

# Polymerization characteristics, flexural modulus and microleakage evaluation of silorane-based and methacrylate-based composites

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**ABSTRACT: Purpose:** To compare the behavior of a new low-shrinkage silorane-based composite (P90) with two conventional methacrylate-based composites, in terms of polymerization shrinkage, polymerization stress, gel point, flexural modulus and microleakage. **Methods:** The materials tested were P90 (3M ESPE), AP-X (Kuraray), Quixfil (Dentsply). Polymerization shrinkage was measured using the Accuvol method. Polymerization stress was assessed using a stress-strain analyzer. Polymerization shrinkage (%) and force (N) were continuously recorded for 300 seconds. Polymerization shrinkage and stress after 300 seconds and gel point were recorded. Flexural modulus was obtained by three-point bending. A laboratory microleakage test was performed. Class V cavities prepared on labial surfaces of 70 freshly extracted human teeth were randomly assigned to the seven groups (N=10): P90/PSA (P90 System Adhesive), AP-X/CBA (Clearfil SE Bond Adhesive), Quixfil/XBA (XP Bond Adhesive), P90/CBA, P90/XBA, AP-X/PSA, Quixfil/PSA. The teeth were immersed in 1% basic fuchsin dye for 24 hours at room temperature. All teeth were cut in a labial-lingual direction and the penetration of dye along the wall of cavities was observed under a stereoscopic light microscope. Statistical analysis was performed with one-way ANOVA, Kruskal-Wallis ANOVA test and post hoc tests ( $P < 0.05$ ). **Results:** The low-shrinkage silorane-based composite (P90) exhibited significantly lower polymerization shrinkage, polymerization stress, flexural modulus and a later gel point than the conventional methacrylate-based composites. In the microleakage test, P90/PSA, AP-X/CBA, AP-X/PSA demonstrated the lowest microleakage scores without a significant difference among them ( $P > 0.05$ ). Quixfil/XPA exhibited the highest microleakage scores. (*Am J Dent* 2011;24:97-102).

**CLINICAL SIGNIFICANCE:** The low-shrinkage silorane-based composite (P90) tested seemed to represent an improvement in terms of extending the gel point and reducing polymerization shrinkage and stress. However, compared with conventional methacrylate-based composite AP-X, P90 did not show significantly better interfacial integrity, suggesting that factors other than polymerization stress influenced the microleakage, for instance, adhesive system and stiffness of uncured filling materials.

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## Introduction

Although composites are now the material of choice for most restorations,<sup>1</sup> the volumetric shrinkage that accompanies the chain-growth polymerization of methacrylate monomers remains a major concern for the clinical performance of composite restorations.

Composite polymerization can be divided in pre- and post-gel phases according to the 'gel point'. In the pre-gel phase, the reactive species present enough mobility to re-arrange and compensate for the volumetric shrinkage without generating significant amounts of internal and interfacial stresses. At gel point, termination prevails over chain propagation forming a continuous network,<sup>2</sup> acquiring adequate elastic modulus to resist plastic flow. At the same time, some viscous deformation is still available, but it is not enough to counterbalance setting shrinkage and thus stresses are generated within the material at the tooth/restoration interface and in the tooth structure.<sup>3,4</sup> This stress state is likely to facilitate gap formation, jeopardizing the longevity of the restoration.<sup>5,6</sup>

Since long, reduced polymerization shrinkage is a highly desired property of composites in order to reduce the occurrence of these clinically problems, a low-shrinkage silorane-based composite, marketed as Filtek P90,<sup>a</sup> was introduced. So-called siloranes replace the methacrylates in the resin matrix of dental composites.<sup>7,8</sup> The ring-opening chemistry of the resin reduces shrinkage of the composite below 1 vol%.<sup>9</sup> The P90

comes with a two-step self-etch adhesive, commercialized as P90 System Adhesive.<sup>b</sup> (PSA). First, a hydrophilic self-etch primer (PSA Self-Etch Primer) is applied and light-cured separately prior to the application of a hydrophobic adhesive resin (PSA Bond). PSA Bond is methacrylate-based and is therefore compatible with conventional methacrylate composites as well.<sup>10</sup> Previous research<sup>10-12</sup> proved that the PSA performed in combination with Filtek Z100<sup>a</sup> equally as well as other two-step self-etch adhesives such as AdheSE<sup>b</sup> or Protect Bond<sup>b</sup> in terms of  $\mu$ TBS.

A previous study<sup>13</sup> showed less microleakage with P90 compared to clinically successful methacrylate-based composite Z250. Yamazak *et al*<sup>14</sup> also showed that silorane-based composite resulted in significantly lower microleakage than Tetric Ceram<sup>c</sup> after loading, but there was no significant difference between them without mechanical loading.

Conversely, Palin *et al*<sup>15</sup> found that the use of a silorane composite in Class V restorations did not affect the development of marginal microleakage when compared to conventional composite. The marginal integrity research<sup>16</sup> investigated by SEM and dye penetration analysis demonstrated that the silorane-based composite system showed greater leakage along the enamel margins than the conventional composite Tetric Ceram with adhesive (Clearfil SE Bond<sup>d</sup>), but the silorane adhesive applied was an all-in-one experimental bond of silorane previously produced by the company and we used the new bond produced with silorane which is a two-step, two-compo-

Table 1. Materials used in the investigation.

Composite	Type	Shade	Filler weight%
Filtek P90	Silorane-based posterior composite	B2	76
AP-X	Universal composite	A3	86
Quixfil	Posterior composite	A3	85.5

nent bond.

A recent study<sup>10</sup> also demonstrated that the conventional composite Filtek Z100<sup>a</sup> and the silorane-based composite P90 bonded equally well to dentin using the two-step self-etch PSA in terms of  $\mu$ TBS.

Actually, marginal gap formation is the result of a localized bond failure and could be attributed to many factors such as the configuration of cavities, polymerization stress, stiffness of uncured restorative materials,<sup>10</sup> adhesive system,<sup>17</sup> and so on. Moreover, the analysis of sectioned specimens is very limiting, and may not be representative of the whole specimen. Considering these facts, it is not surprising that different microleakage results were observed in the silorane-based composite in comparison to different methacrylate-based composites.

This investigation assessed polymerization shrinkage, polymerization stress, gel point, flexural modulus and microleakage of the silorane-based composite (P90) compared with conventional methacrylate composites. The tested hypothesis was that differences in polymerization reaction of the novel silorane-based composite would result in extended gel point, decreased polymerization shrinkage, polymerization stress and microleakage and increased marginal integrity compared with conventional methacrylate composites at the tooth/restoration interface.

### Materials and Methods

**Samples** - AP-X<sup>d</sup> and Quixfil<sup>e</sup> were applied as the control materials because Quixfil is a low-shrinkage posterior composite used as a control for silorane-based composites in other studies,<sup>9,18-20</sup> while AP-X is a clinically successful low-shrinkage methacrylate-based composite.<sup>21</sup> Three light-cured materials were studied as listed in Table 1.

**Polymerization shrinkage** - Volumetric polymerization shrinkage was obtained in the laboratory using Accuvol.<sup>f,22</sup> A 6-9 ml sample (n= 7) of uncured material (2 mm-thick) was placed on the cylindrical PTFE pedestal. The sample was imaged by a digital video-camera. Special software was employed for the acquisition and processing of images. The edges of each sample image were detected by thresholding and the volume was reconstructed after image segmentation. The uncured sample was placed on the pedestal and had reached a stable near-hemispheric shape about 2 minutes later. Then the material was light cured from the top at a 1-2 mm distance using a curing light (Elipar 2500<sup>a</sup>) at 550 mW/cm<sup>2</sup>. A new series of images was then acquired for the set material. Real time percentage values of volumetric changes were monitored until 300 seconds from start of light-cure, and therefore the shrinkage values were read at 300 seconds for all samples. However, this device cannot monitor the dynamic process of exposure, because the strong curing light would affect the imaging of the digital video-camera severely. Each experiment was conducted at room temperature (23-24°C).

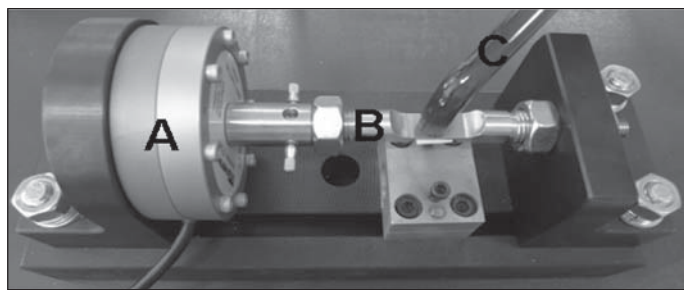


Fig. 1. Polymerization stress test set-up. (A) Load sensor, (B) Combined steel mold, (C) Curing light.

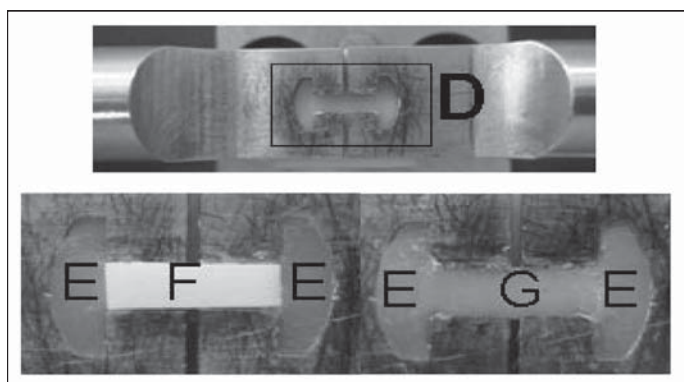


Fig. 2. Detailed drawing. (D) Curing cavity, (E) Composite attachment, (F) A piece of PTFE 6 mm x 2 mm x 2 mm, (G) Composite sample under test.

**Polymerization stress** - Polymerization stress was assessed using a stress-strain analyzer (Fig. 1). The setup consisted of two identical opposing stainless steel molds. One was connected to a 100 N load sensor (1 mN resolution, Instron<sup>g</sup>) and the other was fixed to the steel frame of the device.

Parallel composite attachments were prepared with a preset distance of 6 mm. A PTFE tray was fixed to the testing device, holding the two attachments, building up a simulated cavity (L 6 mm x W 2 mm x H 2 mm) between the attachments (Fig. 2).

As the composites needed free and bonded surfaces to simulate a dental cavity, the inner surface of the mold was burnished and coated with a lubricant to reduce the effects of adhesive friction during the polymerization shrinkage process. The non-adhesive PTFE tray was applied for the same reason. The configuration factor (C-factor) was calculated to be 0.17.

Super-Cor<sup>h</sup> composite was used to prepare the specimens attachments. Attachments were renewed before each measurement and polymerized (40 seconds) with a quartz-tungsten halogen curing light (Elipar 2500<sup>a</sup>) at 550 mW/cm<sup>2</sup>. Then the attachments rested for 10 minutes until their polymerization shrinkage and elastic modulus plateaued to a constant.

Before each measurement, a layer of adhesive (PSA bond) was applied and polymerized for 20 seconds with the halogen curing light. Composite paste for each tested material was placed in the cavity in bulk and then polymerized with the halogen curing unit for 40 seconds. The polymerization force (N) was continuously recorded for 300 seconds after light curing. In our preliminary investigations revealed that a plateau of the curve was achieved after that time. From the force values that were obtained, the polymerization stresses (MPa) were calculated. An arbitrary value for the gel point was also calculated. When the material gels, the force builds up, so, with the help of the force value the gelation point can be determined.

In this experiment, the time at which the force reaches 0.1 N was considered as an indirect measure of the gel point. A force value was required which was at a safety distance from the background noise. This safe distance was being attained at a force value of 0.1 N. This is the value obtained when the noise level from the machine is multiplied by a factor of 10.<sup>23</sup> Each experiment was conducted at room temperature (23-24°C) and repeated 10 times for each material (N= 10).

**Microleakage test** - In the microleakage test, 70 freshly extracted human caries- and restoration-free maxillary premolars were cleaned from calculus, soft tissue and other debris and stored in distilled water at 4°C. Prior to the study commencing, the teeth were placed in 0.5% chloramine T solution for 1 week at 4°C.

Three composites and three adhesives were examined in this study. The composites were listed in Table 1. The adhesives included two two-step self-etch adhesive systems: P90 System Adhesive (PSA) and Clearfil SE-Bond Adhesive (CBA) and a two-step etch-and-rinse adhesive system: XP-Bond Adhesive (XBA). The teeth were randomly divided into the seven groups (N = 10). Three different types of comparisons were made:

A. Comparison of different restorative systems (composite + adhesive of the same manufacturer):

- Group 1 (G1): P90 with PSA;
- Group 2 (G2): AP-X with CBA;
- Group 3 (G3): Quixfil with XBA;

B. Comparison of different composite with the same adhesive (PSA):

- Group 1 (G1): P90 with PSA;
- Control Group 1 (CG1): AP-X with PSA;
- Control Group 2: (CG2): Quixfil with PSA;

C. Comparison of different adhesives with the same composite (P90):

- Group 1 (G1): P90 with PSA;
- Control Group 3 (CG3): P90 with CBA;
- Control Group 4 (CG4): P90 with XBA;

In Comparison A, recommended restorative systems were applied to achieve the optimal restoration effect, respectively. In Comparison B, PSA was applied to rule out differences between adhesive techniques. In Comparison C, it can be proved whether P90 could be compatible with adhesives other than PSA.

A standard reproducible Class V cavity (a standard 3 mm diameter cavity with a depth of at least 1 mm into the dentin) was prepared on the mid-part of buccal surface of the teeth, with a periodontal probe used during the procedure to measure the preparations. The cavities were made with customized cylindrical diamond burs<sup>1</sup> with a high-speed handpiece using air/water spray. The filling procedures were carried out as follows:

Group 1 (G1), Control Group 1 (CG1) and Control Group 2 (CG2):

1. Apply the primer (PSA Self-Etch Primer) to the entire surface of the cavity and massage over the entire area for 15 seconds. Spread the primer to an even film with a gentle stream of air. Cure the primer for 10 seconds with the halogen curing light.
2. Apply the bond (PSA bond) to the entire area of the cavity.

Use a gentle stream of air until the bond is spread to an even film and cure the bond for 10 seconds with the halogen curing light.

3. Fill the cavity with composite materials and cure for 40 seconds.

Group 3 (G3) and Control Group 4 (CG4):

1. Apply the 36% phosphoric acid gel (Conditioner 36) to the entire surface of the cavity, starting acid application on enamel, leaving undisturbed for 15 seconds, then covering the dentin preparation surfaces for an additional 15 seconds (total-etch technique). After thoroughly rinsing with water, gently dry the cavities with oil-free compressed air, taking care to avoid desiccation of the tooth substrate (moist bonding technique).
2. Apply the XPA to the entire surface of the cavity. This surface should remain undisturbed for 20 seconds. Spread the adhesive to an even film with a gentle stream of air and cure the adhesive for 10 seconds with the halogen curing light.
3. Fill the cavity with composite materials and cure for 40 seconds.

Group 2 (G2) and Control Group 3 (CG3):

1. Apply the primer (CBA Self-Etch Primer) to the entire surface of the cavity and massage over the entire area for 20 seconds. Spread the primer to an even film using a gentle stream of air.
2. Apply the bond (CBA bond) to the entire area of the cavity. Use a gentle stream of air until the bond is spread to an even film and cure the bond for 10 seconds with the halogen curing light.
3. Fill the cavity with composite materials and cure for 40 seconds.

All specimens were then stored in distilled water at 37°C for 24 hours and after this time, they were then dried superficially and the roots embedded in chemically activated acrylic (PMMA) resin, while the exposed crown and root structure was covered with two coats of nail varnish, leaving a 1 mm window around the cavity margins. The samples were then immersed in 1% basic fuchsin solution for 24 hours. After this, any surface-adhered dye was carefully rinsed away in tap water.

To measure the vertical extent of microleakage, the teeth were sectioned from buccal to lingual surface with a low speed diamond saw (Isomet<sup>l</sup>). The sections were examined on each side under stereo light microscope (TM-111<sup>k</sup> at x30 magnification. Dye penetration was measured on the gingival and occlusal margins, for a total of two measurements per cavity. As there were 10 cavities per group, 20 measurements were taken for each group. The degree of dye penetration was identified according to ISO specification 11 405:2003:<sup>24</sup>

- 0 = no leakage;
- 1 = leakage extending into the enamel part of the cavity wall;
- 2 = leakage extending into the dentin part of the cavity wall but not including the pulpal floor of the cavity;
- 3 = leakage including the pulpal floor of the cavity.

Each experiment was conducted at room temperature (23-24°C).

**Flexural modulus** - Five specimens of each resin composite were prepared for determination of flexural moduli after 24 hours storage in 37°C water, following the procedure outlined

Table 2. Average maximum polymerization stress, gel point and flexural modulus of the three composites.

Composite	Polymerization shrinkage (vol.%)	Polymerization stress (MPa)	Gel point (s)	Flexural modulus (GPa)
P90	1.20 (0.06) <sup>a</sup>	1.23 (0.08) <sup>c</sup>	6.4 (0.6) <sup>f</sup>	9.5 (0.8) <sup>j</sup>
AP-X	2.21 (0.12) <sup>b</sup>	4.25 (0.25) <sup>d</sup>	2.9 (0.4) <sup>e</sup>	16.3 (1.6) <sup>k</sup>
Quixfil	2.25 (0.16) <sup>b</sup>	5.11 (0.26) <sup>e</sup>	2.2 (0.3) <sup>i</sup>	16.9 (1.6) <sup>k</sup>

Same superscript letters in each column show mean values with no statistically significant differences ( $P > 0.05$ ).

Table 3. Microleakage scores and mean ranks of three composites.

Comparison	Scores					Mean ranks
	0	1	2	3	n	
Comparison A						
G1: P90/PSA	12	8	0	0	20	50.5 <sup>a</sup>
G2: AP-X/CBA	10	10	0	0	20	57.0 <sup>a</sup>
G3: Quixfil/XPA	0	14	5	1	20	103.4 <sup>b</sup>
Comparison B						
G1: P90/ PSA	12	8	0	0	20	50.5 <sup>a</sup>
CG1: AP-X/ PSA	11	9	0	0	20	53.8 <sup>a</sup>
CG2: Quixfil/ PSA	5	13	2	0	20	77.8 <sup>c</sup>
Comparison C						
G1: P90/ PSA	12	8	0	0	20	50.5 <sup>a</sup>
CG3: P90/CBA	6	13	1	0	20	72.3 <sup>c</sup>
CG4: P90/XPA	4	15	1	0	20	78.8 <sup>c</sup>

Same superscript letters in each column show mean values with no statistically significant differences ( $P > 0.05$ ).

in ISO specification 4049.<sup>25</sup> The specimens were prepared in molds (25 mm x 2 mm x 2 mm), placed on and covered with polyester foils, irradiated from both sides with five overlapping footprints of 40 seconds each with the halogen curing light (Elipar 2500) prior to immersion into the water bath. Immediately before testing, material flashes were removed and the specimen dimensions were determined with a micrometer screw (Type 293-561,<sup>k</sup> accuracy  $\pm 1 \mu\text{m}$ ). The samples were loaded by three-point bending in a universal testing machine (Model 3367<sup>8</sup>) at a crosshead speed of 1 mm/minute until fracture occurred. Each experiment was conducted at room temperature (23-24°C). The moduli (GPa) were calculated from the linear portion of the load/deflection curve using the equation:  $FM = l^3 F / 4wh^3 d$ , where FM is the flexural modulus,  $l$  is the length between the supports,  $F$  is the load applied,  $w$  is the width of the specimen,  $h$  is the thickness of the specimen and  $d$  is the deflection at load  $F$ .

**Statistical analysis** - Polymerization shrinkage, polymerization stress, gel point and flexural modulus were analyzed using one-way ANOVA/Tukey's test, with a significance level of 5%. For microleakage results, statistical analysis was performed using Kruskal-Wallis test at a 0.05 level of significance.

## Results

The mean values of polymerization stress, polymerization shrinkage, gel point and flexural modulus for each material are presented in Table 2. P90 demonstrated the lowest polymerization stress, polymerization shrinkage, flexural modulus and the latest gel point, while Quixfil showed the highest polymerization stress and the earliest gel point. AP-X and Quixfil exhibited statistically similar polymerization shrinkage and flexural modulus.

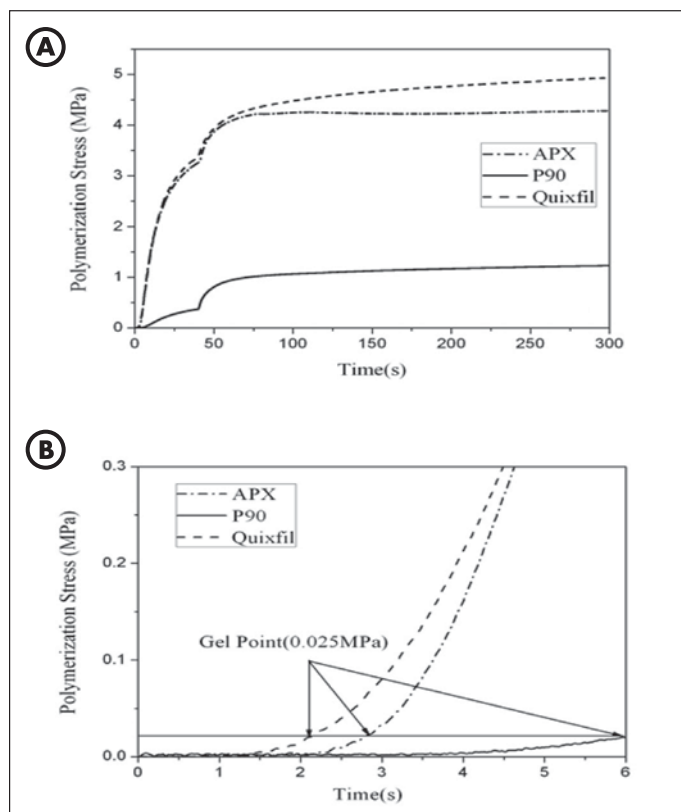


Fig. 3. A. Plot of polymerization stress versus time (0-300 seconds) for the materials tested and B. Plot of polymerization stress versus time during the first 6 seconds after the beginning of photoactivation. The time at which the force reaches 0.1 N (equal to 0.025 MPa in this experiment) is considered as an indirect measure of the gel point.

Plots of polymerization stress versus time are shown in Figs. 3A and B. The gel points of three composites are described in Fig. 3B.

Results of the microleakage test are shown in Table 3. Comparing different restorative systems (Comparison A), showed significantly less marginal gap formation for G1 (P90/PSA) and G2 (AP-X/CBA) than G3 (Quixfil/XBA). Data analysis of the performance of three composites in combination with PSA (Comparison B) revealed significantly less marginal gap formation for G1 (P90/PSA) and CG1 (AP-X/PSA) than CG2 (Quixfil/PSA). In Comparison C, G1 (P90/ PSA) demonstrated significantly lower microleakage scores than CG3 (P90/CBA) and CG 4 (P90/XBA).

## Discussion

The hypothesis cannot be rejected because, overall, the silorane-based composite (P90) presented the lowest polymerization shrinkage and stress and the longest time to gel point. The only exception was microleakage; P90 presented interfacial integrity similar to that shown by methacrylate based composite (AP-X) without a significant advantage, no matter using the same adhesive (PSA) or recommended adhesive (AP-X/CBA and P90/PSA).

The resin composites evaluated are commercial products possessing low polymerization shrinkage. The volume shrinkage value results ranged from low ( $< 2\%$ ) to medium ( $> 2\%$ ). P90 showed the lowest shrinkage values, a finding that is in agreement with previous studies on siloranes.<sup>9</sup> For the methacrylate resin composites, AP-X and Quixfil resulted in

similar polymerization shrinkage, possibly due to the similar filler volume fraction incorporated in the materials.

The development of polymerization stress in dental composites depends on material composition factors such as the type of monomer, type and amount of filler, and filler/matrix interactions, and composite polymerization factors such as the degree and rate of polymerization, and placement and curing technique.<sup>26</sup> Because the monomer composition of the resin matrix can greatly influence the gel point, rate of polymerization shrinkage and the curing efficiency of the composites,<sup>27-29</sup> the final resin matrix composition plays a key role in determining polymerization stress.

In contrast to the polymerization reaction of methacrylates, the ring-opening polymerization of silorane-based composite occurs *via* the cleavage and opening of ring-structures that gain space and counteract the inevitable loss of volume due to bond formation.<sup>30</sup> Observing an enlarged plot of the stress curves during the first 6 seconds of the test (Fig. 3), it was noted that P90 showed the longest time to gel point allowing for flow of material and stress relaxation. One hypothesis for this behavior is that the siloranes are slower to polymerize, resulting in a longer time to reach gel point. Recent studies with siloranes have demonstrated a polymerization reaction with a slow onset because of time needed for cation formation.<sup>31,32</sup> P90 presented the lowest polymerization shrinkage and the most time to gel point due to cationic ring-opening polymerization of the silorane monomers, which can explain their low polymerization stress.

According to Hooke's Law, in a totally elastic situation, stress should be determined by the product of the volumetric shrinkage and the elastic modulus (E-modulus) of the material. Though the setting of a dental composite is not a purely elastic situation, an increased E-modulus has been related to higher stress.<sup>26</sup> The present study found that the E-modulus of P90 was lower than that of AP-X and Quixfil due to its lower filler content. This observation may also explain the lower stress. However, stress build-up depends on the kinetics of the polymerization reaction, so correlating final flexural modulus and shrinkage values with polymerization stress values is a simplified approach. The changes in composite viscoelastic behavior that occur during polymerization, from predominantly viscous to mostly elastic, make polymerization stress development a quite complex event. The results of the present study indicated that similar polymerization shrinkage and flexural modulus of AP-X and Quixfil did not necessarily correspond to similar polymerization stress development. A possible reason for this is the gel point. Quixfil had a significantly lower time to reach gel point than AP-X (Fig. 3B) which meant the time for flow of material and stress relaxation was limited. The accelerator dimethylaminobenzoic acid ester in Quixfil seemed to be the reason for the earliest gel point.<sup>33</sup> Furthermore, earlier gel point might result in higher post-gel shrinkage which had a direct influence on stress development. In addition, a strange stress curve of Quixfil (Fig. 3A) was observed as polymerization stress development slowly plateaued to a constant, which agrees with a previous investigation of polymerization stress.<sup>19</sup> Maybe because it polymerized to a lesser degree in the light curing process and still kept relatively higher reaction rate after the light was turned off (likely related to its novel composition trimethacrylate.<sup>34,35</sup> It was supposed

that this strange stress curve was also a factor for the relatively higher stress of Quixfil.

In the microleakage test, G1 (P90/PSA) showed the lowest microleakage score among the seven groups, but its marginal integrity compared with G2 (AP-X/PSA) and CG1 (AP-X/CBA) was not better. In addition, the marginal integrity of CG3 (P90/CBA) and CG4 (P90/XBA) were significantly worse than G1 (P90/PSA). These phenomena showed that the lowest polymerization stress of P90 were not a guarantee of the best marginal integrity and cannot be extrapolated to predict marginal integrity. There are many other factors responsible for marginal integrity, for instance, adhesive system and stiffness of uncured restorative materials.

To obtain a true comparison between the individual composites, the same adhesive (PSA) was used to restore the cavities in Comparison B. The nonsignificant differences in microleakage of cavities restored with G1 and CG1 suggested that the PSA bond to cavities was sufficiently strong to resist the polymerization shrinkage stress induced by AP-X. This could be due to the higher viscosity of the filled, two-layered adhesive of PSA,<sup>36</sup> enabling a thick layer of adhesive to be placed on the tooth surface. This thick layer may have increased the strain capacity of the restoration.<sup>37-39</sup> Another possible explanation is that the uncured P90 is rather stiff compared with the relatively soft AP-X.<sup>10</sup> Therefore, close adaptation to the dentin surface in the narrow cavity may have been problematic. In some specimens of the microleakage test, air bubbles may have occurred at the composite-dentin interface, which may have led to poor marginal integrity. In the case of Quixfil, the microleakage result was significantly different. The highest polymerization stress of this composite can support this result. For the same reason mentioned above, because of the stiffness of uncured Quixfil it is possible to have an adverse impact on interface adaptation. All these factors may have led up to the poorest marginal integrity of Quixfil compared to the other materials tested.

In Comparison A, the rankings obtained were comparable to those of Comparison B, which can be supported by the thickness of the adhesive. Similar to PSA, CBA also contains microfillers in the bonding resin, and the thickness of its adhesive resin layer has been shown to range from 40-200  $\mu\text{m}$ .<sup>40</sup> On the other hand, XBA produced a thin film of unfilled adhesive at 13-38  $\mu\text{m}$ .<sup>41</sup> The thick adhesive resin layer of CBA was thus likely to absorb some of the shrinkage stress. Interestingly, Yapp<sup>42</sup> reported similar rankings in terms of tensile bond strength that LS System Adhesive/Filtek LS  $\approx$  SE Bond/APX >Xeno III/Quixfil.

The difference in Comparison C demonstrated that P90 must be applied with PSA to accomplish optimal restoration, as in the manufacturer's instructions. However, different from the need of P90 to be applied with PSA, PSA can also be combined with methacrylate-based composite, which was shown by the fact that PSA resulted in better marginal integrity than XBA and similar marginal integrity to CBA, when the same composite (AP-X and Quixfil) was used [G3 (Quixfil/PSA) better than CG2 (Quixfil/XBA) and G2 (AP-X/CBA) similar to CG1(AP-X/ PSA)].

However, as resin composites still undergo polymerization stress over time and damage of marginal sealing after water

storage,<sup>43</sup> long-term data are still necessary. In addition, the association of mechanical loading with thermal cycling may significantly increase leakage values.<sup>14</sup> Thus, further studies evaluating the influence of storage and mechanical loading on microleakage are required.

- a. 3M-ESPE, St Paul, MN, USA.
- b. 3M-ESPE, Seefeld, Germany.
- c. Ivoclar/Vivadent, Schaan, Liechtenstein.
- d. Kuraray, Tokyo, Japan.
- e. Dentsply DeTrey, Konstanz, Germany.
- f. Bisco Inc., Schaumburg IL, USA.
- g. Instron, Canton, MA, USA.
- h. SpofaDental, Jicin, Czech Republic.
- i. KG, Sorensen, Barueri, Brazil.
- j. Buehler, Lake Bluff, IL, USA.
- k. Mitutoyo, Kawasaki, Japan.

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