

# Microleakage of Nanofilled Resin-modified Glass-ionomer/Silorane- or Methacrylate-based Composite Sandwich Class II Restoration: Effect of Simultaneous Bonding

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## Clinical Relevance

One bonding step with SE Bond or Silorane Adhesive with either nano-ionomer or the respective composite could provide adequately sealed restorations in the deep interproximal box with a simplified and time-saving open sandwich technique.

## SUMMARY

**Objectives:** Microleakage of composite restorations at the cervical margin placed apically to the cemento-enamel junction (CEJ) is still a concern. This study evaluated the effect of simultaneous bonding application on cervical sealing of nano-ionomer/silorane- or methacrylate-based composite open sandwich Class II restorations in the modified technique compared with that of conventional bonding.

**Methods and Materials:** In 60 sound maxillary premolars, two standardized Class II cavities

were prepared with cervical margins 1 mm below the CEJ. The teeth were randomly divided into six groups of 10 teeth each. In the first three groups (groups 1-3), Clearfil SE Bond and Clearfil APX (Kuraray) were used for restoration in the total bonding technique (group 1), conventional open sandwich technique associated with a nano-ionomer (Ketac N100, 3M ESPE) (group 2), and modified open sandwich technique with simultaneous bonding application for both nano-ionomer and composite (group 3). In the second three groups (groups 4-6), Silorane Adhesive and Filtek Silorane composite (3M ESPE) were used in the same manner as in the first three groups, respectively.

**Results:** The simultaneous bonding application in the modified sandwich restorations (with SE Bond or Silorane Adhesive) resulted in a significant reduction of the cervical microleakage compared with that of the conventional bonding ( $p < 0.05$ ). However, microleakage of the modified technique was similar to that of

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**the total bonding (with SE Bond or Silorane Adhesive) ( $p > 0.05$ ), both showing good marginal seal.**

## INTRODUCTION

Resin composites have been widely used as a direct restorative material. Despite significant advances in adhesive systems, they are not capable of totally eliminating marginal microleakage, in particular at the gingival margin of deep Class II restorations.<sup>1-3</sup> The stresses induced by polymerization shrinkage, temperature fluctuations, and mechanical load cycling may disrupt the bond between dentin and resin composite, resulting in postoperative sensitivity, bacterial leakage, and secondary caries initiation.<sup>3-5</sup> Moreover, in a deep Class II cavity, effective curing of the first composite increment due to the distance between the light-curing tip and this layer may be a problem if a metal matrix is used.<sup>1,2</sup>

Resin-modified glass-ionomer (RMGI) is a good dentin replacement in such deep cavities; this is referred to as an open sandwich restoration.<sup>6</sup> This technique allows the dentist to benefit from the clinical advantages of RMGI materials, including tri- or dual-cure setting, fluoride release, low coefficient of thermal expansion, greater tolerance to moisture than resin composite, and reduced volume of resin used.<sup>7-9</sup> Furthermore, the high elastic deformation or flow capacity of RMGI during the early stage of setting can act as a stress absorber. This property leads to reduced stress transfer toward the bonding interface.<sup>10</sup> Consequently, improved marginal seal has been reported in several studies.<sup>1,6,9,11,12</sup> In the open sandwich technique, that is highly recommended for patients with a medium or high risk of caries, RMGI is applied on the gingival floor of the proximal box, extending out to the cavosurface margins. This exposure of RMGI to the oral environment may lead to surface deterioration because of the high solubility of RMGI in the oral fluid.<sup>13,14</sup>

In the closed sandwich technique, RMGI is fully veneered by composite.<sup>13-15</sup> However, in the latter technique, correct placement of RMGI short of the gingival cavosurface margin or removal of its excess from this inaccessible area of the cavity is difficult.<sup>15</sup>

Alternatively, a novel, highly packed, nanofilled RMGI, Nano-Ionomer (NI), can be used in an open sandwich technique because of its improved mechanical strength,<sup>16</sup> resistance to biomechanical degradation,<sup>17</sup> and fluoride release comparable to RMGI.<sup>18</sup> Lower polymerization shrinkage and better cervical sealing were reported in Class V NI

restorations.<sup>19</sup> When comparing abrasion resistance, NI behaved as an intermediate material between RMGI and nanocomposite.<sup>17</sup> The two-part paste of NI might facilitate its handling properties.<sup>16</sup>

On the other hand, new silorane-containing resin monomers using a combination of siloxane and oxirane have been developed based on cationic ring opening polymerization, resulting in reduced polymerization shrinkage of the hydrophobic composite.<sup>20,21</sup> The combination of NI and silorane composite in an open sandwich restoration may be a viable method to decrease the overall stresses within the whole restoration. The use of nano primer before NI and use of a silorane adhesive system before silorane composite are essential steps to bond these materials to tooth structure.<sup>16,21,22</sup> These separate and double-bonding applications lead to a time-consuming and complicated procedure. In addition, in the case of applying and light curing nano primer, this would possibly interfere with silorane adhesive conditioning before silorane composite placement. Silorane composite should be used solely with its dedicated adhesive.

The results of a recent study revealed that different self-etch adhesives can adequately bond the NI to the dentin, possibly providing simple NI/methacrylate composite sandwich restorations with one bonding step.<sup>23</sup> Again, the use of the same adhesive system (self-etch silorane adhesive) for NI and silorane composite would reduce the clinical application steps and time. Therefore, the present study aimed to investigate the marginal sealing of NI/silorane-based or methacrylate-based composite open sandwich technique with simultaneous bonding application compared with that of the conventional technique in deep Class II restorations.

## METHODS AND MATERIALS

Sixty sound human maxillary premolars recently extracted for orthodontic treatments were collected, cleaned, and disinfected in 0.5% chloramine solution for 2 weeks. The teeth were then stored in distilled water at 4°C until use.

Two standardized Class II box-only cavities were prepared on the mesial and distal surfaces of each tooth (3 mm wide, 5-6 mm high, and 1.5 mm deep) with gingival margins placed approximately 1 mm below the cemento-enamel junction (CEJ) using new straight fissure diamond burs (ISO 806 314, Hager & Meisinger GmbH, Neuss, Germany) for every five preparations. All dimensions of the preparations were verified with a periodontal probe. The buccal

Table 1: Materials Used and Their Composition			
Material	Batch No.	Manufacturer	Composition
Silorane adhesive	N213019 N213052	3M ESPE, St Paul, MN, USA	Primer: phosphorylated methacrylates, vitrebond copolymer, bis-GMA, HEMA, water, ethanol, silane-treated silica filler, initiators, stabilizers Bond: hydrophobic dimethacrylate, phosphorylated methacrylates, TEGDMA, silane-treated silica filler, initiators, stabilizers
Clearfil SE Bond	1039AA 1550AA	Kuraray Medical Inc, Okayama, Japan	Primer: MDP, HEMA, hydrophilic dimethacrylate, photoinitiator, water Bond: 10-MDP, bis-GMA, HEMA, hydrophilic dimethacrylate, microfiller, photoinitiator
Ketac N100 Primer	N251185	3M ESPE	HEMA, water, Vitrebond copolymer, photoinitiator
Ketac N100	N271282	3M ESPE	HEMA,vitrebond copolymer, water,TEGDMA, PEGDMA, bis-GMA, fluoroaluminosilicate glass, silane-treated zirconia/ silica, photoinitiators
Silorane composite	N384451	3M ESPE	3,4-epoxycyclohexylethylcyclo polymethylsiloxane;bis-3,4-epoxycyclohexylethyl – phenylmethylsilane; silanized quartz; ytiumfluoride; camphorquinone
Clearfil AP-X	1080AB	Kuraray Medical Inc	BisGMA, TEGDMA silanated glass filler, silanated silica filler, silanated colloidal silica, comphorquinone
Abbreviations: bis-GMA, bisphenol A diglycidyl ether dimethacrylate; HEMA, 2-hydroxy-ethyl-methacrylate; TEGDMA, triethylene glycol diemthacrylate; MDP, 10-methacryloxydecyl dihydrogen phosphate; PEGDMA, polyethylene glycol dimethacrylate.			

and lingual walls of the preparations were approximately parallel to each other and connected to the gingival wall with rounded line angles. All cavity preparations and restorations were performed by one operator. The materials used in this study are shown in Table 1.

The teeth were randomly divided into six groups of 10 teeth each (20 boxes in each group). The same materials and techniques were applied in the mesial and distal boxes of each tooth.

The restoration of the prepared teeth was performed as follows:

- Group 1 (total bonding SE): Primer of Clearfil SE Bond (Kuraray Inc) was applied to the cavity for 20 seconds and gently air dried for 5 seconds. The bond was then applied, thinned with a gentle air steam, and light cured for 10 seconds.
- Group 2 (conventional sandwich, NI primer+NI/ Clearfil SE Bond+methacrylate composite): Nano-ionomer primer was applied for 15 seconds, air dried, and light cured for 10 seconds; then, the two pastes were mixed and applied to the gingival floor with approximately 2-mm thickness extending to the periphery of the proximal box. After light curing of NI, Ketac N100 (KN100, 3M ESPE), the remaining cavity walls were treated in the same way as in group 1.
- Group 3 (modified sandwich, simultaneous Clearfil SE Bond application+NI/methacrylate composite): The bonding procedures were the same as in group 1. Then, KN100 was applied as described in group 2.
- Group 4 (total bonding silorane): Primer of silorane adhesive (3M ESPE) was applied to the cavity for 20 seconds and gently air dried for 10 seconds, and

the primer was light cured for 10 seconds. The bond was applied and light cured for 10 seconds.

- Group 5 (conventional sandwich, NI primer+NI/ silorane adhesive+silorane composite): The application of NI primer and KN100 was the same as in group 2. After light curing KN100, the remaining cavity walls were treated in the same manner as in group 4.
- Group 6 (simultaneous silorane adhesive application+NI/silorane composite): The bonding steps were done in the same manner as described in group 4. Then, KN100 was applied as performed in group 2.

After this step, a universal Tofflemire matrix retainer (Miltex Inc, York, PA, USA) with matrix band was placed around the tooth. The matrix was tightened and fixed by applying low-fusing compound so that formation of gingival overhang would not be allowed in the restorations. In the first three groups (groups 1-3), Clearfil APX composite-shade A2 (Kuraray Inc) was used; Filtek silorane composite-shade A2 (3M ESPE) was applied in the remaining three groups (groups 4-6), using an oblique incremental technique. Each layer was cured for 20 seconds for Clearfil APX and 40 seconds for silorane composite, according to manufacturers' instructions. All curing steps were done using a light-curing unit (VIP Junior, Bisco, Schaumburg, IL, USA) at 650 mW/cm<sup>2</sup> light intensity. The intensity was checked after every two restorations.

After matrix removal, the completed restorations were finished and polished with Opti-Disk (Kerr Corporation, Orange, CA, USA).The restored teeth were stored in distilled water at 37°C for 1 week to allow for complete acid-base reaction in NI and then

Table 2: Microleakage Score Frequency in the Six Study Groups

Group	Microleakage Scores					Mean* (SD)	Median
	0	1	2	3	4		
1. Total bonding SE	13	4	0	3	0	0.65 <sup>a</sup> (1.08)	0.0
2. Conventional sandwich SE	3	10	4	2	1	1.40 <sup>b</sup> (1.04)	1.0
3. Modified sandwich SE	15	4	0	1	0	0.35 <sup>a</sup> (0.74)	0.0
4. Total bonding silorane	18	1	1	0	0	0.30 <sup>a</sup> (0.65)	0.0
5. Conventional sandwich silorane	4	6	9	1	0	1.35 <sup>b</sup> (0.87)	1.50
6. Modified sandwich silorane	13	7	0	0	0	0.35 <sup>a</sup> (0.49)	0.0

\* Different superscript letters indicate statistically significant differences among groups.

thermocycled for 1000 cycles at 5°C to 55°C with a 30-second dwell time.

The root apices were sealed with utility wax, and all the surfaces, except for the restorations and an area 1 mm from the margins, were coated with two layers of nail varnish. The teeth were immersed in a 0.5% methylene blue dye solution for 24 hours. Upon removal from the dye, the teeth were rinsed, blotted dry, and sectioned vertically through the center of the restorations from the mesial to distal surface with a water-cooled diamond saw (Leitz 1600, Wetzlar, Germany)

The sections were blindly examined for dye penetration by two independent evaluators using a stereomicroscope (Carl Zeiss Inc, Oberkochen, Germany) at 20× magnification. The extent of the dye penetration was analyzed according to a 0 to 4 scale (0=no dye penetration, 1=dye penetration less than ½ of the gingival wall, 2=dye penetration along the gingival wall, 3=dye penetration along the gingival wall and less than ½ of the axial wall, 4=dye penetration along the gingival wall and axial wall). The worst score from the two sections of each specimen was recorded.

The Kruskal-Wallis test was used to analyze the differences within the groups. Pairwise comparison among different groups was done with Mann-Whitney U-test ( $\alpha=0.05$ ).

## RESULTS

Dye penetration scores in the six groups are presented in Table 2. None of the groups showed complete elimination of microleakage. According to the Kruskal-Wallis test, there was a significant difference in microleakage score among the six groups ( $p<0.001$ ).

Among the first three groups (with methacrylate composite), group 2 (conventional sandwich) revealed significantly higher cervical microleakage than group 1 (SE total bonding) and group 3

(modified sandwich) ( $p=0.009$  and  $p<0.001$ , respectively). There was no significant difference between group 1 and group 3 ( $p>0.05$ ), revealing similar low cervical microleakage in the two groups.

The results of comparing the second three groups (with silorane composite) were similar to those of methacrylate composite groups; group 5 showed a significantly higher microleakage at the cervical margin than groups 4 and 6 ( $p=0.001$  and  $p=0.009$ , respectively). However, no significant difference was found between groups 4 and 6 ( $p>0.05$ ).

Similar groups with methacrylate composite versus silorane composite (group 1 vs group 4, group 2 vs group 5, and group 3 vs group 6) showed no significant difference ( $p>0.05$ ) (Table 2). Examples of the specimens exhibiting different dye penetration scores are presented in Figure 1.

## DISCUSSION

Microleakage is one of the major problems affecting the longevity of resin composite restorations. The dye penetration method is the most common and simplest technique for assessing microleakage along adhesive interface.<sup>24</sup> Although correlation between clinical evaluation and *in vitro* dye penetration testing may not be documented, the latter is still a popular and valuable test as a preclinical screening to compare the sealing ability of different adhesive materials and techniques.<sup>25,26</sup>

According to the results of the present study, the best cervical marginal seal was obtained by total bonding with silorane composite, which revealed no significant difference with the total bonding associated to SE Bond. Both conventional sandwich techniques had significantly higher microleakage than that of the respective total bonding. This result contradicts previous studies that reported that the open sandwich technique resulted in similar or better marginal sealing than total bonding.<sup>1,6,9,11,12</sup> This difference can be attributed to the different

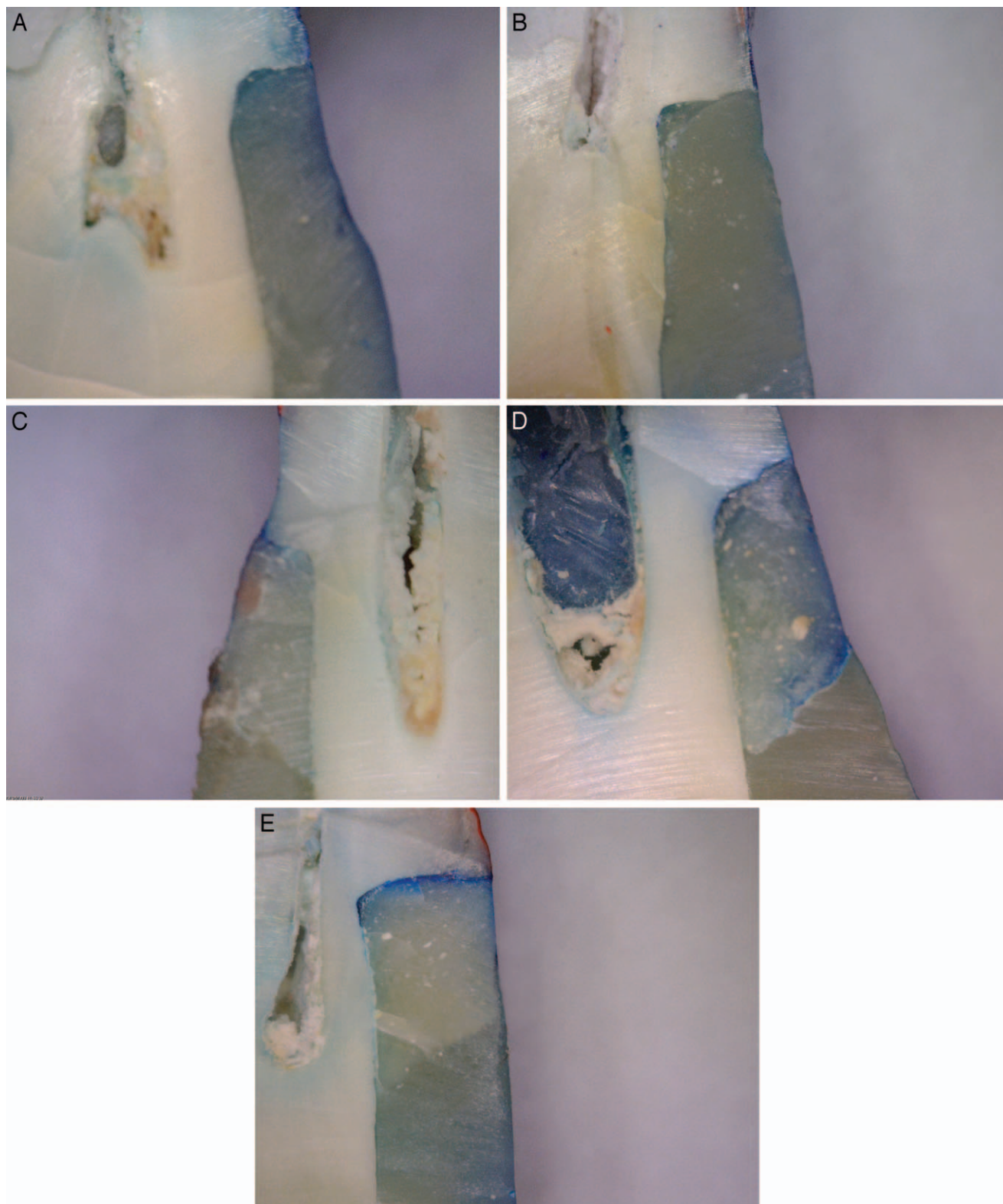


Figure 1. Examples of the restorations exhibiting different dye penetration scores.(A): Total bonding restoration showing score 0; (B): Modified sandwich restoration showing score 0; (C): Modified sandwich restoration showing score 1; (D): Conventional sandwich restoration showing score 2 with dye penetration at the interface; and (E): Conventional sandwich restoration showing score 3.

adhesive/composites and RMGI used in these studies. In particular, no study has compared the marginal quality of bonded silorane composite with sandwich restoration or the use of NI in sandwich technique versus total bonding.

So far, only Fahmy and Farrag<sup>27</sup> have recently evaluated microleakage in Class II primary molar cavities restored with NI/silorane or methacrylate nanofilled composite (open and closed sandwich) and total bonding. They found superior marginal sealing with total bonding compared with two sandwich techniques. This result is in agreement with our findings. Also, Beznos<sup>2</sup> concluded that RMGI could not prevent extensive leakage at the cervical margin of open sandwich restorations.

Previously, some authors speculated that the excellent sealing ability of silorane composite might be related to comparatively low volumetric shrinkage and shrinkage stress in this novel resin.<sup>22,28-30</sup> Slower polymerization reaction with the higher potential for stress relief by viscous flow of the molecules was reported to be responsible for this lower shrinkage stress rate and the resultant reduced stress.<sup>20,31,32</sup> Moreover, the bonding efficiency of the associated silorane adhesive system is an important factor.<sup>22,30</sup> The self-etching primer of this two-step adhesive has been recently claimed to create chemical bonding to the remaining hydroxyapatite crystals around collagens.<sup>21</sup> The tight interface at enamel and dentin was obtained via nano-interaction of this ultra-mild self-etch primer (pH=2.7). The separately applied and cured bond layer may contribute to maintaining the sealed interface.<sup>21</sup> On the other hand, somewhat higher but insignificant microleakage was obtained by total bonding with SE Bond/methacrylate compared with silorane adhesive/silorane composite. Again, it is well demonstrated that functional monomer (10-methacryloxydecyl dihydrogen phosphate [MDP]) in self-etch primer might be capable of bonding to the hydroxyapatite crystals.<sup>33</sup> This chemical bonding might lead to better resistance to hydrolytic degradation and improved sealing reported by some authors.<sup>15,34,35</sup>

Methacrylate composite used with SE Bond was applied in an incremental oblique layering technique. This method has been advocated to reduce overall contraction residual stress at the adhesive interface,<sup>28,36</sup> resulting in reduced microleakage by modifying C-factor and decreasing the composite bulk cured in each increment.<sup>22,37</sup> A recent clinical trial found no significant difference between marginal adaptation of silorane adhesive/silorane com-

posite and one-step self-etch adhesive/methacrylate composite.<sup>38</sup>

The findings of the present study indicate that the two modified sandwich techniques using the same adhesive (SE Bond or silorane adhesive) associated with NI substantially improved the marginal sealing of the restorations compared with the respective conventional sandwich approach so that the modified methods exhibited no significant difference from the total bonding ones.

A number of studies have recently demonstrated that the use of different self-etch adhesives could improve the bond strength or marginal sealing of RMGI.<sup>39-43</sup> The acidic conditioners commonly used with RMGI partially demineralize the smear layer and superficial dentin, facilitating the penetration of the 2-hydroxy-ethyl-methacrylate (HEMA) incorporated in the RMGI into the exposed collagen network.<sup>39</sup> The self-etching primer may act in a similar manner, and copolymerization poly-HEMA and hydrophilic monomers of the self-etching adhesive may occur.<sup>40</sup> On the other hand, RMGI can bond very well to the resin layer formed on the surface of self-etch adhesives via unsaturated carbon-carbon covalent bonds upon copolymerization, providing similar results to bonding of resin composites.<sup>41</sup>

KN100 in association with a light-cured primer was used in the conventional sandwich groups. This self-etch primer (pH=3) may create a resin covering on the primed dentin resembling those of mild self-etch adhesives.<sup>42</sup> Nevertheless, the low acidity of the primer may not allow the primer to totally dissolve the smear layer.<sup>16,42</sup>

According to scanning electron microscopy (SEM) evaluations of two recent studies,<sup>16,23</sup> NI interacted very superficially with the dentin without demineralization or hybrid layer formation. The primary bonding mechanism of NI was found to be micro-mechanical infiltration only into the surface roughness. The secondary one may be a typical polyalkenoic acid copolymer chemical bonding to dentin.

Therefore, it was expected that two self-etch adhesives used with NI in the modified technique could improve the cervical sealing of the restorations. It was recently reported that carboxylic groups of acidic monomers and Vitrebond copolymer in some self-etch adhesives increased the bond strength of NI to the dentin.<sup>23</sup> SE Bond has acidic monomer (MDP) in its primer composition. The ultra-mild self-etch primer of silorane adhesive contained Vitrebond copolymer.<sup>21</sup> This copolymer is also a key component

of NI. These functional monomers contribute to establishing chemical bonding to the dentin and improving the marginal sealing of the modified sandwich restoration examined in this study. The SEM evaluation of these adhesive interfaces is required to detect the real interaction with the dentin.

Although KN100 is a methacrylate-based RMGI, it seems to be compatible with silorane adhesive. The bond of this adhesive creates a hydrophobic methacrylate-based (with phosphate group) layer placed on the cured primer surface, providing bonding to methacrylate resins.<sup>44,45</sup> In the same manner, this resin layer could act between KN100 and silorane composite, and its phosphate group may react with the overlying silorane composite.<sup>45</sup> The compatibility between methacrylate-based composite and silorane adhesive was recently indicated.<sup>44,46</sup>

One possible drawback of the open sandwich technique is the creation of additional interfaces exposed to oral environment in a restoration. In the current study, most of the sandwich restorations showed no dye penetration at the interface of NI/composites; few specimens had slight dye penetration. This observation may be due to the use of the respective adhesive as a bonding layer with high wettability, the low shrinkage composite, and the small increments of the composites applied on NI, achieving a good union between these two materials.<sup>47</sup> The chemical bonding of KN100 and resin layer is accomplished in the presence of unpolymerized HEMA and unreacted methacrylate groups on the poly acid chain.<sup>48</sup>

Although the use of an adhesive layer may somewhat decrease fluoride release from RMGI, fluoride ions could still diffuse through the resin layer.<sup>39</sup> Moreover, this open sandwich technique would take advantage of the cariostatic potential of NI in the proximal surfaces of the adjacent teeth. Further long-term *in vitro* and *in vivo* studies are needed to confirm the obtained results.

## CONCLUSIONS

Within the limitations of this *in vitro* study, and based on obtained results, the introduced modified sandwich technique revealed no beneficial effect in terms of cervical sealing compared with the effects of total bonding. However, this technique exhibited significantly lower microleakage than the conventional technique. From a clinical consideration, the modified sandwich technique would facilitate the bonding procedures with simultaneous self-etching

adhesive application for both the NI and methacrylate- or silorane-based composite. This simplified combination would provide the benefits of two materials in a single restoration performed in a reduced application time.

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## Conflict of Interest

The authors of this manuscript certify that they have no proprietary, financial, or other personal interest of any nature or kind in any product, service, and/or company that is presented in this article.

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