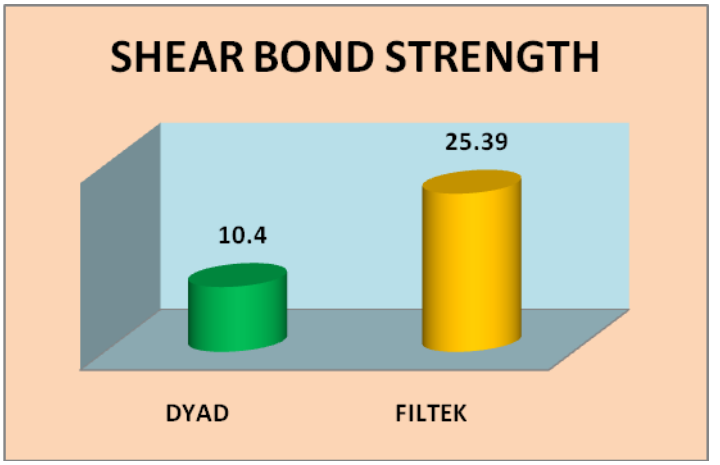
Shear bond strength evaluation

Table no.6: Comparative evaluation of Shear bond strength between Dyad Flow and Nano Filtek Z 350XT

UNPAIRED T TEST					
	Group	**n**	**Mean**	**Std. Deviation**	**p**
SHEAR BOND STRENGTH	DYAD	15	10.40	4.74	<0.001
	FILTEK	15	25.39	13.45	

According to results of present study the Shear bond strength between Dyad Flow (10.4 ± 4.7) and Nano Filtek Z 350XT (25.4 ± 13.5) was found to be statistically significant. (P<0.01)

Graph 3 : Bar graph showing Shear bond strength among tested groups



Discussion

The flowable composites possess many advantages when compared to conventional composites (12,13]. The advantages include high wettability of tooth surface, ability to form layers of minimum thickness there by eliminating air inclusion, high flexibility makes them less likely to be displaced in stress concentration areas, radio-opaqueness and aesthetic colours. The disadvantages include high curing shrinkage due to low filler content and weaker mechanical properties, which limits their use only for low stress bearing areas (14, 15).

Current strategies to promote bonding of the resinous materials to intrinsically wet substrates also include the incorporation of ionic and hydrophilic monomers into the adhesive. These adhesives etch and prime simultaneously, thus addressing the problems of collagen collapse and simplifying the bonding protocol. The original systems were two-step, self-etching systems, but in an effort to increase the efficiency of the procedure and reduce technique sensitivity, the manufacturers developed all-in-one single-step adhesives. The increased

concentration of acidic resin monomers provided a system that etched the dentin and enamel simultaneously (16).

The seventh generation or one-bottle self-etching system represents the latest simplification of adhesive systems. With these systems, all the ingredients required for etching and bonding are placed in and delivered from a single bottle (1,18). This greatly simplifies the bonding protocol as the claim was that could be achieved consistent bond strengths while completely eliminating the errors that could normally be committed with previous complicated systems. However, incorporating and placing all of the chemistry required for a viable adhesive system into a single bottle, and having it remain stable over a reasonable period of time, poses a significant challenge (18,19). These inherently acidic systems tend to have a significant amount of water in their formulations and may be prone to hydrolysis and chemical breakdown. Furthermore, once placed and polymerized, they are generally more hydrophilic than two-step self-etching systems; this condition makes them more prone to water sorption, limits the depth of resin infiltration into the tooth and creates some voids (20, 21).

The self-adhering flowable composite resin is based on bonding technology that relies on the adhesive monomer glycerol phosphate dimethacrylate (GPDM), a functional monomer to etch enamel and dentin. It also contains Hydroxylethylmethacrylate (HEMA) monomer, most commonly used in dental adhesives to improve wetting and resin penetration in dentin (23, 24) Dyad flow bonds in two ways: primarily through the chemical bond between the phosphate functional groups of a GPDM monomer and calcium ions of the tooth and secondarily, through a micromechanical bond as a result of an inter-penetrating network formed between the polymerized monomers of self-adhering flowable composite resin and collagen fibers of dentin (8). It is indicated that GPDM monomer etches rather than bonds to hydroxyapatite (32).

Bond strength is one of the significant factors, when selecting an adhesive system, that plays a major role for the long-term clinical success of the restoration [25]. Shear bond strength test is a simple evaluation procedure, reproducible and accepted commonly used to test the adhesion of dental adhesives (26). *In vitro* bond strength tests are useful and essential for predicting the performance of adhesive systems and possible correlation with clinical issues (27).

The present study analyzed the shear bond strength and microleakage of Dyad Flow (Kerr, Orange, CA, USA), which is a self adhering flowable composite with that of a self - etch based flowable composite Nano Filtek Z350 XT (3M ESPE, St Paul, MN, USA). When shear bond strength to dentin was evaluated, Dyad Flow recorded significantly lower shear bond strength than Nano Filtek flowable composite with Single bond universal adhesive. This may be due to the non-homogenous adhesive layer in Dyad Flow group which might explain its low bond strength. Other reasons could be the low dentin wettability of Dyad Flow might not have allowed an intimate contact between the material and dentin structure and consequently chemical interaction might have been limited. Also, the viscosity of Dyad Flow is considerably higher than that of single bond universal with Filtek flowable composite. Single bond universal, which is the main cause of

adhesion of Filtek flowable has been found to make deeper penetration into the dentin collagen network and more wetting to the dentine substrate than Dyad Flow which works superficially (28,29).

Besides, 10 – methacryloyloxydecyl dihydrogen phosphate (10-MDP) monomer is one of the most commonly used functional monomers. It is present in Single bond universal. The stronger adhesion capability of flowable composite is also due to this component. It is reported to be the most promising monomer for chemical bonding to hydroxyapatite of enamel and dentin due to its stability against hydrolysis and forming strong ionic bonds with calcium (30). It is a hydrophilic phosphate monomer that increases resin diffusion and adhesion by causing acidic decalcification and binding to calcium ions or amino groups of tooth structure. It is reported to be one of the most successful materials in the market for chemical bonding (31).

The results of the present study are in agreement with the result obtained by Tuloglu N et al (2014) (33) who found that shear bond strength values of Vertise flow, another self adhering flowable composite were lower than those for conventional flowable composite resins with one step self-etch adhesive(33). The present study was conducted on specimens in which class V cavities were prepared at level of cemento-enamel junction. The criteria for selecting class V cavities is because of more microleakage, which may lead to a weaker marginal seal; material can be dislodged during polymerization contraction, causing poor adaptation of the restoration at margins (34, 35).

Microleakage analysis of the specimens revealed that Dyad Flow, which is a self-etch and self-adhering flowable composite resin showed lower microleakage scores than Filtek flowable composite, although the difference was not statistically significant. Better marginal seal exhibited by Dyad Flow can be explained by the fact that it does not require additional step for adhesion. Moreover, micromechanical bond as result of intermingling of polymerized monomers of Dyad Flow and dentine collagen fibers also contributes to adhesion (36).

Both the flowable composite restorative materials that were used in the present study were not able to completely eliminate microleakage. This might be attributed to the difference in coefficient of thermal expansion, polymerization shrinkage, cavity configuration, lack of adhesion between restorative material and dentin, lack of adaptation of the restoration to the cavity wall. The present study measured microleakage using confocal microscopy at low magnification (×10), differing from other microleakage studies. Confocal Laser Scanning Microscopy is a nondestructive technique for visualizing subsurface tissue features. Confocal microscopy has a more advantages compared to other microscopy, due to principal of lens focus that occur some microns beneath the observed surface (37). This eliminates the stain spread caused by specimen sectioning and also avoids polishing artifacts that exaggerate dye penetration. Confocal microscopy observes the sample sequentially point by point and line by line and assembles the dried information into one image. By this advantage, assessment of microleakage will be accurate and reliable (38).

Conclusion

Within the limitation of the present study, it can be concluded by stating that there is definite benefit of Nano Filtek Z 350XT a self-etch based flowable composite resin on the property of shear bond strength which was comparatively higher when compared to Dyad flow a self-adhesive based flowable composite resin. However, microleakage was observed regardless of technique and materials used in class V restorations and all the specimens showed some amount of microleakage. Microleakage of Dyad Flow and Nano Filtek Z350XT were satisfactory and no difference between two groups was observed. The clinical relevance of microleakage between two flowable composites remains questionable, therefore there is a need for more clinical trials to make clinically relevant conclusion.

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