



## The Effect of Different Adhesive System Application Modes on Nanoleakage At Superficial And Deep Dentin After Varying Storage Interval

Mohamed Ahmed Wakwak <sup>1\*</sup>, Mahmoud El-Said Ahmed Abd El-Aziz<sup>2</sup>, Karim Sherif Adly Dewedar<sup>3</sup>, Abdelmonem Abdalla Amr<sup>4</sup>, Labib Mohammed Labib Elsebaey<sup>5</sup>

Codex : 12/2023/04

Aadj@azhar.edu.eg

### KEYWORDS

Self-etch, total etch,  
nanofilled resin composite,  
Nanoleakage, SEM,  
G-aenial bond.parameters

1. Department of Operative Dentistry, Faculty of Dental Medicine (Cairo-Boys), Al-Azhar University, Cairo, Egypt.
2. Department of Dental Biomaterials, Faculty of Dental Medicine (Assuit), Al-Azhar University, Assuit, Egypt
3. Department of Crown and bridge, Faculty of Dental Medicine (Cairo-Boys), Al-Azhar University, Cairo, Egypt.
4. Department of Endodontic, Faculty of Dental Medicine (Cairo-Boys), Al-Azhar University, Cairo, Egypt
5. Department of Operative Dentistry, Faculty of Dentistry, Nahada University, Beni-sueif, Egypt.

\* Corresponding Author e-mail:  
drwakwak2006@azhar.edu.eg

### ABSTRACT

**Aim:** The goal of this study was to assess the nanoleakage of nano-filled resin composites utilizing various adhesive systems at varying dentin depths. **Subjects and methods:** In this investigation, 120 freshly removed human molars were tested. The chosen teeth were randomly divided into two main groups (n=60), with each main group being divided into two smaller groups (n=30) based on the adhesive system (etch-&rinse mode and self-etch mode of application). According to the superficial and deep dentin layers used in the adhesion process, each small group was separated into two even smaller subgroups (n = 15). Each smaller group was divided into three different categories depending on the lengths of storage (24 hours, 3 months, and 6 months) (n = 5). Each tooth was gradually reconstructed using the nanofilled resin composite. The specimens were cut longitudinally in the mesio-distal and bucco-lingual orientations to create resin-dentin beams. For the purpose of assessing nanoleakage, the beam was submerged in silver nitrate. **Results:** There was no statistically significant difference between groups in terms of the effect of application mode or dentin depth on nanoleakage, however there was a statistically significant increase in median nano-leakage scores from 24 hours to 3 months and 3 to 6 months of storage duration. **Conclusion:** The use of both bonding chemicals helps to reduce resin composite leakage. Since the self-etch adhesive system excelled total etch in deep cavities, it is advised to use it. While the total etch adhesive method, is advised for use in superficial cavities.

### INTRODUCTION

Resin composites were first used as aesthetic materials for anterior fillings, but their application rapidly progressed to posterior teeth. <sup>1</sup> Composite restorations still have several problems in spite of the advancements in composite resins and the improvement of adhesive techniques. <sup>2</sup> One of the key disadvantages is polymerization shrinkage, which causes polymerization stress and, as a result, de-bonding between tooth structure and resin composite, reducing the longevity of the restoration. <sup>3,4</sup> By using nanotechnology, nanocomposites have been developed to meet these functional requirements. Better

compressive and tensile strength, fracture and wear resistance, minimal polymerization shrinkage, high translucency and polish retention, and better aesthetics are among their improved mechanical qualities.<sup>5</sup> A resin composite that incorporates nanomers and nanoclusters may be able to use nanocomposites, which exhibit improved fracture toughness and adherence to tooth structure and have reduced significantly filler particle sizes.<sup>6</sup> Nanoclusters are agglomerates of primary zirconia/silica nanoparticles (5 to 20 nm in size), which are fused together at points of contact and have a particle size range of 5 to 75 nm. The resulting porous structure is filled with silane.<sup>7</sup> Today, restorative dentistry typically employs two adhesive approaches. The first method, known as “etch and rinse,” necessitates careful rinsing after phosphoric acid etching, whereas the second method, known as “self-etch,” (SE) does not use phosphoric acid and instead requires for the use of mild acidic primers for etching and chemical bonding to hydroxyapatite.<sup>8</sup> When there is no marginal micro-gap around the filling and the composite resin has sufficiently penetrate deeply and adapt well to dental tissue. However Polymerization shrinkage during composite resin hardening results in the creation of micro gaps surrounding composite fills.<sup>9</sup>

In the absence of marginal gaps, nanoleakage refers to leaking along channels with a diameter of a few nanometers. This can happen within the hybrid layer or in the adhesive layer. This phenomenon has received a lot of attention as a significant contributor to the breakdown of the bonding to dentinal tissue.<sup>10</sup> Inadequate resin penetration into the demineralized collagen network or inadequate polymerization of hydrophilic monomers in the submicron interfacial gaps could be the cause. The resulting exposed collagen fibrils may be susceptible to breakdown by oral microbial enzymes. Interfacial sealing, bond strength, and synthetic ageing appear to be linked in vitro, and they can be considered possible clinical predictors of restoration effectiveness.<sup>11</sup> Since the dentine surface is a heterogeneous vital

structure with a low surface energy and dentinal fluid leak might occur onto the prepared surface, dentin bonding is more complicated. Although some modern dentin bonding techniques were able to achieve gap-free margins at the dentin/restoration contact. It was discovered that even in the absence of gap formation between the filling and tooth structure, nanoleakage along the dentinal wall was still detectable.<sup>12</sup> As the ultimate goal of bonded restorations is to produce a marginal and internal seal, hybridization is a critical event in bonding resin composite to dentin.<sup>13</sup> When small molecules or ions invade the hybrid layer, this is considered to as nanoleakage. A number of innovative adhesive solutions have been developed in an effort to achieve a long-term adhesive-restoration interface. Current adhesive methods interact with the tooth substrate in two ways. The total-etch process requires the smear layer to be removed. Incomplete collagen expansion, on the other hand, may impair resin entry and jeopardize bonding with those adhesives.<sup>8,14</sup>

The mechanical, physical, adhesive, and handling characteristics of the various resin composites and adhesive systems, as well as other considerations, all have an impact on how long restorations last. The patient, socioeconomic circumstances, the oral environment, which includes the placement and size of the restoration, the risk of caries, and behaviors like bruxism all have an impact on how long restorations function. A significant factor is the clinician, who decides whether to restore the tooth or replace a restoration, chooses the material, and performs the treatment. The generalizability of past study results is thus difficult from the standpoint of dental materials. The quantity of penetration is determined by the type of bonding agent and other application technique characteristics (e.g. etching time, dentin moisture)<sup>15,16</sup>.

Nanoleakage increases with long-term storage. Although there is no convincing evidence of nanoleakage deleterious impacts, the presence of such a channel in gap-free cavity edges may have long-term implications for adhesion quality.<sup>17</sup> As



a result, the current investigation was carried out to evaluate the nanoleakage of nano-filled resin composites (NFCR) using various adhesive methods at various dentin depths.

## MATERIALS AND METHODS

Two adhesive systems and one type of resin composite were used in the current study as shown in Table I.

### Resin composite material:

1- Filtek Z350XT Universal Restorative (3M ESPE), shade A3 body: A visible light-cured methacrylate-based nano-filled resin composite

### Adhesive bonding systems:

1. Universal single bond adhesive system (3M ESPE): A single component, light curing adhesive utilized with its Scotchbond™ Universal Etchant.

2. G-bond plus (Gaenial bond); (GC, Tokyo, Japan), one step SE adhesive.

### Teeth selection

In this investigation, 120 newly extracted human molars ranging in age from 25 to 40 years were employed. The extracted teeth were obtained from the oral and maxillofacial surgery department's outpatient clinic for periodontal or orthodontic reasons. To be used in the investigation, only healthy teeth were chosen. Carious, broken, and decaying teeth, as well as any other deformities or developmental defects, were also excluded from the study.

### Grouping of the specimens:

According to adhesive system, the chosen teeth were randomly divided into two major groups (n=60): **Group 1:** Cavities were filled with Z350XT and bonded with a universal adhesive technique from Scotchbond.

**Table (I)** *Materials used in the present study*

Material	Composition	Manufacture, website and Batch number
Filtek™ Z350 XT Universal Restorative shade A3	<b>Resin matrix:-</b> Bis-GMA* -BisEMA**-UDMA*** -TEGDMA****-PEGDMA***** <b>Filler:-</b> combination of non-aggregated 20 nm silica-non-aggregated 4-11nm zirconia/silica cluster.(primary particle 5-20 nm 78.5% by weight(63.3% by volum)	3M ESPE, St.Paul,MN U.S.A. www.3mespe.com N932955
3M™ Single bond™ Universal Adhesive	MDP*****-Phosphate Monomer -Dimethacrylates -Vitrebond™ Copolymer Filler -Ethanol -Water -Initiators. -Silane	3M ESPE, St.Paul,MN U.S.A. www.3mespe.com 692513
<b>G-BOND</b> plus (G-aenial)	Acetone,dimethacrylate,4methacryloxyethyltrimellitate anhydride, phosphoric acid ester monomer, silicon dioxide, photo initiator, distilled water Note: The manufacturer does not recommend dentin conditioning with phosphoric acid	GC Corporation Tokyo, Japan, N1510051
Scotchbond™ Universal Etchant	34% phosphoric etchant gel	3M ESPE, St.Paul,MN 55144-1000 U.S.A.www.3mespe.comD-82229

**Group 2:** The Z350XT bonded Genial bond plus adhesive technology was used to restore the cavities.

An application mode was used to divide each main group into two smaller groups (n=30). In Group I, the adhesive system was applied using the etch-and-rinse method, whereas in Group II, the adhesive system was applied using the SE method. According to the dentin layer used for specimen preparation, each small group was divided into two smaller sub-groups (n=15). Group A composite resin adhered to the most superficial dentin layer, while Group B composite resin was adhered to the deepest one. According to storage times, each smaller subgroup was divided into three smaller classes (n=5). Class 1 storage periods were one day, Class 2 storage periods were three months, and Class 3 storage periods were six months. All materials used in this investigation were handled in accordance with the manufacturer guidelines. Until the completion of the storage periods, all specimens were preserved in distilled water at 37°C in an incubator. Every three days, the water was changed.

### Specimen preparation

After wet-grinding the occlusal enamel with a slow-speed water-cooled diamond disc (Isomet, Buehler Ltd., Lake Bluff, IL, USA), a smooth occlusal dentin surface was exposed in all teeth. To standardize the smear layer at the superficial dentin layer, the exposed dentin surfaces were further polished for 60 seconds with wet #600-grit silicon-carbide paper. Further polishing with wet #600-grit silicon-carbide paper for additional 60 seconds was performed to standardize the deepest layer of dentin.

After making a composite resin crown build up, the specimens were sectioned longitudinally in mesio-distal and bucco-lingual directions over the bonded interface with a slow-speed diamond saw to obtain resin-dentin beams with a digital caliper (Digimatic Caliper, Mitutoyo, Tokyo, Japan) measuring its cross sectional area, which was around 0.8 mm<sup>2</sup>.

### Adhesive and resin composite application

The first group (Group I) consists of (etch-&rinse application mode). Using the Single bond universal, the tooth substrate was etched for 30 seconds. For 10 seconds, the etching gel was completely washed with water. Air dried gently to eliminate excess water without over-drying, preserving the moist condition of the dentin.

The adhesive system was applied to the occlusal table in the second group (Group II) (SE application mode) using a disposable applicator and rubbing motion for 20 seconds. After gently air drying for 5 seconds, the excessive solvent was removed, ensuring that the adhesive layer did not migrate and that the surface maintained its uniformly glossy appearance. The adhesive was then cured using an LED curing device in standard mode for 20 seconds as per the manufacturer's instructions, at a light intensity of 1200 mW/cm<sup>2</sup> (3M ESPE Elipar™ S10 U.S.A.).

### Restoration technique

Using the nano-filled resin composite (NFCR) (Filtek™ Z350 XT Universal Restorative), all teeth were sequentially repaired (3M ESPE). Each increment was cured for 40 seconds in accordance with the manufacturer's specifications, using a gold-plated condenser to prevent the composite from attaching to the instrument.

### Storage of teeth

Teeth were stored at 37°C in distilled water which was changed every 24 hours. Each jar was labeled according to variables of the study.

### Testing procedures

For Nanoleakage assessment: the resin-dentin beams were placed in an ammonical silver nitrate solution in darkness for 24 h, rinsed thoroughly in distilled water, and immersed in photo developing solution for 8 h under a fluorescent light to reduce silver ions into metallic silver grains. Specimens



were polished with wet 600 grit Sic paper. Resin-dentin interface were analyzed with a scanning electron microscope (Philips, XL 30, Eindhoven, The Netherlands). The micrograph was taken in the center of the beam. The mean NL (%) of all beams from the same tooth was averaged for statistical purposes. Comparison between the twelve different subgroups was made using four-dimensional mapping which was performed over 100 mm x 100 mm areas across the resin-dentine bonded interface, these areas covered the adhesive layer. The HL (hybrid), partially demineralized and un-affected dentine was visualized and focused at 2500 x magnification.

### Statistical Analysis

Non-parametric data were used to analyze nano-leakage scores. The data were presented in the form of mean, and standard deviation values. Mann-Whitney The U test was performed to compare the two adhesive systems and the two margins. To compare superficial and deep dentin, the Wilcoxon signed-rank test was performed. To compare the three time interval periods, the Kruskal-Wallis test was utilized. When the Kruskal-Wallis test proved significant, Dunn's test was employed for pair-wise comparisons. P 0.05 was used as the significant level. IBM SPSS Statistics for Windows, Version 23.0 (IBM Corp., Armonk, New York), was used for statistical analysis.

## RESULTS

### Effect of application mode

Table II shows the average leakage score values and ranges for resin composites bonded to superficial or deep dentin using SE or total etching across various storage intervals (24 hours, 3 months, 6 months). Nanoleakage results are compared using the Mann-Whitney U test.

Our research showed that samples of resin composite bonded to superficial dentin using a SE adhesive system had the highest mean leakage

value ( $2.6 \pm 0.89$  SD) at 6 months of storage but the lowest mean leakage value ( $0.4 \pm 0.55$ ) recorded from resin composite samples bonded to superficial dentin at 24 hours of storage. The leakage of resin composites bonded with total etch bonding agent, however, varied from the lowest mean value ( $0.4 \pm 0.55$ ) for samples of superficial dentin at 24 hours to the highest leakage value ( $2.4 \pm 1.14$ ) which was recorded for both deep dentin and superficial dentin at 6 months of storage time.

**Table (II)** *The effect of adhesive systems on nanoleakage.*

Adhesive system	Dentin depth	24 Hours	3-Months	6-Months
Self-etch	Superficial dentin mean $\pm$ SD	0.4 (0.55)	1.2 (0.45)	2.6 (0.89)
	Deep dentin mean $\pm$ SD	0.6 (0.55)	1.4 (0.55)	2 (0.71)
Total-etch	Superficial dentin mean $\pm$ SD	0.4 (0.55)	0.6 (0.55)	2.4 (0.55)
	Deep dentin mean $\pm$ SD	0.8 (0.45)	1.6 (0.55)	2.4 (1.14)

### Effect of dentin depth on nanoleakage

Table III shows the mean values and standard deviations for the leakage scores of resin composites bonded to shallow or deep dentin using SEing or total-etching techniques throughout various storage intervals (24 hours, 3 months, and 6 months). Use of the Wilcoxon signed-rank test for comparison of nano-leakage scores

In our study, resin composite samples bonded by total etch in superficial dentin recorded the highest leakage score with a mean value ( $2.6 \pm 0.89$ ) at 6 months, while resin composite samples bonded by SE or total etch in superficial dentin at 24 hours storage time revealed that there is no statistically significant difference in leakage mean value as they both scored leakage value with a standard deviation ( $0.4 \pm 0.55$ ). In contrast, samples of resin composite bonded to deep dentin using a total etch adhesive system at six months scored the highest

mean value for leakage ( $2.4 \pm 1.14$ ), while samples of resin composite bonded to deep dentin using a SE adhesive system at 24 hours storage time showed the least leakage value ( $0.6 \pm 0.55$ ).

**Table (III)** *The effect of dentin depth on nanoleakage*

Dentin depth	Adhesive system	24 hours	3-months	6-months
Superficial dentin	Self-etch mean $\pm$ SD	0.4 (0.55)	1.2 (0.45)	2.6 (0.89)
	Total-etch mean $\pm$ SD	0.4 (0.55)	0.6 (0.55)	2.4 (0.55)
Deep dentin	Self-etch mean $\pm$ SD	0.6 (0.55)	1.4 (0.55)	2 (0.71)
	Total-etch mean $\pm$ SD	0.8 (0.45)	1.6 (0.55)	2.4 (1.14)

#### Effect of storage times on nanoleakage

Table IV shows the mean values and standard deviations of the leakage scores of resin composites bonded at various storage times (24 hours, 3 months, and 6 months) using either SEing or total etching at superficial or deep dentin. Use of the Kruskal-Wallis test to compare nano-leakage scores.

According to our research, samples of resin composites bonded at 24 hours storage time using SE or total etch in superficial dentin recorded the lowest leakage mean value ( $0.4 \pm 0.55$ ), while samples of resin composites bonded by total etch adhesive system in deep dentin recorded the highest leakage mean value with a score of ( $0.8 \pm 0.45$ ). samples of resin composite bonded by total etch in superficial dentin recorded the lowest leakage value ( $0.6 \pm 0.55$ ) for a period of three months of storage, while samples bonded by total etch in deep dentin scored the greatest leakage value ( $1.6 \pm 0.55$ ), and after six months of storage, samples of resin composite bonded by SE adhesive system at deep dentin had the lowest mean leakage value ( $2 \pm 0.71$ ) while samples of resin composite bonded by SE adhesive system in superficial dentin had the greatest mean leakage value ( $2.6 \pm 0.89$ ).

**Table (IV)** *The effect of storage times on nanoleakage*

Storage time	Self-etch		Total etch	
	Superficial Dentin	Deep Dentin	Superficial Dentin	Deep Dentin
24 hours mean $\pm$ SD	0.4 (0.55)	0.6 (0.55)	0.4 (0.55)	0.8 (0.45)
3 months mean $\pm$ SD	1.2 (0.45)	1.4 (0.55)	0.6 (0.55)	1.6 (0.55)
6 months mean $\pm$ SD	2.6 (0.89)	2 (0.71)	2.4 (0.55)	2.4 (1.14)

#### Scanning electron microscope study

Leakage score of resin composites attached to superficial or deep dentin using either a SE or total etching bonding agent was assessed using a scanning electron microscope in high vacuum mode at a magnification of X2.500 (Figure .1, 2).

#### The effect of 24 hours of storage time on nanoleakage

SE group at superficial dentin is seen in a SEM photomicrograph as a hybrid layer with resin tags impregnated into dentinal tubules and nanoleakage near the tubules. Silver nitrate particles on resin tags in the entire etch group indicate nanoleakage. Deep dentin has greater nanoleakage, whereas superficial dentin exhibits less nanoleakage.

#### Effect of three months of storage time on nanoleakage

More nanoleakage is shown by the silver nitrate particles in the SE group that are scattered across the superficial and deep dentin. SEM specimens from deep dentin demonstrate only slight nanoleakage.

#### Effect of six months of storage time on nanoleakage

SEM A photomicrograph of a SE group demonstrating nano-gaps between dentinal tubules and resin tags at (6) months of superficial dentin and deep dentin and demonstrates open dentinal tubules.



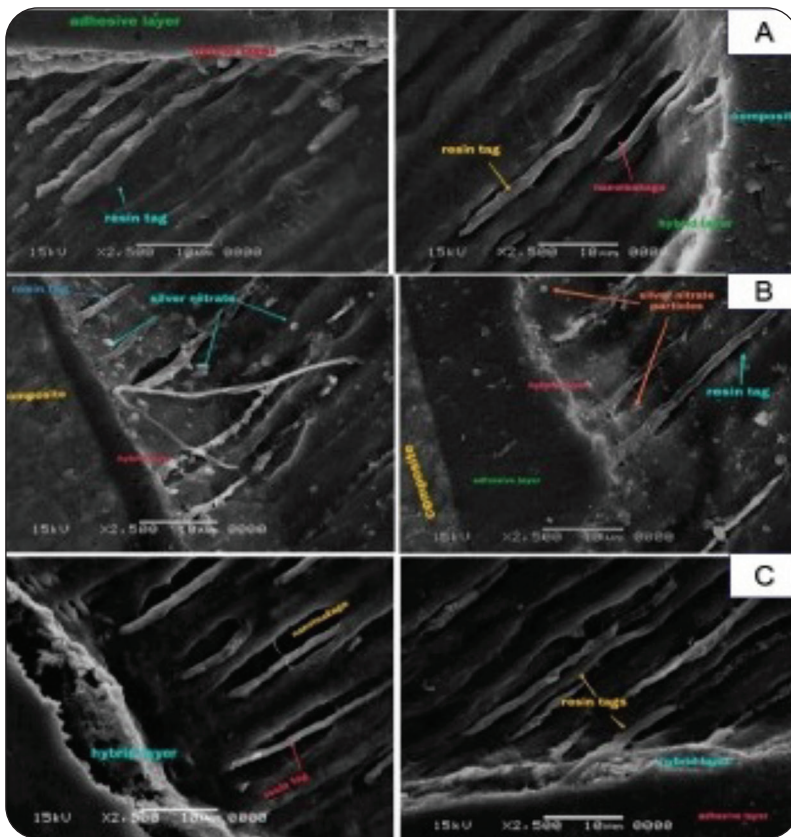


Fig. (1) A) Left: A self-etch group at superficial dentin with a hybrid layer and resin tags at a time of (24 hours) is represented in the SEM photomicrograph. A) Right. SEM photomicrograph at (24) hours exhibiting scattered silver nitrate particles at the deep dentin interface. B. Left, demonstrating resin tags' uptake of silver nitrate at three months. B) Right. Self-etch group at deep dentin hybrid layer with resin tags at (3) months. C) Left. A. demonstrating nanogaps between resin tags and dentinal tubules at six months C) Right. At six months, indicating resin tag uptake of silver nitrate.

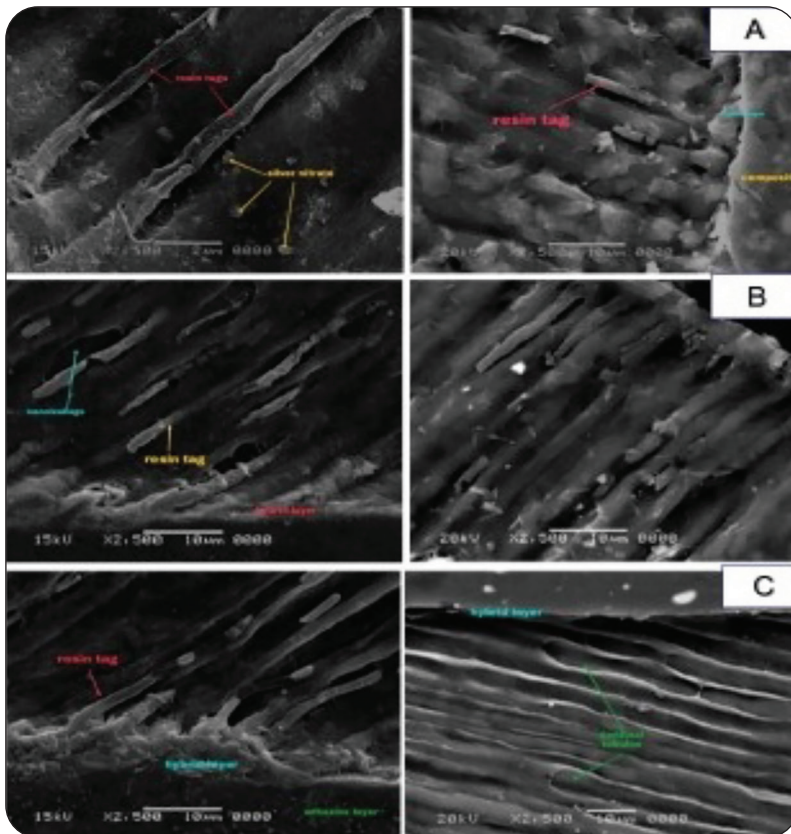


Fig. (2) A) Left: SEM photomicrograph of the total etch group at the superficial dentin, showing the hybrid layer and resin tags after 24 hours. A. Right: SEM photomicrograph of the total etch group at deep dentin, showing the hybrid layer and resin tags after (24) hours. B) Left: SEM photomicrograph of total etch group at superficial dentin demonstrating nanoleakage along resin tags after three months. B) Right: SEM photomicrograph of total etch group at deep dentin showing hybrid layer and resin tags after three months. C) Left: A. SEM photomicrograph of the total etch group at the superficial dentin, showing the hybrid layer and resin tags after (6) months. C) Right: SEM photomicrograph of the total etch group at deep dentin showing resin penetration in dentin canals after (6) months.

## DISCUSSION

The phenomenon of nanoleakage was initially documented by Sano et al. (1994), who investigated the transport of ions or molecules within the hybrid layer's nanometric gaps<sup>18</sup>. Our results revealed that the highest leakage mean value score was for resin composite samples bonded to superficial dentin using SE adhesive system at 6 months storage time, which was confirmed by our SEM study which indicated leakage spaces at hybrid layer interface as this because it contains an increased concentration of water, acidic monomer, and hydroxyethylmethacrylate, which makes these polymers very hydrophilic and likely to absorb.<sup>19</sup>.

This could be explained by the fact that the hybrid layer is fairly porous and accessible to the silver dye even in the absence of a marginal gap, and that the leakage is caused via penetration channels through the network of interfibrillar spaces with a size of only a few nanometers or less. Since SE materials have higher pH values than those used with total-etch adhesive systems and are not rinsed away, the smear layer or its components are incorporated into the bonded layer, meaning that the SE adhesive is not completely removing the smear layer or opening all the tubules.<sup>20</sup>. Additionally, because the SE adhesive systems only change the smear layer, the presence of remaining water may prevent the adhesive from fully polymerizing, lowering the quality of the resin-dentin connection.<sup>21</sup>.

This result was in accordance with the findings of Hashimoto M.(2011)<sup>4</sup>, who used transmission electron microscopy (TEM) to study nanoleakage of adhesive systems. He reported that while some nanoleakage was seen in all of the groups tested, it was more noticeable for the SE adhesives when compared to the total-etch adhesives.

A visible light cured nanofilled resin composite (NFCR) was used in our study because it has a filler cluster that forms nanoclusters in a wide range of sizes, enabling a high filler loading and, consequently, high strength and wear resistance.

Filler technology may have a significant impact on how well the dental composite performs<sup>22-25</sup>. Because of its exceptionally small dimension molecule, ammoniacal silver nitrate solution [Ag(NH<sub>3</sub>)<sub>2</sub>]<sup>+</sup>+ 50% (wt/vol) (pH = 9.5) was employed in this investigation because it is the most often utilized material for nanoleakage evaluation. Because of its small size(0.059 nm) and great reactivity to stain after tightly adhering to any exposed collagen fibrils that are not surrounded by the adhesive resin, silver nitrate is the best agent for detecting nanoporosities within the hybrid layer.<sup>26-28</sup>

Our SEM investigation verified the SEM finding that the complete etched dentin surfaces were constantly coated by the adhesive and sent very fine processes into the anastomosing tubules. This might be because different adhesive systems have unique properties that affect how much of the smear layer is removed, how much of the underlying dentin is demineralized, and how well the adhesive can penetrate and moisten the dentin.<sup>29</sup>. Due to varying penetration capabilities, differences in resin monomer composition, or solvent, the difference in nanoleakage appeared to be influenced by the dentin adhesive systems used. SE adhesives are more hydrophilic than total-etch adhesive solutions, more permeable to water derived from dentin, and thus more vulnerable to the deterioration of resin-dentin bonds.<sup>30</sup>. This was also in agreement with Ulker M et al. (2013)<sup>31</sup>, who discovered that using the field emission-SEM(FE-SEM), traditional total-etch adhesive solutions tended to exhibit less nanoleakage than adhesives.

Results do not support Zidan A.(2019)<sup>32</sup> conclusion's that total-etch adhesives resulted in the least penetration of silver nitrate within the hybrid layer using FE-SEM and TEM because they have the potential to form a hybrid layer and seal dentin while collagen fibrils are completely deprived of hydroxyapatite. Instead, SE adhesive systems have these potentials as well as the potential to form a hybrid layer and seal dentin. Additionally, the findings did not agree with those of Souza MY et al (2020)<sup>33</sup>,





who examined the impact of the ethanol-wet-bonding technique on dentin adhesive infiltration using nanohardness, elastic modulus, and nanoleakage and came to the conclusion that the use of universal adhesives is an alternative to achieve a stable bond and may have clinical applications.

As SE adhesive systems condition and simultaneously leak into the underlying substrate, a discrepancy between demineralization and resin infiltration depths may occur<sup>34</sup>. This result was also confirmed by our SEM study which revealed leakage around resin tags interface. Several publications demonstrate that nanoleakage in SE adhesive surfaces is not solely caused by resin leakage in demineralized dentin.<sup>35</sup> Other reported causes include hydrophilic regions of acidic monomers that are more susceptible to water caption, occasionally, in both etch-and-rinse and SE adhesive systems, channels filled with water form in the adhesive layer—water trees—and these are manifested by water bubbles in the resin-dentin interface.<sup>16,36</sup> This process, which is caused by the gradual extraction of nonpolymerized monomers or tiny oligomers over time, might directly contribute to resin degradation.<sup>37–40</sup>

According to Arora A et al(2019)<sup>41</sup> study Effect of Beveling of Enamel on Microleakage and Shear Bond Strength of Adhesive Systems in Primary and Permanent Teeth, SE adhesive outperformed total-etch adhesive because water-based dentin bonding systems demonstrated less microleakage in dentin margins than acetone-based, water-free dentin bonding systems. This was also consistent with Ferrerira JC et al findings from 2017<sup>42</sup>, which showed that SE systems had good morphology, a high-quality hybrid layer, and low nanoleakage percentages after examining their effects on dentin's etch-and-rinse and SE adhesive behavior. These outcomes seem to be related to coating the hydrophilic primer with a hydrophobic resin, and it seems that the solvent has a big impact on morphology and nanoleakage.

In contrast to Zhuge RS. et al findings from 2017<sup>43</sup>, which examined the effects of subpressure on the bond properties of total-etching adhesive to dentin and came to the conclusion that subpressure provided a reliable interfacial morphology, enhanced the short- and long-term bonding strength, and decreased nanoleakage, which had a positive impact on the total-etching dentin bonding and increased bond durability.

Statistical study revealed that the two adhesive systems have no significant nanoleakage. In terms of adhesive systems, the SE adhesive system (single bond universal) exceeded Genial-Bond plus (GBP). Bonding penetration most likely occurred at the same depth as demineralization using acidic monomers, removing the chance of inadequate impregnation with the bonding resin and ensuring minimal sealing. As in earlier research, total etch (Single Bond plus) systems demonstrated greater microleakage than SEing (Single Bond universal) systems, perhaps due to incomplete monomer penetration in the demineralized dentin zone achieved after phosphoric acid etching.<sup>44</sup>

Once 10-MDP molecular structures have hydrophilic functional groups that are vulnerable to cycling environments, the SE adhesive system degrades with water degradation<sup>19</sup>. These data support the findings of this investigation, which found that baseline levels of baseline nanohardness and elastic modulus were higher than baseline values for 6 months regardless. Since the sorption of water reduces the bonding forces between the polymer chains during polymerization, ageing was able to lower the nanohardness and elastic modulus values, which may suggest degradation by hydrolysis and lower the mechanical properties of the adhesive interface.<sup>45</sup> Additionally, SE adhesives may function as semi-permeable membranes even after polymerization, making them more prone to ageing.<sup>46</sup>

The fact that the bonding mechanism of total etch (Single Bond plus) is predicated on the

complete removal of the smear layer after etching with phosphoric acid, leaving an exposed collagen network suitable for primer impregnation, can also be used to explain why total etch results in more leakage. To avoid the collagen network from collapsing, primer application must be done after a small drying period. Dentin's marginal integrity could be compromised as a result of the challenges in determining the optimal amount of moisture to be left in it, leading to greater amounts of microleakage<sup>47</sup> as this outcome was supported by our SEM analysis.

This finding agreed with the findings of Atash Biz et al(2015)<sup>48</sup>, who conclusively proved that the SEing adhesive system had significantly lower microleakage values at the gingival margin than the Single Bond one-bottle system, either in the immediate assessment (24 hours) or after 3 months of water storage. Also The findings of this study also showed that SE recorded less nanoleakage than total etch at 6 months. This may be because the active monomers' acidic properties are causing the smear layer to dissolve and demineralize the dentin underneath. Because the mineral content of the dentin eventually buffers the acidity of the monomers, this demineralization is self-limiting.<sup>49</sup>

This finding was consistent with that of Kaczor K et al. (2018)<sup>50</sup>, who assessed how etching modes affect the nanoleakage of universal adhesive from in vitro studies and drawn the conclusion that All-Bond Universal's SE mode reduced nanoleakage. Seven universal adhesives were assessed in his review. Although El Sayed HY et.al (2014)<sup>51</sup>, who investigated marginal microleakage of composite resin restorations bonded by desensitising one step SE adhesive, found different results. He demonstrated that at both the enamel and dentin edges. Etch and rinse adhesive single bond2 recorded much lower scores of dye penetration than the SE adhesive I-bond.

## CONCLUSIONS

The reduction of resin composite leakage is facilitated by the employment of total etch or SE bonding agents. In order to reduce resin composite leakage in deep dentin, it is advised to employ self-etching bonding agents rather than total etching agents. The process by which resin composite leaks into tooth structure is time-dependent.

## REFERENCES

1. Chan KHS., Mai Y., Kim H., Tong KCT., Ng D., Hsiao JCM. Review: Resin composite filling. *Materials (Basel)* 2010;1228–43. Doi: 10.3390/ma3021228.
2. Cangul S., Adiguzel O. The Latest Developments Related to Composite Resins. *Int Dent Res* 2017;7(2):32. Doi: 10.5577/intdentres.2017.vol7.no2.3.
3. Soares CJ., Faria-E-Silva AL., Rodrigues M de P., Vilela ABF., Pfeifer CS., Tantbirojn D., et al. Polymerization shrinkage stress of composite resins and resin cements - What do we need to know? *Braz Oral Res* 2017;31(suppl 1):e62. Doi: 10.1590/1807-3107BOR-2017.vol31.0062.
4. Hashimoto M., Nagano F., Endo K., Ohno H. A review: Biodegradation of resin-dentin bonds. *Jpn Dent Sci Rev* 2011;47(1):5–12. Doi: 10.1016/j.jdsr.2010.02.001.
5. Hegde MN., Hegde P., Bhandary S., Deepika K. An evaluation of compressive strength of newer nanocomposite: An in vitro study. *J Conserv Dent* 2011;14(1):36–9. Doi: 10.4103/0972-0707.80734.
6. Lyapina M., Cekova M., Krasteva A., Dencheva M., Yaneva-Deliverska M., Kisselova A. Physical Properties of Nanocomposites in Relation To Their Advantages. *JIMAB - Annu Proceeding (Scientific Pap* 2016;22(1):1056–62. Doi: 10.5272/jimab.2016221.1056.
7. Rodríguez HA., Casanova H. Effects of Silica Nanoparticles and Silica-Zirconia Nanoclusters on Tribological Properties of Dental Resin Composites. *J Nanotechnol* 2018;2018. Doi: 10.1155/2018/7589051.
8. Giannini M., Makishi P., Ayres APA., Vermelho PM., Fronza BM., Nikaido T., et al. Self-etch adhesive systems: a literature review. *Braz Dent J* 2015;26(1):3–10. Doi: 10.1590/0103-6440201302442.
9. Al-Zain AO., Eckert GJ., Lukic H., Megremis S., Platt JA. Polymerization pattern characterization within a resin-based composite cured using different curing units at



- two distances. *Clin Oral Investig* 2019;**23**(11):3995–4010. Doi: 10.1007/s00784-019-02831-1.
10. Breschi L., Maravic T., Cunha SR., Comba A., Cadenaro M., Tjäderhane L., et al. Dentin bonding systems: From dentin collagen structure to bond preservation and clinical applications. *Dent Mater* 2018;**34**(1):78–96. Doi: 10.1016/j.dental.2017.11.005.
  11. Manuja N., Nagpal R., Pandit IK. Dental adhesionnnnn. *J Clin Pediatr Dent* 2012;**36**(3):223–34. Doi: 10.17796/jcpd.36.3.68805r1r037m063.
  12. Kumar JS., Jayalakshmi S. Bond failure and its prevention in composite restoration – A Review. *J Pharm Sci Res* 2016;**8**(7):627–31.
  13. Spencer P., Ye Q., Park J., Topp EM., Misra A., Marangos O., et al. Adhesive/Dentin interface: the weak link in the composite restoration. *Ann Biomed Eng* 2010;**38**(6):1989–2003. Doi: 10.1007/s10439-010-9969-6.
  14. Yuan Y., Shimada Y., Ichinose S., Tagami J. Qualitative analysis of adhesive interface nanoleakage using FE-SEM/EDS. *Dent Mater* 2007;**23**(5):561–9. Doi: 10.1016/j.dental.2006.03.015.
  15. Ástvaldsdóttir Á., Dagerhamn J., van Dijken JW V., Naimi-Akbar A., Sandborgh-Englund G., Tranæus S., et al. Longevity of posterior resin composite restorations in adults – A systematic review. *J Dent* 2015;**43**(8):934–54. Doi: 10.1016/j.jdent.2015.05.001.
  16. Cardoso M V., de Almeida Neves A., Mine A., Coutinho E., Van Landuyt K., De Munck J., et al. Current aspects on bonding effectiveness and stability in adhesive dentistry. *Aust Dent J* 2011;**56 Suppl 1**:31–44. Doi: 10.1111/j.1834-7819.2011.01294.x.
  17. Ayad N., Abdelaziz K. Interface Characterization and Nanoleakage of One-step Self-etch Adhesive Systems. *Internet J Dent Sci* 2012;**5**(1):1–7. Doi: 10.5580/1b25.
  18. Pinna R., Maioli M., Eramo S., Mura I., Milia E. Carious affected dentine: its behaviour in adhesive bonding. *Aust Dent J* 2015;**60**(3):276–93. Doi: 10.1111/adj.12309.
  19. Zhou W., Liu S., Zhou X., Hannig M., Rupf S., Feng J., et al. Modifying Adhesive Materials to Improve the Longevity of Resinous Restorations. *Int J Mol Sci* 2019;**20**(3). Doi: 10.3390/ijms20030723.
  20. Reham M Mohamd HYES. Marginal Seal of Water-Based Formulation of Light Activated Bonding Agent for Use in Combination with Adhesive Restorations. *Dentistry* 2015;**5**(4). Doi: 10.4172/2161-1122.1000291.
  21. Thanatvarakorn O., Prasansuttiporn T., Thittaweerat S., Foxton RM., Ichinose S., Tagami J., et al. Smear layer-deproteinizing improves bonding of one-step self-etch adhesives to dentin. *Dent Mater* 2018;**34**(3):434–41. Doi: 10.1016/j.dental.2017.11.023.
  22. Alencar MF., Pereira MT., De-Moraes MDR., Santiago SL., Passos VF. The effects of intrinsic and extrinsic acids on nanofilled and bulk fill resin composites: Roughness, surface hardness, and scanning electron microscopy analysis. *Microsc Res Tech* 2020;**83**(2):202–7. Doi: 10.1002/jemt.23403.
  23. Perdigão J., Geraldeli S., Hodges JS. Total-etch versus self-etch adhesive: effect on postoperative sensitivity. *J Am Dent Assoc* 2003;**134**(12):1621–9. Doi: 10.14219/jada.archive.2003.0109.
  24. Sahay R., Reddy VJ., Ramakrishna S. Synthesis and applications of multifunctional composite nanomaterials. *Int J Mech Mater Eng* 2014;**9**(1):1–13. Doi: 10.1186/s40712-014-0025-4.
  25. Aydın F., Demirel G., Bilecenoğlu B., Ocak M., Gür G. Effect of different artificial aging protocols on marginal sealing ability of self-etch dental adhesives: micro-computed tomography evaluation. *J Adhes Sci Technol* 2020;**34**(4):388–99. Doi: 10.1080/01694243.2019.1670581.
  26. El-Badrawy W., Hafez RM., El Naga AIA., Ahmed DR. Nanoleakage for Self-Adhesive Resin Cements used in Bonding CAD/CAD Ceramic Material to Dentin. *Eur J Dent* 2011;**5**(3):281–90.
  27. Amin RA., Mandour MH., Abd El-Ghany OS. Fracture strength and nanoleakage of weakened roots reconstructed using relined glass fiber-reinforced dowels combined with a novel prefabricated core system. *J Prosthodont Off J Am Coll Prosthodont* 2014;**23**(6):484–94. Doi: 10.1111/jopr.12139.
  28. Al-Nabulsi M., Daud A., Yiu C., Omar H., Sauro S., Fawzy A., et al. Co-Blend Application Mode of Bulk Fill Composite Resin. *Mater (Basel, Switzerland)* 2019;**12**(16). Doi: 10.3390/ma12162504.
  29. Ahmeda AA., Mustafa M. Hassanb AIA. Microshear bond strength of universal adhesives to dentin used in total-etch and self-etch modes. *Tanta Dent J* 2018;**15**:91–8. Doi: 10.4103/tjdtj.
  30. Tjäderhane L. Dentin bonding: Can we make it last? *Oper Dent* 2015;**40**(1):4–18. Doi: 10.2341/14-095-BL.
  31. Ulker M., Ulker HE., Karabekiroglu S., Botsali MS., Cetin AR. Effect of alternative modes of application on

- microleakage of one-step self-etch adhesives. *J Dent Sci* 2013;**8**(4):425–31. Doi: 10.1016/j.jds.2012.09.017.
32. Zidan A. Effect of chitosan on resin-dentin interface durability: A 2 year in-vitro study. *Egypt Dent J* 2019;**65**(3):2955–65. Doi: 10.21608/edj.2019.72691.
  33. Souza MY., Andrade JL., Ferraz Caneppele TM., Bresciani E. Assessment of nanohardness, elastic modulus, and nanoleakage of the adhesive interface using the ethanol-wet-bonding technique. *Int J Adhes Adhes* 2020;**99**:102572. Doi: <https://doi.org/10.1016/j.jadhadh.2020.102572>.
  34. Miyazaki M., Tsubota K., Takamizawa T., Kurokawa H., Rikuta A., Ando S. Factors affecting the in vitro performance of dentin-bonding systems. *Jpn Dent Sci Rev* 2012;**48**(1):53–60. Doi: 10.1016/j.jdsr.2011.11.002.
  35. Ferreira JC., Pires PT., Azevedo AF., Oliveira SA., Melo PR., Silva MJ. Influence of solvents and composition of etch-and-rinse and self-etch adhesive systems on the nanoleakage within the hybrid layer. *J Contemp Dent Pract* 2013;**14**(4):691–9. Doi: 10.5005/jp-journals-10024-1386.
  36. Ahmed EM. Hydrogel: Preparation, characterization, and applications: A review. *J Adv Res* 2015;**6**(2):105–21. Doi: <https://doi.org/10.1016/j.jare.2013.07.006>.
  37. Bettencourt AF., Neves CB., de Almeida MS., Pinheiro LM., Oliveira SA e., Lopes LP., et al. Biodegradation of acrylic based resins: A review. *Dent Mater* 2010;**26**(5):e171-80. Doi: 10.1016/j.dental.2010.01.006.
  38. Mine A., De Munck J., Cardoso MV., Van Landuyt KL., Poitevin A., Van Ende A., et al. Dentin-smear remains at self-etch adhesive interface. *Dent Mater* 2014;**30**(10):1147–53. Doi: 10.1016/j.dental.2014.07.006.
  39. Nagpal R., Manuja N., Pandit IK. Effect of ethanol wet bonding technique on the durability of resin- dentin bond with contemporary adhesive systems. *J Clin Pediatr Dent* 2015;**39**(2):133–42. Doi: 10.17796/jcpd.39.2.p14u4x3q14272452.
  40. Sofan E., Sofan A., Palaia G., Tenore G., Romeo U., Migliau G. Classification review of dental adhesive systems: from the IV generation to the universal type. *Ann Stomatol (Roma)* 2017;**8**(1):1–17. Doi: 10.11138/ads/2017.8.1.001.
  41. Patanjali S., Arora A., Arya A., Grewal MS. An In Vitro Study of Effect of Beveling of Enamel on Microleakage and Shear Bond Strength of Adhesive Systems in Primary and Permanent Teeth. *Int J Clin Pediatr Dent* 2019;**12**(3):205–10. Doi: 10.5005/jp-journals-10005-1623.
  42. Ferreira JC., Pires PT., de Azevedo ÁF., Arantes-Oliveira S., Silva MJ., de Melo PR. Morphology of the dentin-resin interface yielded by twostep etch-and-rinse adhesives with different solvents. *J Contemp Dent Pract* 2017;**18**(10):947–58. Doi: 10.5005/jp-journals-10024-2155.
  43. Zhuge R-S., Tian Y-M., Zhang Z-T., Ding N., Li Y-M., Zheng D-X. Improvement of Total Etching Dentin Bonding with Subpressure. *Sci Rep* 2017;**7**(1):6831. Doi: 10.1038/s41598-017-07281-x.
  44. Gupta A., Tavane P., Gupta PK., Tejolatha B., Lakhani AA., Tiwari R., et al. Evaluation of microleakage with total etch, self etch and universal adhesive systems in class V restorations: An in vitro study. *J Clin Diagnostic Res* 2017;**11**(4):ZC53–6. Doi: 10.7860/JCDR/2017/24907.9680.
  45. Catelan A., Pollard T., Bedran-Russo A., Santos P Dos., Ambrosano G., Aguiar F. Light-curing time and aging effects on the nanomechanical properties of methacrylate- and silorane-based restorations. *Oper Dent* 2014;**39**(4):389–97. Doi: 10.2341/12-504-L.
  46. Tezvergil-Mutluay A., Pashley D., Mutluay MM. Long-Term Durability of Dental Adhesives. *Curr Oral Heal Reports* 2015;**2**(4):174–81. Doi: 10.1007/s40496-015-0070-y.
  47. Carrilho E., Cardoso M., Marques Ferreira M., Marto CM., Paula A., Coelho AS. 10-MDP Based Dental Adhesives: Adhesive Interface Characterization and Adhesive Stability-A Systematic Review. *Mater (Basel, Switzerland)* 2019;**12**(5). Doi: 10.3390/ma12050790.
  48. Atash Biz Yeganeh L., Seyed Tabai E., Mohammadi Basir M. Bonding Durability of Four Adhesive Systems. *J Dent (Tehran)* 2015;**12**(8):563–70.
  49. Matos AB., Trevelin LT., Silva BTF da., Francisoni-Dos-Rios LF., Siriani LK., Cardoso MV. Bonding efficiency and durability: current possibilities. *Braz Oral Res* 2017;**31**(suppl 1):e57. Doi: 10.1590/1807-3107BOR-2017.vol31.0057.
  50. Kaczor K., Gerula-Szymańska A., Smektała T., Safranow K., Lewusz K., Nowicka A. Effects of different etching modes on the nanoleakage of universal adhesives: A systematic review and meta-analysis. *J Esthet Restor Dent Off Publ Am Acad Esthet Dent . [et Al]* 2018;**30**(4):287–98. Doi: 10.1111/jerd.12375.
  51. El Sayed HY., Abdalla AI., Shalby ME. Marginal microleakage of composite resin restorations bonded by desensitizing one step self etch adhesive. *Tanta Dent J* 2014;**11**(3):180–8. Doi: 10.1016/j.tdj.2014.10.001.





## تأثير أوضاع تطبيق النظام اللاصق المختلف على التسريب الدقيق عند اعاج الاسنان السطحي والعميق بعد فترات من التخزين متنوع

محمد احمد احمد وكوكا\*، محمود عبد العزيز2، كريم عدلي3، عبد المنعم عمرو4، ولييب محمد ولييب السباعي5

1. قسم العلاج التحفظي كلية طب الاسنان جامعة الأزهر بنين القاهرة، مصر
  2. قسم خواص المواد كلية طب الاسنان جامعة الأزهر أسيوط، مصر
  3. قسم تركيبات الاسنان الثابتة كلية طب الاسنان جامعة الأزهر بنين القاهرة، مصر
  4. قسم علاج الجذور كلية طب الاسنان جامعة الأزهر بنين القاهرة، مصر
  5. قسم العلاج التحفظي كلية طب الاسنان جامعة النهضة بني سويف، مصر
- \* البريد الإلكتروني: DRWAKWAK2006@AZHAR.EDU.EG

### الملخص :

**الهدف:** كان الهدف من هذه الدراسة هو تقييم التسرب النانوي لمركبات الراتنج المملوءة بالنانو باستخدام أنظمة لاصقة مختلفة في أعماق مختلفة من العاج.

**المواد والاساليب:** في هذا التحقيق . تم اختبار 120 ضرسًا بشريًا تمت إزالتها حديثًا. تم تقسيم الأسنان المختارة بشكل عشوائي إلى مجموعتين رئيسيتين (ن = 60) . مع تقسيم كل مجموعة رئيسية إلى مجموعتين أصغر (ن = 30) بناءً على نظام اللصق (وضع الحفر والشطف ووضع الحفر الذاتي للتطبيق). وفقًا لطبقات العاج السطحية والعميقة المستخدمة في عملية الالتصاق. تم فصل كل مجموعة صغيرة إلى مجموعتين فرعيتين أصغر (ن = 15). تم تقسيم كل مجموعة أصغر إلى ثلاث فئات مختلفة اعتمادًا على أطوال التخزين (24 ساعة و3 أشهر و6 أشهر) (ن = 5). تم إعادة بناء كل سن تدريجيًا باستخدام مركب الراتنج النانوي. تم قطع العينات طوليًا في اتجاهات الوسط البعيدة واللغوية الشدقية لإنشاء حزم من الراتنج العاج. لغرض تقييم التسرب النانوي . تم غمر الحزمة في نترات الفضة.

**النتائج:** لا توجد فروق ذات دلالة إحصائية بين الحفر الذاتي والحفر الكلي من حيث تأثير وضع التطبيق أو عمق العاج على التسرب النانوي . ومع ذلك كانت هناك زيادة ذات دلالة إحصائية في متوسط درجات التسرب النانوي من 24 ساعة إلى 3 أشهر و3 مدة التخزين تصل إلى 6 أشهر.

**الخلاصة:** إن استخدام مواد كيميائية للحفر الكلي أو مواد كيميائية ذاتية الربط يساعد على تقليل تسرب مركب الراتنج. نظرًا لأن نظام اللصق بالحفر الذاتي تميز بالحفر الكلي في التجاويف العميقة . يُنصح باستخدامه. في حين أن طريقة اللصق بالحفر الكلي . ينصح باستخدامها في التجاويف السطحية.

**الكلمات المفتاحية:** وضع الحفر والشطف، وضع الحفر الذاتي للتطبيق، التسرب النانوي، مركبات الراتنج المملوءة بالنانو، المؤشرات الكيميائية الحيوية، معايير اللصق