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MICROMORPHOLOGICAL ANALYSIS OF DIFFERENT BIOACTIVE RESTORATIVE MATERIALS/DENTIN INTERFACE: A COMPARATIVE IN VITRO STUDY

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ABSTRACT

Objective: This study was conducted to analyze the interfacial micromorphology of three commercially available bioactive restorative materials to dentin.

Materials and methods: Three different bioactive restorative materials were utilized: Surefill One (self-adhesive hybrid composite), Cention Forte (Alkasite-based restorative material), and Fuji II LC (resin-modified glass ionomer). A total of fifteen sound permanent molars were collected and randomly divided into three groups (n=5) according to the restorative material used. Following exposure of mid-coronal dentin of teeth through occlusal grinding, the restorative materials were applied according to manufacturer instructions. Specimens were prepared for SEM analysis through vertical sectioning with a diamond saw, polishing, acid-base challenge exposure, and final cleaning and drying. Scanning electron microscopy examination was conducted at 2000x magnification in secondary electron mode.

Results: Regarding the interfacial micromorphological analysis of the three tested materials using SEM, there was a significant difference between the tested groups; The hybrid layer and acid resistance layer were observed in alkasite based restorative material and resin-modified glass ionomer groups but the interfacial gaps were observed in self-adhesive hybrid composite group.

Conclusion: Alkasite based restorative material bonded most effectively and demonstrably to dentin compared to the other materials.

KEYWORDS: Alkasite, Self-adhesive hybrid composite, ion releasing restorative materials , Microscopic analysis

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INTRODUCTION

Composite restorative materials have dominated dental practice for the past five decades, primarily due to their excellent aesthetics and mechanical properties. However, early iterations presented limitations. They often allowed plaque formation and bacterial proliferation, leading to recurrent caries and restoration failure.[9]

This shortcoming spurred advancements in material science. One key development involved enriching composite resins with desirable properties of glass ionomer cements (GICs). GICs offer advantages such as ease of application and the ability to release fluoride ions, promoting tooth remineralization.[14] This led to the development of hybrid materials, a significant leap forward in conservative dentistry.

These innovative hybrid materials, encompassing self-adhesive, bulk-fill, and ion-releasing categories, offer distinct advantages. Self-adhesive properties eliminate the need for separate adhesives, reducing the risk of contamination from blood or saliva and mitigating potential post-operative sensitivity associated with traditional adhesives. Bulk-fill capability further streamlines the restoration process. The ability to release ions, another crucial feature, fosters a more bioactive environment. This study focused on three specific types of bioactive restorative materials: self-adhesive hybrid composites, Alkasites, and resin-modified glass ionomer (RM-GIC) cements.

Self-adhesive hybrid composites were introduced in 2019, this novel category presents a unique approach by combining the established aesthetics of composites with properties similar to traditional GICs. Notably, these materials are comprised of two key components: MOPOS monomers (Modified Polyacid System), which play a crucial role in adhesion and strength, and BADEP monomers (N,N'Bis-acrylamido-N,N'-diethyl-1,3-propandiamine), acting as cross-linking agents.[5, 7]

Alkasites based restorative material is a subgroup within resin-based composites (RBCs), Alkasites utilize an alkaline filler system that neutralizes acids, hence their moniker. Introduced in 2016, this category boasts self-curing properties.[3] The liquid component is predominantly composed of methacrylate monomers, while the powder component consists of a unique blend of inorganic glass fillers, including an "Alkasite" filler, a standard inert glass, and an ionomer glass.[12, 13]

GICs hold a distinguished position among restorative materials due to their inherent bioactivity, facilitating a direct interaction with hard tooth structures through a self-adhesion mechanism.[11,8] This characteristic has established them as the gold standard for self-adhesive direct restorations. Developed in the late 1960s, conventional GICs faced limitations regarding setting time, mechanical properties, and early moisture sensitivity.[6] The introduction of 2-hydroxyethylmethacrylate (HEMA) as a resin component led to the development of RM-GICs, addressing these shortcomings while retaining the bioactivity of GICs.[4] Conventional and RM-GICs inherently bond to tooth structure via a combination of micromechanical interlocking and ionic interactions.[14]

Given the continuous development in restorative materials and their potential impact on clinical outcomes, a thorough understanding of their interfacial micromorphology with tooth dentin is crucial. This study aims to evaluate and compare the interfacial micromorphology of these three commercially available bioactive restorative materials to dentin using scanning electron microscopy (SEM) By elucidating the interfacial characteristics, this research can contribute valuable insights into the long-term success and efficacy of these restorative materials.

MATERIALS AND METHODS

Three different self-adhesive bulk-fill restorative materials were used in this current study. These materials included: Self-adhesive bulk-

fill composite under the trade name Surefil One ('SU-O') (Dentsply Sirona, Konstanz, Germany), New category of restorative material so-called"Alkasite" which was Cention forte ('CNF') (Ivoclar Vivadent; Schaan, Liechtenstein) and Resin-modified glass ionomer restorative material (GIC) which was Fuji II LC ('FJI') (GC; Tokyo, Japan). All restorative materials were applied following the respective manufacturer's instructions. All details about the tested materials are shown in **Table 1.**

Teeth selection

Fifteen freshly extracted, intact, non-carious, and unrestored human permanent molars were used. Teeth were obtained from the outpatient clinic of the Faculty of Dentistry, Mansoura University, with informed consent following protocols approved by the Faculty Ethical Committee (A09030123). The reason for extraction was solely due to periodontal disease.

Upon extraction, teeth were cleaned to ensure optimal sample preparation. Calculus and soft-tissue deposits were removed using a hand scaler. Subsequently, the molars were cleaned with a rubber cup and a fine pumice water slurry to eliminate any residual debris. Following this cleaning procedure, the teeth were stored in a 0.5% chloramine T solution at a controlled temperature of 4°C (Faculty of Pharmacy, Mansoura University, Egypt) for a period of three months. The storage procedures employed adhered to current international and institutional infection control guidelines.

TABLE (1) Materials used in the study

Materials	Specification	Manufacturer	Composition	Code Batch no.(lot)
Cention Forte	"Alkasite-based restorative material"	Ivoclar Vivadent; Schaan, Liechtenstein	Barium aluminum silicate glass, ytterbium trifluoride, pre-polymerized filler, calcium barium aluminum fluorosilicate glass, and calcium fluoro-silicate glass, UDMA, tricyclodecan-dimethanol dimethacrylate, tetramethyl-xylylene diurethane dimethacrylate, polyethylene glycol, 400 dimethacrylate, and Ivocerin	**('CNF')** ZL08SV
Cention primer	A two-component self etching and self-curing primer	Ivoclar Vivadent, Schaan, Liechtenstein	Liquid: bisphenol A glycerolate dimethacrylate, 2-hydroxyethyl methacrylate, methacrylated phosphoric acid,1,10-decandiol dimethacrylate, methacrylate modified polyacrylic acid, 2-dimethylaminoethyl methacrylate, ethanol, camphorquinone.	**('CP')** Z031Z2
Fuji II LC	Resin-modified glass-ionomer.	GC; Tokyo, Japan	Fluoro-alumino-silicate glass, Polybasic carboxylic acid, UDMA, HEMA, Water,Inatiator.	**('FJI')** 2302132
Dentin Conditioner	polyacrylic acid-etch	GC; Tokyo, Japan	(GC; 20% polyacrylic acid, 3%aluminum chloride, distilled water)	**('DC')** 5040518
Surefil one	Self-adhesive bulk-fill hybrid resin composite.	Dentsply Sirona, Konstanz, Germany	Aluminum-phosphor-strontium-sodium-fluoro-silicate glass, highly dispersed silicon dioxide, ytterbium fluoride, iron oxide pigments, titanium dioxide pigments ,polycarboxylic acid, acrylic acid, bifunctional acrylate, water, self-cure initiator, camphorquinone, stabilizer.	**('SU-O')** 2201000713

Study design

The teeth (n=15) were used for micromorphplogical analysis and were randomly assigned to three groups (n=5 per group) corresponding to the type of restorative material being investigated: (Each group had five molars) Surefil one (self-adhesive bulk-fill composite group), Cention fort (Alkasite group), and Fuji II LC (resin-modified glass ionomer control group). (**Figure 1)**

Mounting of teeth

The roots of each extracted tooth were embedded in self-cure acrylic resin (Acrostone, Heliopolis, Cairo, Egypt). The embedding process ensured that the cement-enamel junction (CEJ) remained exposed for approximately 2 mm. Polyvinyl chloride (PVC) tubes (2 cm diameter and 2.7 cm height) were employed as molds for this procedure.

Each tooth specimen was positioned within a PVC mold, ensuring its flat occlusal surface rested flush against a glass slab. Subsequently, the self-cure acrylic resin was carefully packed into the surrounding space within the mold, completely encasing the tooth roots. This embedding technique provided a stable base for subsequent grinding and polishing procedures required for the micromorphological analysis

Specimen preparation

For each tooth, occlusal enamel and superficial dentin were cut away exposing the mid-coronal dentin producing flat dentin surface. To access the mid-coronal dentin for analysis, a pre-operative radiograph was used in conjunction with a pre-measured periodontal probe to determine the location of the mid-coronal dentin, a ruler was employed to measure the occlusal enamel thickness. Subsequently, the total dentin thickness was measured from the occlusal surface to the roof of the pulp chamber. Finally, the mid-point of the dentin layer was identified, pinpointing the target region for sectioning. A clear line was scribed on the external tooth surface to mark the predetermined mid-coronal dentin location.

A low-speed, automated diamond saw (PICO 155 precision saw, Pace technologies, Tucson, AZ, USA) equipped with a water coolant system (Diacut Water-based Cutting Fluid Pace technologies, Tucson, AZ, USA) at a 1:33 lubricant-to-water ratio was utilized to perform a precise cut perpendicular to the longitudinal tooth axis. This sectioning technique ensured a clean and controlled exposure of the mid-coronal dentin. The exposed dentin surfaces were then meticulously polished using 600-grit silicon carbide paper (Microcut, Buehler, Lake Bluff, IL, USA) under running water for a standardized duration of 30 seconds with a rotational motion. This polishing step aimed to create a uniform smear layer on the dentin surface for optimal analysis.

Following the manufacturer's instructions, each designated self-adhesive restorative material was applied to the prepared dentin surfaces. A standardized height of 2 mm was achieved using a Tofflemire matrix system with periodontal probe (used to detect 2mm hight of Tofflemire band). A glass slide was employed to gently compress the restorative material, ensuring excess material was expelled and minimizing surface voids.

The specimens were sectioned buccolingully using a low-speed, water-cooled diamond saw (PICO 155 precision saw, Pace technologies, Tucson, AZ, USA). Two vertical sections were made along the long axis of each tooth,

Perpendicular to the restoration-dentin interface, resulting in semi-equal halves. Subsequently, horizontal sections were made at the level of the cement-enamel junction (CEJ) to separate the teeth from the acrylic resin embedding material.

Individual resin-dentin slabs with a thickness of 2 mm were obtained from each half. These slabs were meticulously polished using a sequence of

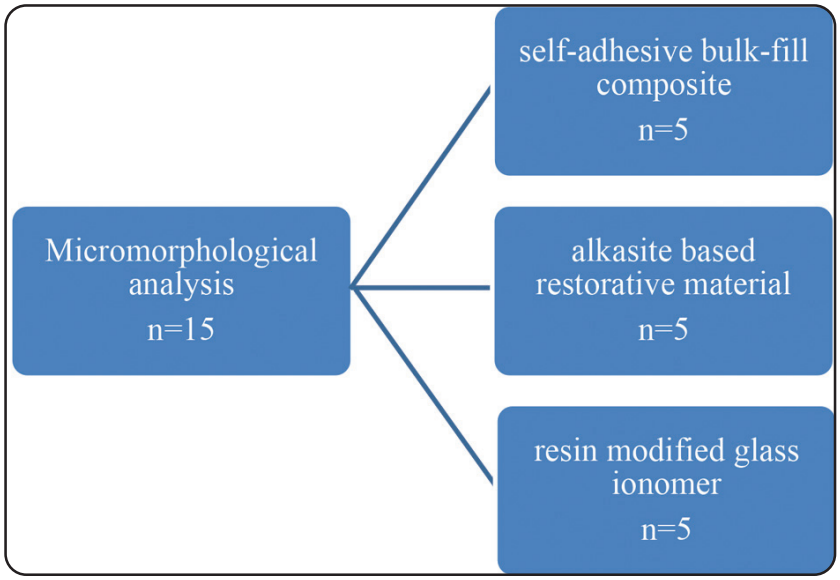

Fig. (1) Diagram illustrate study design for micromorphological pattern.

silicon carbide papers (600, 1000, 1200, and 2000 grit) followed by increasingly finer diamond pastes (6 µm, 4 µm, and 1 µm) on a polishing cloth (Meta-di™, Buehler, Lake Bluff, IL, USA). Ultrasonic cleaning in a bath (XH-E412 ultrasonic cleaner, Xinghua Ltd, China) for 10 minutes ensured the removal of any remaining debris.

To prepare the specimens for SEM analysis, an acid-base challenge was performed. Specimens were immersed in a saline solution at room temperature for 10 minutes, followed by sequential exposure to 10% orthophosphoric acid solution (5 seconds) and 5% sodium hypochlorite solution (5 minutes). This process demineralized any non-infiltrated dentin, facilitating dehydration. The samples were subsequently dried and stored for 24 hours to prevent over-hydration during the gold plating step.

Finally, the specimens were gold-sputtered using (SPI Module Sputter Carbon/Gold Coater, EDEN instruments, Japan). This coating allows for optimal conductivity during SEM imaging. SEM analysis was performed using scanning electron microscope ((JSM6510LV, JEOL, Japan) in secondary electron detection mode. The accelerating voltage was set at 30 kV with a working distance of 10-15 mm, and images were captured at a magnification of 2000x

RESULTS

Micromorphological evaluation

Scanning electron microscopy (SEM) micrographs were obtained at a magnification of 2000x to evaluate the morphology of the resin-dentin interface for each restorative material group (Figures 2-4). The SEM image of SU-O group revealed a discontinuous material-dentin interface. Furthermore, there was a complete absence of resin tags and the presence of separation. A hybrid layer was not observed. **(Figure 2)**

In contrast to the SU-O group, the CNF group displayed a continuous material-dentin interface. The interface was characterized by the presence of long and thick resin tags with a broad tubular pattern, indicating effective penetration of the adhesive resin into the dentin tubules. Minor interfacial gaps were evident, potentially representing remnants of the smear layer. A well-defined hybrid like layer was demonstrably present at the interface. **(Figure 3)**

The dentin surface in the FJI group exhibited complete removal of the smear layer, exposing open dentinal tubules. The FJI material demonstrated intimate contact with the underlying dentin. Resin tags were observed, including budding tags and

a limited number of short, thick tags with a funnel-shaped morphology, indicating micromechanical interlocking within the dentin tubules. Importantly, there was an absence of separation or interfacial gaps, and a hybrid-like layer was evident. **(Figure 4)**

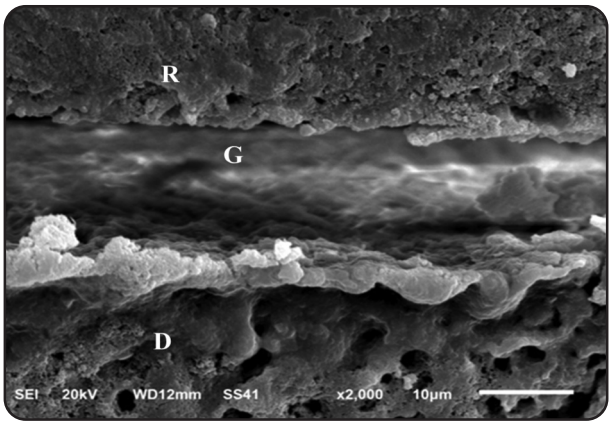

Fig. (2) SEM micrographs showing the resin-dentin interface of surefil one restorative material. SEM micrographs in (X2000) magnification respectively showing no resin tags penetrating the dentin surface with evidence of separation, and the smear layer was not completely removed. The material/dentin interface appears discontinuous. R; restoration, D; dentin, layer, G; interfacial gap

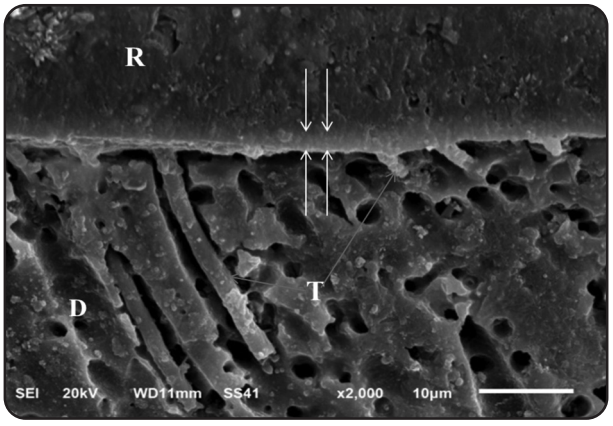

Fig. (3) SEM micrographs showing the resin-dentin interface of cention forte with cention primer restorative material. SEM micrographs at magnifications of (X2000), show long thick resin tags arranged in a tubular form that penetrate dentin surface. There is minor interfacial gap that represent the smear layer can be observed. Displaying the production of acid base resistance layer and thick hybrid like layers in reticular patterns. R: restoration, D: dentin, white arrows: hybrid like layer, T: resin tags.

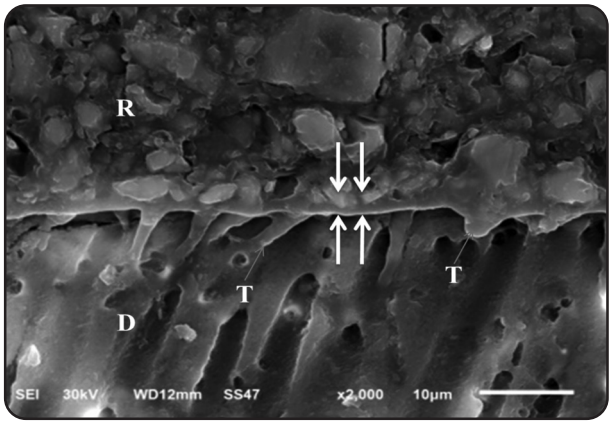

Fig. (4) SEM micrographs showing the resin-dentin interface of Fuji II LC (RMGIC) group restorative material. (X2000) high magnification SEM micrographs are presented. They show budding resin tags penetrating into dentin surface. It shows a hybrid like layer formation and acid base resistance zone (white arrows) with the production of short thick funnel-shaped configuration of resin tags penetrating conditioned dentin surface with no signs of separation throughout the interface with absence of smear layer. R: restoration, D: dentin, T: resin tags.

DISCUSSION

Bioactive restorative materials have emerged as a focal point of investigation in contemporary restorative dentistry. Their potential to promote remineralization within dental tissues represents a significant advancement in this field. This unique characteristic transcends the realm of simply restoring tooth structure; bioactive materials possess the ability to actively contribute to the regeneration and repair of dentin following caries or other insults**.**

The present study investigated the interfacial micromorphology of three commercially available bioactive restorative materials: self-adhesive hybrid composites (the protocol of this study was designed while this material still available in the market), Alkasites, and resin-modified glass ionomer (RM-GIC) cements that used as control material. By employing scanning electron microscopy (SEM), aimed to evaluate and compare the interfacial characteristics of these materials with dentin. Understanding the nature of this interface is crucial for optimizing the long-term efficacy and clinical success of these novel restorative materials.

The present study evaluated the efficacy of Surefil one, this self-adhesive bulk-fill composite was applied directly to the prepared dentin surface without any pre-treatment, Cention Forte (CNF), to promote adhesion, the dentin surface was treated with Cention Primer, a dedicated self-adhesive bonding system designed for use with CNF. Subsequently, CNF was applied according to the manufacturer's instructions. As a control material, a resin-modified glass ionomer cement (RMGIC), Fuji II LC, was utilized. Following the manufacturer's recommendations, the dentin surface was pre-conditioned with polyalkenoic acid before FJI application. RMGICs are known for their inherent self-adhesive properties and represent a widely used bioactive material category for permanent restorations. The pre-conditioning step with polyalkenoic acid is a well-established approach to enhance the bond strength of RMGICs to dentin.

Scanning Electron Microscopy (SEM) offers a powerful tool for examining the intricate details of dental hard tissues and restorative materials. This advanced imaging technique allows for: the evaluation of surface topography, roughness, and interfacial interactions between various dental materials. SEM analysis plays a critical role in assessing the marginal integrity between restorative dental materials and tooth enamel/dentin. A strong and well-sealed interface is essential for ensuring optimal restoration longevity and preventing the infiltration of bacteria and fluids that can lead to failure. SEM can be employed to investigate the bioactive properties of novel dental materials. By analyzing the interaction of these materials with the surrounding dentin, assess their potential to promote remineralization or exhibit other beneficial biological effects.[1, 10]

The findings of this in-vitro investigation utilizing scanning electron microscopy (SEM) analysis at 2000x magnification (Figures 2-4) elucidate distinct characteristics at the resin-dentin interface for each of the investigated bioactive restorative materials. Scanning electron microscopy (SEM) analysis revealed obvious differences in the interfacial micromorphology between the restorative materials and dentin across the investigated groups.

Surefil One (SU-O) group displayed a concerning lack of adhesion to the dentin substrate. The material-dentin interface appeared discontinuous, devoid of material deposition within dentinal tubules, resin tag penetration, or a discernible hybrid layer (Figure 2). These observations were consistent with the absence of a pre-treatment step for SU-O, suggesting an inability to form a chemical bond with dentin. The presence of interfacial gaps and irregularities further supports this conclusion.

Cention Forte (CNF) group , in contrast to SU-O, exhibited a demonstrably superior interface with dentin (Figure 3). The CNF group presented a continuous interface characterized by long, thick resin tags with a broad tubular pattern, indicative of substantial penetration into the dentin substrate.. Notably, a well-defined hybrid layer was evident. These morphological features strongly suggest effective adhesion between CNF and dentin, likely attributable to the utilization of the dedicated Cention Primer. This pre-treatment presumably enhances the material's bonding capability through a targeted mechanism.

Fuji II LC group displayed an intimate contact at the interface with complete removal of the smear layer and exposed dentinal tubules (Figure 4). While short, thick resin tags were present, their penetration depth was considerably less extensive compared to the CNF group. A hybrid-like layer was also discernible. The pre-conditioning step employing polyalkenoic acid likely facilitated a certain degree of adhesion between FJI and dentin. However, the observed morphology suggests a potentially weaker bond compared to CNF.

These observations align with the established functionalities of the materials. SU-O, lacking a dedicated pre-treatment step, may not achieve sufficient dentin conditioning for optimal

bonding. CNF, with its pre-treatment primer, appears to effectively prepare the dentin surface for micromechanical interlocking. FJI, a pre-conditioned RM-GIC, likely benefits from the combined effect of its chemical bonding mechanism and the pre-conditioning process.

These findings are consistent with established principles of adhesion in dental restorations. As highlighted in the introduction, an ideal restorative material should exhibit exceptional bonding to dentin to ensure long-term clinical success. Inadequate adhesion can lead to a detrimental cascade of events, including marginal leakage, discoloration, and ultimately, restoration failure.[2]

LIMITATIONS

It is important to acknowledge that the current investigation focused solely on the interfacial micromorphology. Further research is warranted to evaluate the mechanical properties of the bond between these materials and dentin. Additionally, in-vivo studies would provide invaluable insights into the long-term clinical performance of these restorative materials.

CONCLUSION

The results suggest that Cention Forte exhibited the most favorable interface, the self-adhesive bulk-fill composite Surefil One (SU-O) demonstrated markedly inferior interfacial characteristics compared to Cention Forte (CNF) used in conjunction with its dedicated primer. The pre-conditioned Fuji II LC (FJI) exhibited an intermediate level of adhesion. These findings unequivocally emphasize the importance of a compatible pre-treatment agent in significantly enhancing the bonding efficacy of self-adhesive restorative materials to dentin. For optimal clinical outcomes, meticulous consideration of the material's bonding characteristics is paramount when selecting a restorative material.

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Not applicable

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