



Evaluation of Nanoleakage Patterns of Self-Adhering Flowable Composite Restoration / Dentin Interface

Maro George, Radwa Ibrahim, Hamdi Hosni

Date of Submission: 20-06-2024

Date of Acceptance: 30-06-2024

ABSTRACT:

Statement of problem: Self adhering flowable composite has a faster rate of polymerization shrinkage, a higher coefficient of thermal expansion, and weaker mechanical characteristics because of its lower filler content. The purpose of this vitro study was to evaluate nanoleakage patterns as this test is an important indication of a restorative material's sealability and hybrid-layer quality which both affect the longevity of the restoration.

KEYWORDS: Self-adhering flowable composite, Conventional flowable composite, Nanoleakage patterns

I. INTRODUCTION

Indeed, the introduction of self-adhering flowable composites (SAFC) represents a notable advancement in dental materials. These materials are designed to simplify the restorative process by eliminating the need for a separate adhesive, relying on chemical and micromechanical interactions to bond to tooth structure.

Glycerophosphate dimethacrylate (GPDM), a functional monomer, is used in bonding technology to etch dentin and enamel, and this process forms the basis of self-adhering flowable composite resin. Additionally, it contains hydroxyethyl methacrylate (HEMA), another functional monomer that is most commonly used in dental adhesives to enhance resin penetration and dentin wetting. This resin forms two types of bonds: a chemical bond between the phosphate functional groups of a GPDM monomer and the calcium ions of the tooth, and a micromechanical bond between the polymerized monomers of the self-adhering flowable composite resin and collagen fibers (as well as the smear layer) of dentin.

The essential stages of etching, washing, priming, and bonding are no longer necessary thanks to the self-etch technology integrated into the self-adhering flowable composite resin.

Consequently, SAFC required less effort, time, and complexity to employ. Furthermore, a decrease in postoperative sensitivity was seen. Clinical simplification of the restorative process has long been the goal, with the dual goals

of lowering the number of operational operations and potential errors resulting from several processes.

While polymerization shrinkage still poses a challenge, most restorations today use composites of all kinds as their preferred material. Also, at the composite-tooth interface, cuspal deflection or debonding may result from the contraction stress caused by this shrinkage.[1],[2],[3]

Unfortunately, because of their lower filler content, these materials have a faster rate of polymerization shrinkage, a higher coefficient of thermal expansion, and weaker mechanical characteristics. Collagen network collapse, partial resin infiltration into the exposed collagen network, insufficient polymerization, or the presence of residual water around collagen fibrils could all be contributing factors to "nanoleakage," a distinct pattern of leaking within the hybrid layer in nanoscaled areas. This pattern can occur in both the adhesive and hybrid layers, allowing oral fluid or bacterial products to pass through the interface and affecting the strength of the resin-dentin bond by hydrolyzing the collagen in the hybrid layer or the adhesive resin. Thus, evaluation of nanoleakage may be regarded as an important indication of a restorative material's sealability and hybrid-layer quality, both of that affect the longevity of the restoration.

"Nanoleakage" is a different type of leakage that happens in the hybrid layer in nanometer scaled spaces. It can be caused by incomplete polymerization, residual water around collagen fibrils, collapse of the collagen network, or imperfect infiltration of resin into the exposed collagen network. Both the adhesive layer and the hybrid layer may exhibit this pattern. The strength of the resin-dentin connection was affected by bacterial products or oral fluids penetrating across the interface because they hydrolyzed the collagen in the hybrid layer or the adhesive resin.[4]

II. MATERIALS AND METHOD

Twelve molars (N=12) used to evaluate Nanoleakage patterns test. Each group will be subdivided into 2 groups according to the material into : Self adhering and conventional flowable



composite. This subgroups are divided according to the time of the test into : Immediate (after 24 hours) and delay(after 6 months). The samples were divided into four main groups (n=6). Each main group was again subdivided into two groups according to testing time. The immediate sub group (n=3) and the delayed sub group (n=3)

Next, a block made of acrylic resin (Acrostone, Cario, Egypt) was placed over the samples. For every tooth, the occlusal enamel and superficial dentin were removed, revealing the middentinal region. Cutting was done perpendicular to each tooth's longitudinal axis using a precision diamond saw (IsoMet 4000 saw, Buehler Ltd., LakeBluff, USA) and plenty of water cooling (1:33 lubricant: water ratio).

The exposed dentin surfaces were prepared with 600-grit silicon carbide paper while running water was present in order to create a uniform smear layer.

A transparent self-adhesive matrix was fixed around the tooth to support composite resin material build up. A composite resin layer of (2 mm thickness) was applied over the treated dentin surface and the buildup was continued until a (4mm) thickness layer of composite was created covering the bonded dentin surface, then in accordance with the manufacturer's recommendations, all bonding agent was applied to the cut surfaces, and flowable composites restorative material were placed on top. (Fusio liquid dentin and Flow it-ALC). Specimens were stored in de-ionized water and placed in an incubator at 37°C until testing. De-ionized water was changed every week during the 6-month storage of the delayed groups till testing. Using an LED light-curing device. The band was taken off once the composite had fully cured.

The restored teeth were then divided into a series of 1 mm thick slabs under water cooling then they were mounted horizontally and again sectioned longitudinally into a series of 1 mm thickness. The sectioning was performed using a diamond disc of 4 diameter × 0.3 mm thickness with diamond cutting blades and water-resistant titanium coating with low speed saw to obtain beams of 1.0 mm² cross-section area. The specimens were then subjected to the nanoleakage evaluation. Two coats of Nail Vanish were applied to every surface, one millimeter away from the bonding interface.

The specimens were left in an aqueous solution containing 50% ammoniacal silver nitrate (PH = 9.5) for 24 hours at 37°C in total darkness after the nail polish had dried. They were then rinsed with distilled water for five minutes and

exposed to an 8-hour photo-developing solution (Kodak GBX fixer and replenishers, Kodak; Rochester, NY, USA) under fluorescent light to allow the diammine silver ions to be reduced to metallic silver grains within voids along the bonded interface.[5]

After a 5-minute soak in distilled water, the nail varnish was removed with a periodontal scaler. Using waterproof silicon carbide papers with grits of 800-, 1200-, 2000-, and 4000-grits, specimens were wet polished to remove the silver's surface layer. The desired finish was attained by gradually applying fine diamond pastes (20 µm, 6 µm, 4 µm, and 1 µm, respectively) using a polishing cloth. After removing debris from the specimens with an ultrasonic bath for ten minutes (using a digital ultrasonic cleaner from Codyson, China), they were air dried and cleaned ultrasonically. Using graphite paint and carbon adhesive tape, specimens were fastened on aluminum stubs (Ted Pella, Inc., Moorestown, NJ, USA). With the use of a high resolution SEM operating in backscattered mode and capable of an accelerating voltage of 20 K V, a working distance of 7–13 mm, and magnifications of x500, x1000, and x2000, all specimens were gold sputtered (SPI Module - Sputter Carbon / Gold Coater, EDEN instruments, Japan).(FEI Company, the Netherlands;SEM Quanta FEG 250 with field emission gun).[6]

III. RESULT

For both conventional flowable and self-adhering flowable composites, a nanoleakage test demonstrates the intake patterns of silver nitrate at the resin/dentin interfaces both before and after six months of storage.

Three separate patterns of nanoleakage were identified: reticular pattern, which are silver deposits in a dendritic form within the bulkiness of the HL; spotted pattern, which are tree-like and grow vertically from the HL surface in the shape of terminal branches towards the adhesive; and water-trees pattern.

A thick continuous layer of silver nitrate absorption along the resin-dentin interface and reticular pattern extending toward dentin are present in the delay self adhering flowable composite group. However, in the immediate self adhering flowable group with water tree pattern as well, there were areas of silver grain deposits at the interface and reticular pattern as terminal branches that extended vertically from the HL surface to the adhesive layer.

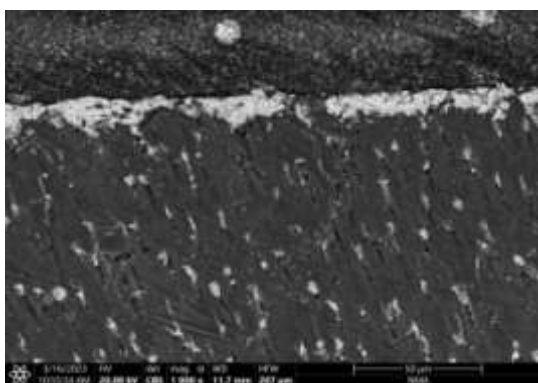
Following the storage of the conventional flowable composite, little spots of silver grains are



discovered along with a thin reticular pattern of discontinuous islands of silver deposits at the base of the HL. The water-tree structures in the immediate group, on the other hand, showed a significant increase in size and density and extended toward the dentin at the base of the HL.

Overall, the delayed groups' uptake of silver grains in HL is greatly more than that of the immediate groups' uptake of silver grains in HL.

Silver deposits within the adhesive layer of the hybrid layers made by self-adhering flowable composite for both the immediate and delayed groups demonstrated the most severe silver uptake. On the other hand, in both the immediate and delayed groups, the spot mode of silver uptake was lower in conventional flowable composite.



IV. DISCUSSION

An ideal restorative material should have a natural appearance, be biocompatible, and be mechanically durable. It should also exhibit bulk-fill properties, bioactivity, and rapid, long-lasting adhesion to tooth tissues. Recent developments include the introduction of resinous self-adhering flowable composite.

Fusio Liquid Dentin is a novel material that is now available to the dentistry market. It is a self-adhering flowable composite (SAFC). It is stated that this material will simplify the restorative process by removing the requirement for an additional bonding step. Because of this, SAFC might be seen as the beginning of the eighth generation of dental adhesive systems or as a connection between flowable composite and all-in-one adhesive systems.

Based on adhesive technology, it etches enamel and dentin using a functional monomer (glycerophosphate dimethacrylate [GPDM]) and enhances wetting and resin infiltration into the dentin substrate using a hydrophilic monomer (e.g., hydroxyethyl methacrylate). This resin forms two types of bonds: one is micromechanical, connecting the polymerized SAFC resin to collagen fibers and

the dentin smear layer, and the other is chemical, connecting the functional monomer to the hydroxyapatite of the tooth structure.

Stable calcium-phosphate complexes can be formed using a functional monomer, such as 10-methacryloyloxydecyl dihydrogen phosphate (10-MDP). Another functional monomer, GPDM, has the advantage of having two polymerizable groups that can react with other monomers in resin composites and adhesive systems, improving the mechanical characteristics and polymer network quality.

GPDM, on the other hand, demonstrated hydrophilicity and more demineralization of dentin than hydroxyapatite's calcium bonding, resulting in an unstable complex of di-calcium phosphate dehydrated deposited on the surface of the material that will eventually dissolve in an aqueous environment, compromising the interfacial integrity.[7]

Flow it- ALC is a conventional flowable composite which was selected as the comparator. Resin matrix of this flowable composite contains Bis-GMA, TEGDMA,

EBADMA with size Filler content 0.04–4 um and load 66% wt.

In this study, a unique one-step adhesive system called Bond 1 SF (Pentron Clinical, California, USA) was used with conventional flowable composite. It was developed to eliminate technical concerns regarding solvent evaporation and the durability of the resin-dentin bond by excluding both water and organic solvents from the ingredients.

This current study included nanoleakage testing. Regarding both short- and long-term adhesion, the impact of nanoleakage on adhesive/dentin bonding stability has been a source of concern.

Numerous investigations have connected the development of blisters or gaps in the adhesive or HL to the following: insufficient resin tags and monomer infiltration; unfinished solvent/water removal from the adhesive layer; and functional monomer-hydroxyapatite interaction.

An important factor in evaluating the material sealing capacity and HL quality—both of which affect the restoration's long-term endurance—is the identification of nanoleakage both within the HL and at the adhesive-resin contact.[8]

When Sano et al.[9] noticed gap-free restorations when the microleakage test scored zero, they created a novel testing method they called "nanoleakage." The specimens were submitted to a 24-hour period of complete darkness



in a silver nitrate solution by Sano et al.[10]Following this, the bonded specimens were submerged in a photo-developing solution and exposed to fluorescent light for an additional 8 hours. Next, employing the backscattered electron mode in SEM, the bonded interface was detected. Three distinct patterns of nanoleakage—reticular, spotted, and water-trees—were noted.

It has been demonstrated in later research that universal adhesives exhibit nanoleakage in the hybrid layer as well.

The impregnation of silver grains within the hybrid layer's porosities that were improperly filled with adhesive glue is what was first defined as "nanoleakage." Additionally, the "reticular mode," or second mechanism of nanoleakage, has been explained. These thin, branching nanovoid channels are believed to represent morphological expressions of the water treeing phenomena, most likely caused by aging. Water-induced polymer degradation is assumed to be caused by aging."Spotted" and "reticular" patterns of nanoleakage were noted in this study. The lack of a "water-tree" pattern may be explained by the fact that all samples were processed and examined under a scanning electron microscope on the same day, removing the influence of age. depict the Dyad Flow, Fusio Liquid Dentin image obtained with a scanning electron microscope.[11]The purpose of this study was to assess and compare, using the nanoleakage test, the sealability of self-adhering flowable composite. The study's findings demonstrated that conventional flowable composites have sealability comparable to that of self adhering flowable composites.

Compared to self-etch adhesives, Mobarak and Seyam [12]discovered that all of the self-adhesive solutions they evaluated exhibited very little nanoleakage.This could be explained by the fact that when the hybrid layer is thicker and the demineralized dentin is deeper, there is a greater chance of nanoleakage, which allows silver ions to penetrate into the hybrid layer and partially or completely demineralized dentin.

However, studies by Tay et al.[13], Hashimoto et al.[14], and El-Badrawy et al.[15]revealed that self-adhesive systems had a significant level of nanoleakage and this is agree with our study.

According to the authors, this could be because there is some residual water present, which is held in place because of its poor vaporization in the presence of HEMA, increasing the uptake of silver. Fine silver is deposited at the bonded interface as a result of HEMA copolymerizing with low pH resin monomers to create homologous

hydrogels in the presence of water . Remaining water may be retained because of its low vaporization in the presence of HEMA. Lastly, in the presence of water, HEMA copolymerizes with low pH resin monomers to generate homologous hydrogels that permit fine silver deposition at the bonded interface.

In contrast with this study , Al-Agha EI et al.[16]examined the nanoleakage of Class V composite restorations in order to evaluate the sealability of a self-adhesive flowable composite in comparison to two adhesive systems. When compared to solvent-free and self-adhesive adhesives, the self-adhesive flowable composite demonstrated the statistically substantially lowest mean nanoleakage, indicating that its sealability was superior. This was in line with the findings of Mobarak and Seyam [17], who discovered that, in comparison to self-etch adhesives, all investigated self-adhesive systems exhibited very little nanoleakage.This may be because silver ions can permeate both the partially or totally demineralized dentin and the hybrid layer, as well as partially polymerized adhesive resin, when the demineralized dentin is deeper and the hybrid layer is thicker. Due to the fact that the critical index of hybrid-layer quality is the amount of silver uptake per unit depth or volume, and because the self-adhering flowable composite prevented any prior demineralization of dentin, the hybrid layer's thickness and the depth of demineralized dentin were minimal when compared to the other self-etching adhesive systems used in this study.[18]

Additionally,compared to other resin restorative materials ,SAFC is more self-adaptable because it is flowable .flowable composites may have less stress development than hybrid ones with stiffer materials,larger polymerization strees are produced due to the limitef mobility of polymer chains [19][20]

V.CONCLUSION

The amount of silver nitrate uptake increased in self-adhering flowable after storage.So,nanoleakage is increased in self-adhering flowable composite more than conventional flowable composite.

REFERENCE

- [1]. Chan K, Yanjie M, Harry K, Tong K, Desmond N, Hsiao J. Review: Resin Composite Filling. Materials. 2010;3.
- [2]. Demarco FF, Corrêa MB, Cenci MS, Moraes RR, Opdam NJ. Longevity of posterior composite restorations: not only



- a matter of materials. *Dent Mater.* 2012;28:87-101.
- [3]. Bicalho AA, Valdívia AD, Barreto BC, Tantbirojn D, Versluis A, Soares CJ. Incremental filling technique and composite material--part II: shrinkage and shrinkage stresses. *Oper Dent.* 2014;39:E83-92.
- [4]. El-Keredy D, Etman W, Salama M. Nanoleakage of different composite restoration systems. *Tanta Dental Journal.* 2020;17:97.
- [5]. Bakhsh T, Al-Zayer M, Al-Sahwan N, Al-bahran Z, Bakry A, Jamleh A, et al. Comparative SEM Observation of Silver-Nitrate at Resin-Dentin Interface: Nanoleakage Study. *Oral Health and Care.* 2017;2.
- [6]. Alkaffas A, Hamama H, Mahmoud S. Do universal adhesives promote bonding to dentin? A systematic review and meta-analysis. *Restorative Dentistry & Endodontics.* 2018;43.
- [7]. Shaalan OO, Abou-Auf E, El Zoghby AF. Clinical evaluation of self-adhering flowable composite versus conventional flowable composite in conservative Class I cavities: Randomized controlled trial. *J Conserv Dent.* 2018;21:485-490.
- [8]. Andia-Merlin RY, Garone-Netto N, Arana-Chavez VE. SEM evaluation of the interaction between a three-step adhesive and dentin. *Oper Dent.* 2001;26:440-444.
- [9]. Sano H, Shono T, Sonoda H, Takatsu T, Ciucchi B, Carvalho R, et al. Relationship between surface area for adhesion and tensile bond strength--evaluation of a micro-tensile bond test. *Dent Mater.* 1994;10:236-240.
- [10]. Tay FR, King NM, Chan KM, Pashley DH. How can nanoleakage occur in self-etching adhesive systems that demineralize and infiltrate simultaneously? *J Adhes Dent.* 2002;4:255-269.
- [11]. Sachdeva P, Goswami M, Singh D. Comparative evaluation of shear bond strength and nanoleakage of conventional and self-adhering flowable composites to primary teeth dentin. *Contemp Clin Dent.* 2016;7:326-331.
- [12]. Mobarak E, Seyam R. Interfacial nanoleakage and bonding of self-adhesive systems cured with a modified-layering technique to dentin of weakened roots. *Oper Dent.* 2013;38:E154-165.
- [13]. Mobarak E, Seyam R. Interfacial nanoleakage and bonding of self-adhesive systems cured with a modified-layering technique to dentin of weakened roots. *Oper Dent.* 2013;38:E154-165.
- [14]. Hashimoto M, De Munck J, Ito S, Sano H, Kaga M, Oguchi H, et al. In vitro effect of nanoleakage expression on resin-dentin bond strengths analyzed by microtensile bond test, SEM/EDX and TEM. *Biomaterials.* 2004;25:5565-5574.
- [15]. El-Badrawy W, Hafez RM, El Naga AI, Ahmed DR. Nanoleakage for Self-Adhesive Resin Cements used in Bonding CAD/CAD Ceramic Material to Dentin. *Eur J Dent.* 2011;5:281-290.
- [16]. Al-Agha EI, Alagha MI. Nanoleakage of Class V Resin Restorations Using Two Nanofilled Adhesive Systems. *J Int Oral Health.* 2015;7:6-11.
- [17]. Mobarak E, Seyam R. Interfacial nanoleakage and bonding of self-adhesive systems cured with a modified-layering technique to dentin of weakened roots. *Oper Dent.* 2013;38:E154-165.
- [18]. Naga AA, Yousef M, Ramadan R, Fayeze Bahgat S, Alshawwa L. Does the use of a novel self-adhesive flowable composite reduce nanoleakage? *Clin Cosmet Investig Dent.* 2015;7:55-64.
- [19]. Vichi A, Goracci C, Ferrari M. Clinical study of the self-adhering flowable composite resin Vertise Flow in Class I restorations: Six-month followup. 2011;5:14-24.
- [20]. Sano H, Takatsu T, Ciucchi B, Horner JA, Matthews WG, Pashley DH. Nanoleakage: leakage within the hybrid layer. *Oper Dent.* 1995;20:18-25.