

A Preliminary Study to Compare the Adaptability and Nanoleakage of Resin-Based Materials at the Cervical Dentin of Class II Cavity

Sukamon Kochotwuttinont¹, Mayurach Pipatphatsakorn¹ and Kornchanok Wayakanon¹

¹Department of Restorative Dentistry, Naresuan University, Phitsanulok

Abstract

Resin composite is the most popular tooth-colored material for the dental restoration. The polymerization shrinkage is an unavoidable disadvantage of this material which is associated with the gap formation and the secondary caries. Bonding system is used to eliminate these problems. However, the gingival margin of the proximal cavity remains the most common area found the restorative defects. An open sandwich technique has been suggested to address this problem. The aim of this study is to evaluate the degree of nanoleakage and adaptability of different lining materials in open sandwich technique. The slot cavities were prepared on the proximal surface of teeth with the gingival margin 1 mm below the CEJ. The teeth were divided into 5 groups (n=5): group I a flowable resin composite, group II a bulk fill flowable resin composite, group III a resin modified glass ionomer cement, group IV and V no lining material. Samples in the group I-III and V were restored with nanofilled resin composite while group IV were restored with bulk fill resin composite. All groups were thermocycled, processed with silver nitrate solution and observed under SEM. The silver nitrate deposited entire thickness of hybrid layer, in the dentinal tubules and on the resin tags in group I, II, IV and V. In group III, it deposited within the modified hybrid layer. The silver nitrate deposition was highest in group V (70.44 %) while the group II (55.29 %) was the lowest. The gap formation was found in almost all outer 1/3 of samples. The width of gap was different among materials. In the conclusion, the type of lining materials had an influence on the degree of adaptability to dentin while it did not effect on nanoleakage of bonding system. The bulk fill resin composite could improve the adaptability of the restoration to cervical dentin margin.

Keywords: Adaptability, Nanoleakage, Bulk fill resin composite, Open sandwich technique, Cervical dentin

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Correspondence to:

Kornchanok Wayakanon. 99 M. 9 Tha Pho, Muang, Phitsanulok 65000 Thailand Department of Restorative Dentistry, Naresuan University, Phitsanulok, Thailand Tel: 094-336-4994 E-mail: wkornchanok@hotmail.com

Introduction

A resin composite material becomes more popular as a filling material for the proximal cavities of the posterior teeth. It requires an uncomplicated form of the cavity preparation along with the preservation of tooth structure. However, the undeniable problems of light cured resin composite is the polymerization shrinkage. It is associated with the shrinkage stress consequently the occurrence of the gap formation, the post-operative sensitivity, secondary caries and the bond failure.^{1,2} Many studies reported marginal leakage at the cervical dentin of proximal cavities.^{3,4} The moisture contamination and the incomplete light curing at the bottom of the proximal cavities influence on this defect. The stiffness of the resin composites may not establish the proper adaptation to the internal surfaces or cavosurface of the proximal cavities.⁵ Applying the adhesive system on dentin is challenged due to the difference components in the natural structure of dentin.⁴ The different techniques have been suggested to reduce polymerization shrinkage and improve the adaptability of resin composite restorations such as the technique of light curing, placing of materials into cavities or applying other materials along with resin composite namely the open sandwich technique. For the latter technique, the materials with low modulus of elasticity or low polymerization shrinkage such as the flowable resin composite and resin modified glass ionomer cement (RMGIC) are advocated as a the gingival liner or intermediate layer.⁶⁻¹²

RMGICs have a chemical adhesion to the dentin and an anti-cariogenic effect. When applying RMGIC as the gingival lining materials along with the resin composite restoration, the total volume of the resin composite decreases resulting in the reduction of the shrinkage stress within the resin composite materials.¹³ Some studies found the substantial improvement of the marginal adaptation of the filling materials at the gingival margins.^{6,7} However, some studies showed the gaps at the RMGIC-dentin interface.¹⁴

An elastic cavity wall concept has been presented by using the flowable resin composite as a lining material. Due to the low modulus of elasticity, this gingival liner functions as a stress absorbing layer and reduces shrinkage stress at resin-dentin interface.⁸ Various studies reported that the placement of the flowable resin composite as the gingival liner minimizes the leakage at the gingival floor⁹⁻¹¹ and reduces the gap formation at the internal margins.¹² However some studies did not find any advantages from flowable resin composite because of the low filler content and high polymerization shrinkage.^{15,16}

Bulk fill resin composites have been recently developed to facilitate the clinical procedure. This material has the modification of the fillers by either reducing the filler content or increasing the filler-particle size in order to reduce the light scatter at the filler-matrix interface and increase the degree of light transmission.¹⁷⁻²⁰ They are subsequently able to fill into the cavity with the thickness 4-5 mm. Some studies reported that the bulk fill resin composites had less polymerization shrinkage stress and better marginal adaptation.^{17,21} This material has been also suggested to use as an alternative gingival lining material.

A nanoleakage investigation is a common method to investigate the quality of an adhesive system. It had been shown as nanometer-sized spaces within a hybrid layer even there was a gap-free gingival margin. A silver nitrate is the most popular substance to be used to detect the nano-spaces by observing its deposition under a high magnification Scanning Electron Microscope (SEM).^{22,23} This leakage is the result of an incomplete polymerization and infiltration of adhesive resin including the contamination at the bonding area. The nanoleakage is the considerable pathway for the penetration of bacterial products, oral fluid and dentinal fluid related to a hydrolytic degradation of adhesive resin and the bond failure.^{22,24}

Although using the lining materials in the class II resin composite open sandwich techniques improve

the marginal adaptability, there are some studies reported nanoleakage at the cervical dentin.²⁵ Both the marginal adaptability and the nanoleakage influence on the quality of the resin composite restorations. Nevertheless, there have been few studies to evaluate both adaptability and degree of nanoleakage of different liner materials in the class II resin composite open sandwich technique at the cervical dentin margin. Since the limitation of the information, the aims of this study were 1) to evaluate a degree of nanoleakage at a cervical dentin of three lining materials and high viscosity bulk fill resin composite in class II open sandwich technique and 2) to evaluate adaptability to a cervical dentin of three liner materials and high viscosity bulk fill resin composite in class II open sandwich technique.

Materials and Methods

1. Sample preparation

This study was approved by the Ethics committee of Naresuan University (IRB No. 578/59). The maxillary premolar teeth of patients aged above 20 years old recently extracted for orthodontic reasons were collected. They must have the normal morphological feature, no cavity, no restorations and no crack line or craze line. The extracted teeth were collected in 10 % formalin solution no longer than one month. Calculus and soft tissue were removed. All teeth were then submerged in fresh 10 % formalin solution for 2 weeks and stored in 0.1 % Thymol solution at room temperature.²⁶⁻²⁹

All teeth were mounted with sticky wax in the silicone blocks. After that occluso-distal slot cavities were prepared with 4-mm width in bucco-lingual direction. The gingival wall was finished 1 mm cervically to the CEJ to keep gingival margin on dentin. The width of gingival wall was 1.5 mm in the mesio-distal direction by high speed fissure diamond burs (#835 FG 016 Jota, Switzerland). Each bur was replaced with a new one after five cavity preparations. After that all prepared

teeth were horizontally sectioned on the occlusal surface by low speed diamond saw device with water coolant to receive the tooth samples with 4 mm occluso-cervical height. Then a tofflemire matrix holder and a metal band were placed. The teeth were assigned into 5 groups (N=24).

- Group I (FC): Conventional flowable resin composite and nanofilled resin composite;
 Filtek Z350 XT flowable resin®, 3M ESPE, n=5
 Filtek™ Z350XT Universal Restorative, 3M ESPE
- Group II (BF): Bulk fill flowable resin composite and nanofilled resin composite;
 SureFil SDR Flow®, Dentsply Caulk, n=5
 Filtek™ Z350XT Universal Restorative, 3M ESPE
- Group III (GI): Resin-modified glass ionomer cement and nanofilled resin composite;
 Fuji II LC capsule®, Accord, n=4
 Filtek™ Z350XT Universal Restorative, 3M ESPE
- Group IV (BFCo): No liner material, high viscosity bulk fill resin composite;
 Filtek™ Bulk Fill Posterior Restorative, 3M ESPE, n=5
- Group V (Co): No liner material, conventional nanofilled resin composite;
 Filtek™ Z350XT Universal Restorative, 3M ESPE, n=5

All cavities in group I and II were etched with 37 % phosphoric acid (Scotchbond™Etching liquid, 3M ESPE) for 15s then rinsed with water jet for 30s and gently air dried for 30 seconds. The bonding agent (Adper™Single Bond2, 3M ESPE) was applied according to the manufacturer's instruction. For the group III, the GC conditioner liquid was applied to gingival floor for 10s then rinsed with water jet for 30s and gently dried for 30 seconds. The thickness of lining materials in group I, II and III was 1 mm. It is considered and adjusted with the periodontal probe by measuring the occlusally remaining space before light curing for 20s.

After placement the lining materials, the cavities (group I, II and III) were restored by incremental technique with 2-mm increments of nanofilled resin

composite (Flitek™Z350 XT Universal restorative, 3M ESPE) and light cured for 20s on each layer. For the group IV and V, the cavities were treated with 37 % phosphoric acid (Scotchbond™Etching liquid, 3M ESPE) and the bonding agent (Adper™ Single Bond², 3M ESPE) as mentioned. The group IV was bulkily restored with bulk fill resin composite (Filtek™ Bulk Fill Posterior Restorative, 3M ESPE). The group V was incrementally restored with the nanofilled resin composite (Flitek™Z350 XT Universal restorative, 3M ESPE).

After the tofflemire matrix holder and metal band were removed, all samples were light cured for 20s at buccal and palatal aspects by using LED light curing unit (Mini LED ACTEON, France) with light intensity 2,000 mW/cm². Then all samples were stored in distilled water at 37°C for 24h and subjected to thermal cycling for 2000 cycles with temperature range of 5°C to 55°C with dwell time of 15s and 7s transferred time.^{30,31}

2. Nanoleakage evaluation

The samples in group I, II and III were coated with two layers of nail varnish excepted 1 mm surrounding the liner material and 1 mm around the cervical dentin margin in group IV and V. All samples were immersed in a 50 % ammoniacal silver nitrate solution (pH=9.5) for 24 h in the dark. Then, they were thoroughly rinsed with distilled water and immersed in a photo-developing solution for 8 h under fluorescent light to reduce diamine silver ions to metallic silver grains.³²

The samples were fixed in 2.5 % glutaraldehyde in 0.1 M PBS buffer at pH 7.4 for 12 h at 4°C. After fixation, the specimens were rinsed by distilled water for 1 min. The samples were longitudinally sectioned in a mesio-distal direction through the center of the restorations. They were processed,³³ mounted on aluminum stubs, sputter-coated with gold and observed under SEM using backscattered electron mode (magnification x1000).

3. Data analysis

The distance of silver nitrate deposition along the gingival floor was measured from the SEM micrographs by Image J software program. The extension of nanoleakage

was calculated as the percentage of silver nitrate deposition on the gingival floor. In addition, the SEM micrographs were measured the gap width in three points of each sample (inner point, middle point and outer point of silver nitrate deposition). Normal distribution was verified with the Shapiro-Wilk test and homogeneity by Levene's test. The mean percentages of silver nitrate deposition among groups were compared by Kruskal-Wallis Test. The mean gap widths at the gingival floor among groups were compared by Kruskal-Wallis Test followed by Mann Whitney U test. ($p<0.05$).

Results

The SEM micrographs presented the thickness of hybrid layer, the pattern of silver nitrate deposition and the gap formation (Fig. 1). In group I, II, IV and V the silver nitrate deposited entire thickness of hybrid layer, penetrated into the dentinal tubules and deposited on the resin tags. The inner and middle areas of the gingival wall presented the thicker hybrid layers compared with that of the outer area. In group III, all specimens presented modified hybrid layer which is clearly thinner than the hybrid layers of group I, II, IV and V. These modified hybrid layer had the silver nitrate deposition and it also penetrated into the dentinal tubules.

The percentages of silver nitrate deposition were shown in Table 1. Group V (control) had the highest percentage of silver nitrate deposition (70.44 %), followed by group I (64.78 %), III (62.49 %), IV (58.96 %) and II (55.29 %). However there was no statistically significant difference among the restorative materials ($p>0.05$).

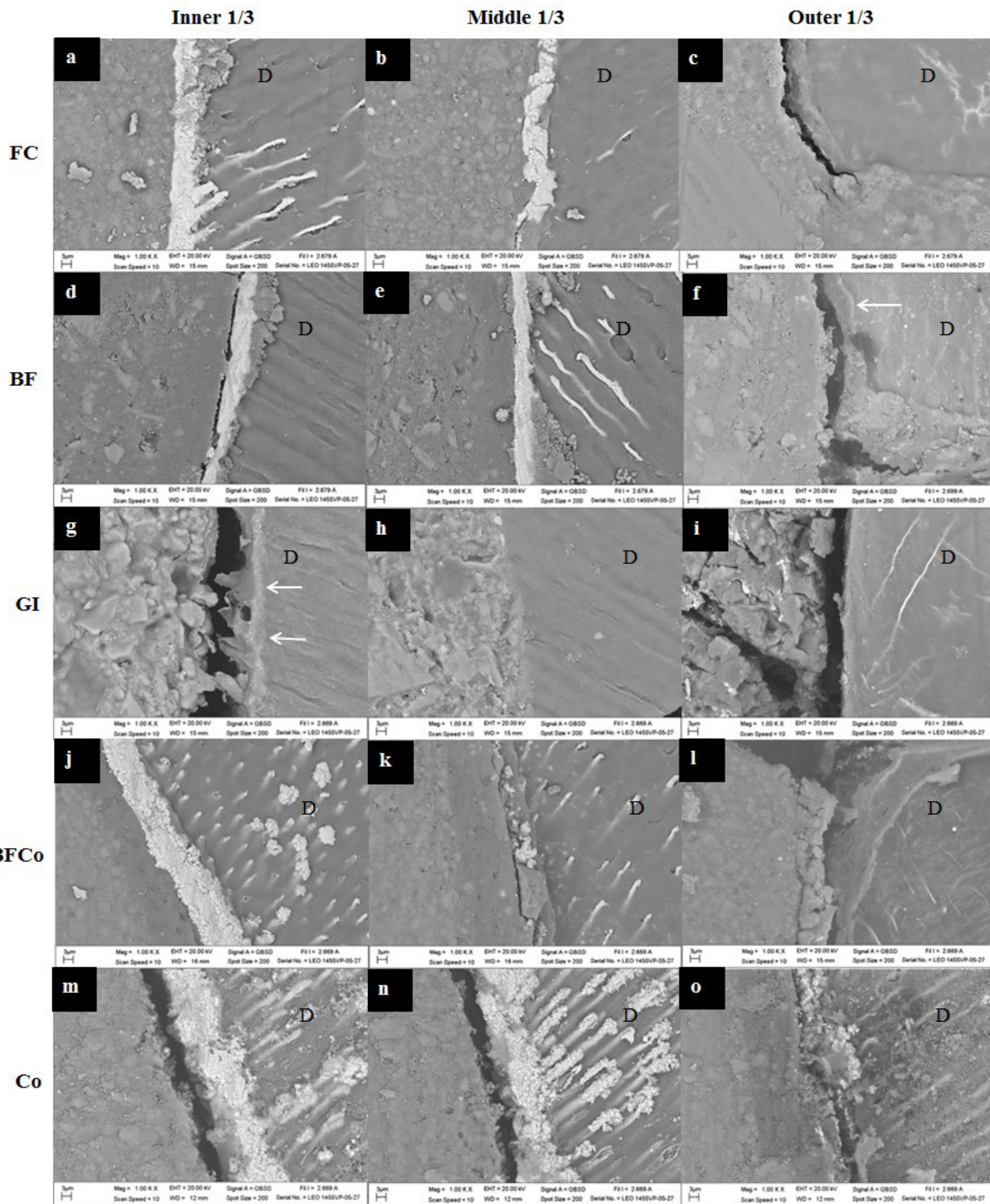


Figure 1 The micrographs from SEM (1000x) present the thickness of hybrid layer, pattern of silver nitrate deposition and gap formation. Silver nitrate has a similar deposition pattern in all groups. It deposits in the dentinal tubules, resin tags and entire thickness of hybrid layer. Gaps are found between hybrid layer and bonding layer. Some areas have a silver nitrate deposition at the base of hybrid layer (arrow). The GI group shows the modified hybrid layer. The density of silver nitrate deposition in this group is clearly lower than others. (D=Dentin, FC=conventional flowable resin composite, BF=bulk fill flowable resin composite, GI=resin modified glass ionomer cement, BFCo = bulk fill resin composite, Co=conventional nanofilled resin composite)

Table 1 The mean and standard deviation (SD) of percentage of silver nitrate deposition at the cervical dentin

Type of restoration	N	% of silver nitrate deposition Mean (SD)
Group I (FC+Co)	5	64.78 (14.14) ^a
Group II (BF+Co)	5	55.29 (13.47) ^a
Group III (GI+Co)	4	62.49 (4.14) ^a
Group IV (BFCo)	5	58.96 (1.46) ^a
Group V (Co)	5	70.44 (16.37) ^a

Lower case characters represent statistically significant differences ($p < 0.05$)

The gap formation was found in almost all outer area of samples. The gap formation presented between the hybrid layer or modified hybrid layer and

materials (Fig. 1). The width of gap was different among materials and the positions in the cavity as shown in table 2.

Table 2 The mean and standard deviation (SD) of the gap width at the cervical dentin margin

Type of restoration	N	Gap width between hybrid layer & liner mean (SD), μm		
		Outer	Middle	Inner
Group I (FC+Co)	5	5.23 (2.94) ^{A,a}	0.41 (0.92) ^{B,a,b}	0.00 (0.00) ^{B,a}
Group II (BF+Co)	5	1.35 (1.30) ^{A,a}	0.00 (0.00) ^{A,a}	0.18 (0.26) ^{A,a}
Group III (GI+Co)	4	2.70 (3.12) ^{A,a}	0.00 (0.00) ^{A,a}	4.45 (2.36) ^{A,b}
Group IV (BFCo)	5	1.73 (0.91) ^{A,a}	0.00 (0.00) ^{B,a}	0.00 (0.00) ^{B,a}
Group V (Co)	5	3.88 (1.56) ^{A,a}	1.18 (0.74) ^{A,b}	2.38 (2.19) ^{A,a,b}

Lower case characters represent statistically significant differences ($p < 0.05$) within columns

Upper case characters represent statistically significant differences ($p < 0.05$) within rows

The group I, II, IV and V had the largest gaps in the outer area (5.23, 1.35, 1.73 and 3.88 μm respectively) while the inner area of group III showed the largest ones (4.45 μm). The gap width of the outer area of group I and IV was significantly larger than other areas ($p=0.043$). However only group IV did not present gap formation at inner and middle area. In the group III had significantly larger size of gap at the inner area when compared with

group I, II and IV ($p=0.016$). There was no gap formation in the middle area in all samples in group II, III and IV. The group V had the significantly larger size of gap in the middle when compared with group II, III and IV ($p=0.032$).

The simple correlation analysis presented no correlation between the mean percentage of silver nitrate deposition and the gap width at gingival floor among five groups ($p > 0.05$) (data not shown).

Discussion

The silver nitrate deposition represents the incomplete bonding of either an adhesive systems or restorative materials. These defects occur as the nanometer-sized spaces around the collagen fibrils within the hybrid layer. They are a result of incomplete infiltration of adhesive resin into a demineralized dentin.^{22-24,34,35}

The percentages of silver nitrate deposition among three lining materials in class II resin composite open sandwich technique were no statistical difference. The pattern of silver nitrate deposition in group I, II, IV and V had the similar pattern because these four groups used the same adhesive system (Single Bond2). The degree of polymerization shrinkage of all resin-based materials did not influence on the nanoleakage of bonding agent.

The majority of silver nitrate deposited at the dentin side of hybrid layer. This probably denoted the accumulation of shrunk collagen fibers on the dentin surface after applying etchant.³⁴ This thin layer (0.2-0.3 μm) might interfere the adhesive resin infiltration, consequently the silver ion precipitation.²² In addition, the components of the adhesive reagent probably affected the silver nitrate deposition. The adhesives with high percentage of hydrophilic monomers demonstrated a high degree of permeability after polymerization and the silver nitrate deposition.³⁶ The Single Bond2 containing HEMA, which is hydrophilic monomer, improves their infiltrating ability into the moist substrate. However, HEMA has low water vapor pressure, so water commonly retains in the layers which have the adhesive reagent. Consequently, the hybrid layer acted as a hydrogel which promotes silver nitrate deposition.³⁷

Some specimens presented the silver nitrate deposition at the material side of hybrid layer. This result was similar to the previously studies.^{24,38-40} Van Meerbeek *et al.* found an amorphous electron-dense phase on the top of the hybrid layer for Scotchbond Multi-Purpose, which contained polyalkenoic acid. They

suggested that it represented a phase separation of the polyalkenoic acid copolymer from the other primer ingredients, which reacted with calcium to form calcium-polycarboxylate salts.^{39,40} In addition, Vargas *et al.* found amorphous hybrid layer-like structures above the hybrid layer in SEM observations, for Single Bond and Scotchbond Multi-Purpose.³⁸ Li *et al.* observed the amorphous structure in Single Bond and One Coat Bond, which contain polyalkenoic acid. This amorphous structure uptake silver ions on the top of the hybrid layer.²⁴

Almost all samples showed the thick hybrid layer and silver nitrate deposition at the inner area. The air blowing for evaporating solvent probably caused the accumulation of adhesive resin at the inner line angle. The thick layers of adhesive resin might prevent the proper evaporation of solvent, resulting in poor polymerization.⁴¹ The residual monomer probably caused the infiltration of silver nitrate within the resin^{42,43} consequently the deposition of silver nitrate within a hybrid layer or entire thickness of hybrid layer. Additionally, the moist bonding technique might leave the excess water along the line angle interfering the evaporation of solvent and resulting in the incomplete polymerization of resin.

The discontinuous silver nitrate deposition within hybrid layer may be indicated that the hybrid layer is not uniform. Some parts of hybrid layer are probably well polymerized and others poorly polymerized.

The group III (GI+Co) showed the deposition of silver nitrate within the modified hybrid layer and within the mass of the resin modified glass ionomer cement. The hydrophilic functional monomers contained in resin modified glass ionomer cement can absorb water resulting in hydrolytic degradation and silver nitrate deposition.⁴⁴ In addition, the porosity of material probably causes silver nitrate deposition within the material mass.^{45,46}

Almost all specimens demonstrated a gap formation at the outer area of the cervical dentin. The

gap width of the outer area of group I (5.23 μm) was larger than that of group V (control) (3.88 μm). The gap width of the outer area of group II (1.35 μm), III (2.70 μm) and IV (1.73 μm) were smaller than that of group V (control). However they were insignificantly different. These results might imply that the bulk fill flowable resin composite and the resin modified glass ionomer cement using as the liners in the class II resin composite open sandwich technique might be able to improve the adaptability of the restoration at the outer area of the internal wall. In the other hand, applying the conventional flowable resin composite as the liner in the class II resin composite open sandwich technique cannot improve the marginal adaptability. Moreover this present study advocated that the bulk fill resin composite (either flowable bulk fill or conventional bulk fill resin composites) with the bulk filled technique can improve the marginal adaptability.

Regarding the inner area, the gap width of group III was insignificantly larger than the control group. The RMGI material is more viscous than the bonding agent, which has the chemical bond to resin composite materials. The flow rate of RMGI is low, so its adaptability is less than the bonding agent. The inner area is the most critical area for the adaptability of filling materials. When the gap is formed at this area, the restorative material is more susceptible to the hydrolytic degradation. From this issue, it might need further studies for the degradation of RMGI due to the large gap formation at the inner area.

The conventional flowable resin composite has a low modulus of elasticity. A placement of flowable resin composite as a lining material can dissipate stress and reduce shrinkage stress of resin composite restorative material at tooth-restoration interface.^{8,47,48} In addition, the flowable resin composite has low surface tension, therefore this material can penetrate into the irregularity surface resulting in better adaptability.^{9-11,49} In the other hand, the conventional flowable resin composite contains 20-25 % less filler than conventional materials and larger amount of diluent monomers resulting in high

polymerization shrinkage.^{16,50} The diluent monomer especially TEGDMA, which contains in Filtek™ Z350 XT flowable, has a small molecule with more active sites leading to negative effect on polymerization shrinkage.⁵¹

The results from this study were similar to the previous ones which reported that the bulk fill flowable resin composites and high viscosity bulk fill resin composites showed better dentin marginal adaptation and less gap formation compared with conventional flowable resin composite.^{19,21,30} The bulk fill resin composites demonstrated the low polymerization shrinkage stress and the high degree of light transmission because of the reduction of light scattering at filler-matrix interface by either reducing the filler contents or increasing the filler particle size.^{17,18} The SureFil SDR Flow® was added a modified urethane dimethacrylate in an organic part together with the photoactive groups leading to the reduction of the shrinkage stress.⁵² The occurrence of gap formation might be related with the polymerization shrinkage of materials. The SureFil SDR Flow® has small gap formation compared with others probably due to low polymerization shrinkage. Nevertheless, the bulk fill flowable resin composite has significantly lower mechanical properties compared with the high viscosity bulk fill nanohybrid and conventional flowable resin composite.^{18,52} Therefore, the manufacturers recommend using this material as the intermediate layer.

The Filtek™ Bulk fill Posterior restorative is a high viscosity bulk fill resin composite. It contains two novel methacrylate monomers; a high molecular weight aromatic dimethacrylate (AUDMA) and an additional fragmentation monomer (AFM). The AUDMA has less reactive groups than conventional dimetahcrylate monomer. This might decrease the polymerization shrinkage of polymers. The AFM has a reactive site to cleave through a fragmentation process during polymerization. This can lead to the stress relief after polymerization.⁵³

The resin modified glass ionomer cement was an alternative lining material for class II resin composite

open sandwich technique which may improve the marginal adaptability regarding to the outer area.^{6,7,54,55} However the longevity of RMGI is likely to have further study due to the large gap formation at the inner area.

Discussion

Type of resin-base materials has no influence on the nano-leakage of restorative materials at the cervical dentin of class II cavity when using the same kinds of bonding agent. The bulk fill flowable resin composite (SureFil SDR Flow[®]) and the high viscosity bulk fill resin composite (Filtek[™] Bulk fill Posterior restorative) can improve the marginal adaptability at the cervical dentin of class II cavity.

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