Flow, Strength, Stiffness and Radiopacity of Flowable Resin Composites

(Fluage, résistance, rigidité et radio-opacité des résines composites à faible viscosité)

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Sommaire

Objectif : *Cette étude avait pour but de caractériser 9 résines composites à faible viscosité de marque déposée, actuellement offertes sur le marché, quant à leur fluage, leur résistance à la flexion, leur rigidité (module d'élasticité) et leur radio-opacité.*

Méthodologie : *Sept résines composites à faible viscosité (Aelite Flo, Filtek Flow, Heliomolar Flow, PermaFlo, Revolution Formula 2, Tetric Flow, Wave) et 2 compomères à faible viscosité (Compoglass Flow, Dyract Flow) ont été évalués. Un composite hybride universel (Filtek Z250) et un compomère de restauration (Dyract AP) ont été utilisés comme témoins. Des essais mécaniques standards sur des spécimens en forme de barre de 25 × 2 × 2 mm ont été effectués après 24 heures et après 1 mois. Les essais de fluage ont été réalisés à l'aide d'un volume fixe de matériau soumis à une charge constante, et la radio-opacité a été mesurée simultanément pour tous les matériaux, sur des spécimens circulaires de 1 mm d'épaisseur.*

Résultats : *Comme prévu, les composites à faible viscosité ont présenté un indice de fluage plus élevé et des propriétés mécaniques moindres que les témoins. Ainsi, le module d'élasticité des composites a été d'au moins 50 % inférieur à celui des matériaux témoins, ce qui indique une grande flexibilité. La résistance à la flexion a été comparable à celle des composites témoins, mais les propriétés de fluage ont varié considérablement. Le matériau Tetric Flow est celui qui a présenté la plus grande radio-opacité, supérieure à celle de l'émail et du composite témoin, tandis que les matériaux Wave et Revolution Formula 2 ont obtenu la plus faible radio-opacité, laquelle s'est révélée inférieure ou égale à celle de la dentine.*

Conclusions : *Les matériaux à faible viscosité offrent un large éventail de propriétés mécaniques et physiques. Cependant, leurs propriétés mécaniques moindres portent à croire que ces matériaux ne devraient pas être utilisés en épaisseur importante aux endroits exposés à une forte charge occlusale. De plus, à l'intérieur des restaurations intracoronaires, il est recommandé d'utiliser des matériaux à radio-opacité élevée. Les matériaux varient considérablement quant à leurs propriétés de fluidité, et les applications cliniques et le rendement de ces matériaux devront être étudiés plus en profondeur.*

Mots clés MeSH : *comparative study; composite resins/chemistry; pliability; stress, mechanical*

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Resin composite formulations with greater fluidity have been introduced to the marketplace in recent years. These "flowable" materials (**Fig. 1**) have either less filler loading or a greater proportion of diluent monomers in the composite formulation. They are purported to offer higher flow, better adaptation to the internal cavity wall, easier insertion and greater elasticity than previously available products.[1] Dentists can now choose from composites with a wide range of viscosities for different clinical applications, from the most fluid resin fissure sealants through flowable, microfill and hybrid composite formulations, to the high-viscosity packable posterior composites. Each category has certain advantages and limitations, the universal hybrid composites providing the best general blend of good material properties and clinical performance for

Figure 1: *Proprietary flowable composites.*

Table 1 Materials tested

Test material	Manufacturer and location
Control materials	
Filtek Z250 (universal hybrid composite)	3M ESPE Dental Products St. Paul, Minnesota
Dyract AP (restorative compomer)	Dentsply Konstanz, Germany
Flowable materials	
Aelite Flo	Bisco Inc. Schaumburg, Illinois
Compoglass Flow	Vivadent Schaan, Liechtenstein
Dyract Flow	Dentsply Konstanz, Germany
Filtek Flow	3M ESPE Dental Products St. Paul, Minnesota
Heliomolar Flow	Vivadent Schaan, Liechtenstein
PermaFlo	Ultradent Products Inc. Jordan, Utah
Revolution Formula 2	Kerr Corporation Orange, California
Tetric Flow	Vivadent Schaan, Liechtenstein
Wave	Southern Dental Industries Cologne, Germany

routine anterior and posterior restorations.[2] The proprietary materials within each category vary considerably in handling, physical and mechanical properties.

Bayne and others[3] characterized earlier flowable formulations. The filler content was found to be 20% to 25% less than that of the universal composite materials, which demonstrated better performance for all mechanical properties tested. A wide range of values was demonstrated by the flowable composites, and it was advised that they be restricted to low-stress clinical situations. Handling properties also varied widely. Some materials did not flow any more than the universal composite controls, and for others, flow was difficult to control.

Low-modulus flowable resin composites have been described as potentially radiopaque “filled adhesives” with implications for improved clinical dentin bonding.[4] In contrast, restorative composites have a relatively high modulus of elasticity, and it has been suggested that this high stiffness contributes to their inability to compensate for contraction stress during polymerization. This can lead to either bond failure or fracture of the tooth structure, resulting in microleakage and postoperative sensitivity. Employing an intermediate layer of low-modulus composites can relieve some of the contraction stress during polymerization.[5,6] Application of increased thickness of low-stiffness adhesive has a similar effect.[7] Use of flowable composites in conjunction with the very high viscosity, high-modulus packable composites is a common clinical technique. However, the effects of the higher-than-expected polymerization shrinkage of the flowable material (because of lower filler loading) and the effects of possible flexure of the restoration when it is supported by the lower-modulus flowable “liner” are unknown.[3]

A prerequisite of all restorative materials is adequate radiopacity to allow the clinician to evaluate restoration integrity at subsequent recall appointments, distinguish caries from restorative material on radiographs, and detect voids, overhangs and open margins. A restorative material with radiopacity slightly greater than, or equal to, enamel is ideal for detection of secondary caries.[8] It is widely recognized that unfilled resin adhesives are radiolucent and that use of thick layers of such materials can present a diagnostic challenge on subsequent radiography. It has been the authors’ personal observation that the radiopacity of some of the flowable composite resin materials used beneath posterior restorations is lower than desirable.

Many dentists have readily accepted flowable composites for a wide variety of uses. Although some in vitro studies have shown that use of flowable composites reduces restoration microleakage and the occurrence of voids,[1,9,10] other research has shown no apparent advantage over universal hybrid composites.[11–13] Despite limited scientific information, flowable composite materials are being used for a wide range of applications, from liners and pit and fissure sealants, to margin or void repairs and even Class I and V restorations. Newer formulations that have recently been introduced to the market include flowable compomers or polyacid-modified resin composites. Because flowable materials are being used in many clinical applications, dentists need comparative information so that they can select the materials with the most appropriate properties for any particular use.

The purpose of this study was to determine the key properties of flow, flexural strength, modulus of elasticity and radiopacity of 7 currently available flowable composites and 2 flowable compomer materials. One universal composite and one compomer were included in the study as controls.

Methods and Materials

The materials used in this study are listed in **Table 1**.

Flow

Flow measurements for each of the 11 materials were carried out using a method similar to that of Bayne and others,[3] who employed a modification of the American Dental Association flow test for dental cements. A disposable 1-mL B-D syringe (Becton Dickinson & Co., Franklin Lakes, New Jersey) without a needle tip was filled with the test material, and a standard volume (0.5 mL) was extruded onto a glass plate and immediately covered by 3 stacked glass slides (weighing a total of 18 g). After 30 seconds the samples were transferred to a Triad curing unit (Triad 2000, Dentsply, York, Pennsylvania) and cured for 60 seconds. The diameter (in millimetres) of the resulting nearly circular disk was measured twice (along perpendicular lines). For each material, the average diameter of 3 disks was used to generate comparative flow results.

Flexural Strength

Flexural strength was determined according to International Organization for Standardization (ISO) standard 4049.[14] Sixteen rectangular bar specimens of each material, measuring 25 × 2 × 2 mm, were prepared by filling a Teflon mould, placing and clamping a glass lid over the mould, and curing for 60 seconds in the Triad light curing unit. After polymerization, 8 bar specimens of each material were stored in distilled water at 37°C for 24 hours and 8 were stored under the same conditions for 1 month. Before loading, height and width were measured at 3 locations on each specimen; a digital micrometer (Digimatic, Mitutoyo Corp., Tokyo, Japan) with an accuracy of 0.01 mm was used for these measurements. The mean of these 3 measurements was used to calculate flexural strength and modulus of elasticity.

Each specimen was subjected to a 3-point bend test on an Instron uniaxial servo-mechanical testing machine (model 4301, Instron Corporation, Canton, Massachusetts) at a crosshead speed of 1 mm/minute, with loading until failure. Testing was conducted with each specimen immersed in a 37°C water bath to simulate clinical conditions. The maximum load recorded at the time of failure was captured electronically. Flexural strength (F, in megapascals) was calculated using the following formula:

$$F = 3P_fL/2WH^2$$

where P_f is the maximum measured load at failure (in newtons), L is the distance between the supports (fixed at 20 mm), W is the mean width of the specimen measured before testing (in millimetres), and H is the mean height of the specimen between the tension and compression surfaces (also in millimetres).

Modulus of Elasticity

The elastic modulus (E, in gigapascals) was determined from the slope of the load deflection curve generated during the 3-point bend test according to the following formula:

$$E = \delta F/\delta Y \times L^3/4WH^3$$

where $\delta F/\delta Y$ is the change in force (δF) per unit change in deflection (δY) of the centre of the specimen, L is the distance between the supports on the tension surface (20 mm), W is the width of the specimen (in millimetres) and H is the thickness of the specimen between the tension and compression surfaces (also in millimetres). The slope, in newtons per millimetre, was measured in the initial straight-line portion of the load deflection graph.

Radiopacity

For each material, a split-ring metal mould and clamps were used to produce 5 disk specimens measuring 6.0 mm in diameter and 1.0 mm in thickness, in accordance with ISO 4049.[14] The specimens were photo-polymerized as before and ground through 400-grit sandpaper to create a flat surface. Specimens were measured after finishing to verify the critical tolerance of 1.0 ± 0.01 mm. Five longitudinal sections of human permanent molar teeth were also prepared to the same thickness using an Accutom saw (Struers Co., Copenhagen, Denmark). One specimen of each material, one tooth section and a standard proprietary aluminum step wedge were positioned side by side on occlusal radiographic film (Ekta Speed Plus, EO-42P, Kodak Canada Inc., Toronto, Ontario). The wedge's maximum thickness was 13.5 mm and the step size was 1 mm. The step wedge served as an internal standard for each radiographic exposure and allowed calculation of the radiopacity of each material in terms of aluminum thickness. Films were exposed for 0.37 milliseconds with a dental radiography unit (Belmont-Takara Phot-X 2001 CP, Belmont Takara Co., Frankfurt, Germany) at 70 kV and 10 mA; the object-to-film distance was 400 mm. The films were processed in a standard automatic processor (Dent-X, Elmsford, New York).

The optical density of the radiographic images was measured with a transmission densitometer (Macbeth TD-504, Macbeth Corp., Newsburgh, New York) (mean of at least 4 readings per specimen). Following the method of El-Mowafy and Benmergui[15] the optical density data for the aluminum steps were entered into a computer, and the best possible exponential fit was used for curves of aluminum optical density. Aluminum equivalency for each sample of test material and each dentin and enamel section was extrapolated directly from the graph and used to calculate mean aluminum thickness equivalency.

Statistical Analysis

One-way analysis of variance (ANOVA) and Duncan's multiple-range statistical tests ($p < 0.05$) were conducted on all physical and mechanical test results.

Results

Flow

The flow of behaviour of all of the flowable composites was distinct from that of the universal hybrid composite and compomer restorative materials (**Fig. 2**). The proprietary flowable materials demonstrated a wide range of flow behaviour, with the most fluid (PermaFlo) providing almost twice the flow of the least fluid (Aelite Flo) under the same conditions.

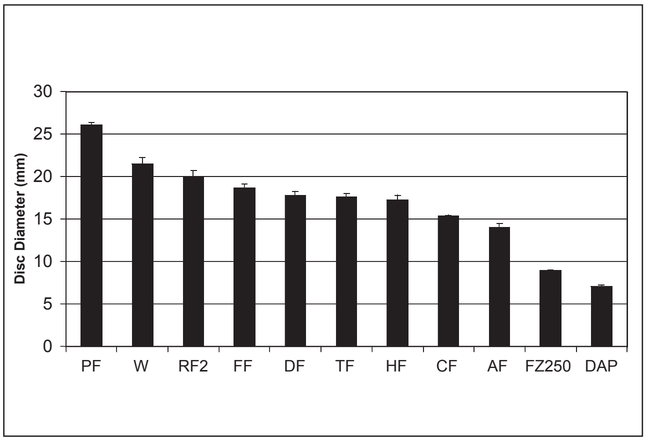

Figure 2: *Flow (disk diameter [in mm]) of flowable composites. PF = PermaFlo, W = Wave, RF2 = Revolution Formula 2, FF = Filtek Flow; DF = Dyract Flow, TF = Tetric Flow, HF = Heliomolar Flow, CF = Compoglass Flow, AF = Aelite Flo, FZ250 = Filtek Z250, DAP = Dyract AP.*

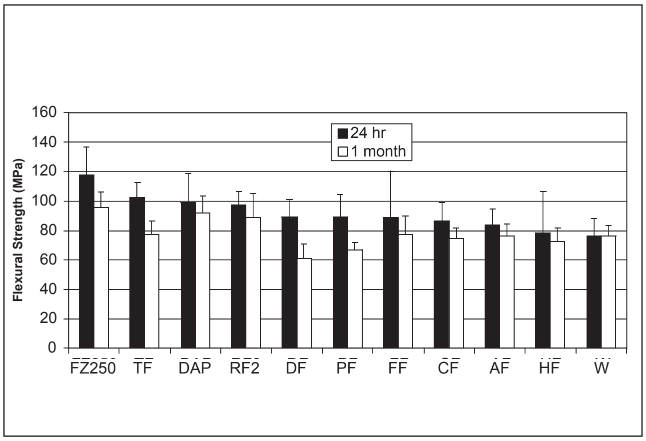

Figure 3: *Flexural strength (in megapascals) of flowable composites. FZ250 = Filtek Z250, TF = Tetric Flow, DAP = Dyract AP, RF2 = Revolution Formula 2, DF = Dyract Flow, PF = PermaFlo, FF = Filtek Flow, CF = Compoglass Flow, AF = Aelite Flo, HF = Heliomolar Flow, W = Wave.*

Table 2a Flexural strength at 24 hours[a]

Test material	Mean flexural strength ± SD (MPa)
Filtek Z250	117.4 ± 19.2
Tetric Flow	102.0 ± 10.6
Dyract AP	98.9 ± 19.9
Revolution Formula 2	97.4 ± 9.2
Dyract Flow	89.4 ± 12.0
PermaFlo	88.9 ± 15.6
Filtek Flow	88.7 ± 31.3
Compoglass Flow	86.5 ± 12.3
Aelite Flo	83.7 ± 10.7
Heliomolar Flow	78.1 ± 28.5
Wave	66.9 ± 12.0

SD = standard deviation.

[a] *Vertical lines indicate values that are not significantly different from one another (p < 0.05).*

Table 2b Flexural strength at 1 month[a]

Test material	Mean flexural strength ± SD (MPa)
Filtek Z250	95.6 ± 10.6
Dyract AP	91.8 ± 11.7
Revolution Formula 2	88.7 ± 16.3
Filtek Flow	77.4 ± 28.5
Tetric Flow	77.0 ± 9.3
Aelite Flo	76.3 ± 7.9
Wave	76.3 ± 6.9
Compoglass Flow	74.7 ± 7.1
Heliomolar Flow	72.5 ± 9.2
PermaFlo	67.2 ± 4.6
Dyract Flow	61.1 ± 9.5

SD = standard deviation.

[a] *Vertical lines indicate values that are not significantly different from one another (p < 0.05).*

Flexural Strength

The control composite had the highest mean value for flexural strength at 24 hours (117.4 MPa) and 1 month (95.6 MPa) (**Tables 2a** and **2b**, **Fig. 3**). Flexural strength values for the flowable composites varied from 66.9–102 MPa at 24 hours and from 61.1–88.7 MPa at 1 month. For most materials, flexural strength was significantly lower at 1 month than at 24 hours. There was a significant interaction between material and time ($F = 6.667$, $p < 0.05$).

Modulus of Elasticity

The modulus of elasticity at 24 hours and 1 month were highest for the control composite and the control compomer (**Tables 3a** and **3b**, **Fig. 4**). The moduli for the flowable materials were approximately one-third to one-half that of the control composite. For all materials there was a significant interaction between material and time ($F = 73.775$, $p < 0.05$).

Radiopacity

Tetric Flow had the highest radiopacity (**Fig. 5**), above that of both enamel and the control composite. Both Wave and Revolution Formula 2 showed lowest radiopacity, below or equivalent to that of the dentin. All materials met the ISO minimum standard of radiopacity, equal to or greater than that of the same thickness of aluminum.

Discussion

Mechanical and radiopacity tests performed according to ISO test methods[14] for resin-based restorative materials provided basic comparative information for the test materials. The ISO has set 80 MPa as the minimum flexural strength for polymer-based filling and restorative materials claimed suitable for restorations involving outer occlusal surfaces. All of the flowable materials tested exceeded or came very close to fulfilling this requirement. Although Bayne and others[3] used biaxial flexural strength tests, comparison of their results with those for the newer materials tested here showed a general trend toward an increase in flexural strength, to a level more comparable to that of the control composite. However, despite achievement of minimum ISO flexural strength requirements,

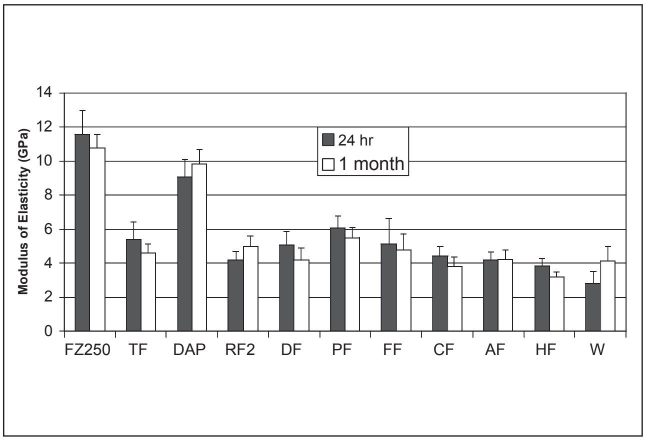

Figure 4: *Modulus of elasticity (GPa) of flowable composites. FZ250 = Filtek Z250, TF = Tetric Flow, DAP = Dyract AP, RF2 = Revolution Formula 2, DF = Dyract Flow, PF = PermaFlo, FF = Filtek Flow, CF = Compoglass Flow, AF = Aelite Flo, HF = Heliomolar Flow, W = Wave.*

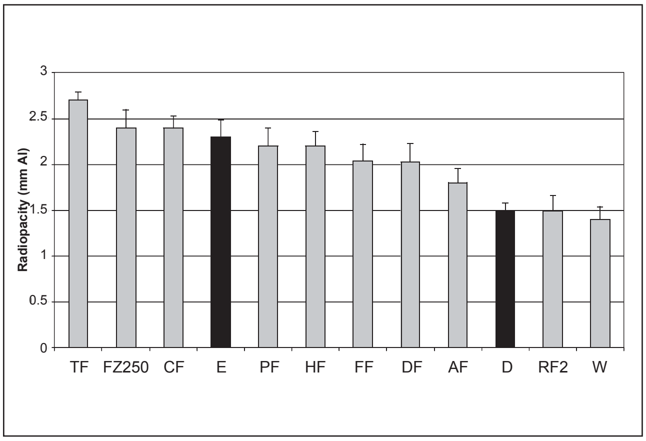

Figure 5: *Radiopacity of flowable composites. Data are indicated as aluminum thickness equivalents. TF = Tetric Flow, FZ250 = Filtek Z250, CF = Compoglass Flow, E = enamel, PF = PermaFlo, HF = Heliomolar Flow, FF = Filtek Flow, DF = Dyract Flow, AF = Aelite Flo, D = dentin, RF2 = Revolution Formula 2, W = Wave.*

Table 3a Modulus of elasticity at 24 hours[a]

Test material	Mean modulus of elasticity ± SD (GPa)
Filtek Z250	11.6 ± 1.4
Dyract AP	9.1 ± 1.0
PermaFlo	6.0 ± 0.7
Tetric Flow	5.4 ± 1.1
Filtek Flow	5.1 ± 1.5
Dyract Flow	5.1 ± 0.8
Compoglass Flow	4.4 ± 0.6
Revolution Formula 2	4.2 ± 0.5
Aelite Flo	4.2 ± 0.4
Heliomolar Flow	3.9 ± 0.4
Wave	2.8 ± 0.7

SD = standard deviation.
[a]*Vertical lines indicate values that are not significantly different from one another* ($p < 0.05$).

Table 3b Modulus of elasticity at 1 month[a]

Test material	Mean modulus of elasticity ± SD (GPa)
Filtek Z250	10.8 ± 0.8
Dyract AP	9.8 ± 0.9
PermaFlo	5.5 ± 0.6
Revolution Formula 2	5.0 ± 0.6
Filtek Flow	4.8 ± 0.9
Tetric Flow	4.6 ± 0.6
Aelite Flo	4.2 ± 0.6
Dyract Flow	4.2 ± 0.7
Wave	4.1 ± 0.9
Compoglass Flow	3.8 ± 0.6
Heliomolar Flow	3.2 ± 0.3

SD = standard deviation.
[a]*Vertical lines indicate values that are not significantly different from one another* ($p < 0.05$).

it is still recommended that flowable materials be restricted to minimal or adjunctive clinical situations.

The properties of resin composites depend primarily on the material composition. A correlation exists between filler content and mechanical properties, particularly for modulus of elasticity:[16] the higher the filler content, the higher the modulus and the greater the resistance to deformation. Conversely, the lower the filler content, the greater the polymerization shrinkage and the lower the ability to resist deformation. The best compromise for most restorations appears to be a fine hybrid resin composite with a flexural modulus of approximately 10 GPa.[4] In this study the flowable materials as a group consistently exhibited markedly lower stiffness than the hybrid composite and compomer restorative materials. This suggests that flowable materials (elastic modulus at 24 hours ranging from 2.8 to 6.0 GPa) are not sufficiently rigid to withstand significant occlusal forces when used in bulk. In addition, the literature is equivocal regarding the effectiveness of low-modulus flowable composites in reducing microleakage.[11–13,17] However, use of flowable composites did not increase microleakage in any study, and there were favourable effects on reducing microleakage in some studies.[1,9,18] It has been suggested that these favourable effects are due to the improved cavity adaptation and stress-absorbing ability outweighing the effects of increased polymerization shrinkage.[18] Thicker layers appear to be more effective.[9,10] The causes of and preventive measures for microleakage are multifactorial and complex; these factors will confound the results of in vitro leakage studies of resin composites, in which leakage tends to be the rule rather than the exception. Using only a composition-based approach to relieve shrinkage stress may be too simplistic. Ensuring optimal dentin bonding and using incremental layers of composite in appropriate configurations are key factors in minimizing the deleterious effects of polymerization shrinkage.

Radiopacity is an essential property for all restorative materials, and ISO standards stipulate that minimum radiopacity be equal to or greater than that of an equivalent thickness of aluminum (1 mm in this study). Although the radiopacity of dentin and enamel specimens varies, pure aluminum provides a constant value. All of the flowable materials tested in this study would pass the ISO minimum standards; however, the radiopacity of 2 of the products, Revolution Formula 2 and Wave, was comparable to or less than that of the dentin samples. The highest radiopacity was exhibited by Tetric Flow, which had radiopacity higher than that of both enamel and the control composite. Studies have emphasized the desirability of high radiopacity in flowable composites that may be used beneath posterior composite restorations.[19,20]

Flowable composites may offer significant advantages when used as intermediate layers, according to the concept of radiopaque filled adhesives. They can also be used to improve adaptation to the cavity surface in areas that are difficult to access, especially when high-viscosity posterior composite materials are used subsequently. The proprietary materials tested here offer a wide range of properties reflecting the fact that optimal physical, mechanical and handling properties have not been defined for this group of materials. The optimal material would provide controlled fluidity combined with optimal radiopacity and high strength. The diversity among the various proprietary flowable composite materials indicates that the clinician should consider the anticipated clinical use and select the material with the most appropriate properties. Further studies are required to ascertain the potential clinical benefits and limitations of this class of flowable materials.

Conclusions

The flowable composite and compomer materials tested had greater fluidity and lower rigidity than the universal composite or compomer materials. Although disparate in terms of relative flow and radiopacity, the flowable materials had similar strength parameters, with approximately 50% of the rigidity of regular composites and approximately 80% of the flexural strength. Their lower mechanical properties suggest that these materials should not be used in bulk in areas that experience high occlusal loading. For intracoronal restorations, clinicians are advised to use materials with high radiopacity. Use of materials with radiopacity close to or less than dentin may result in future diagnostic challenges.

The clinical applications and performance of these materials require further study. ❖

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