

Viscoelastic Properties of Contemporary Bulk-Fill Restoratives: A Dynamic-Mechanical Analysis

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Clinical Relevance

Viscoelastic properties of bulk-fill restoratives varied between materials and were environment dependent. Resin-coating of reinforced bulk-fill glass ionomers does not positively influence elastic properties.

SUMMARY

This study investigated the viscoelastic properties of contemporary bulk-fill restoratives in distilled water and artificial saliva using dynamic mechanical analysis. The materials eval-

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DOI: 10.2341/16-365-L

uated included a conventional composite (Filtek Z350), two bulk-fill composites (Filtek Bulk-fill and Tetric N Ceram), a bulk-fill giomer (Beautiful-Bulk Restorative), and two novel reinforced glass ionomer cements (Zirconomer [ZR] and Equia Forte [EQ]). The glass ionomer materials were also assessed with and without resin coating (Equia Forte Coat). Test specimens $12 \times 2 \times 2$ mm of the various materials were fabricated using customized stainless-steel molds. After light polymerization/initial set, the specimens were removed from the molds, finished, measured, and conditioned in distilled water or artificial saliva at 37°C for seven days. The materials ($n=10$) were then subjected to dynamic mechanical testing in flexure mode at 37°C and a frequency of 0.1 to 10 Hz. Storage modulus, loss modulus, and loss tangent data were subjected to normality testing and statistical analysis using one-way analysis of variance/Dunnett's test and *t*-test at a significance level of $p < 0.05$. Mean storage modulus ranged from 3.16 ± 0.25 to 8.98 ± 0.44 GPa, while mean loss modulus ranged from 0.24 ± 0.03 to 0.65 ± 0.12 GPa for distilled water and artificial saliva. Values for loss

tangent ranged from 45.7 ± 7.33 to 134.2 ± 12.36 (10^{-3}). Significant differences in storage/loss modulus and loss tangent were observed between the various bulk-fill restoratives and two conditioning mediums. Storage modulus was significantly improved when EQ and ZR was not coated with resin.

INTRODUCTION

Due to the declining popularity of amalgam, the pursuit of "tooth-colored alternatives" has intensified over the past few years.¹ Composite resins, glass ionomer cements, and hybrids of these materials are constantly being enhanced to improve their clinical handling and performance.^{2,3} Innovative bulk-fill composites were introduced to address the need for incremental material placement arising from limited depth of cure and polymerization shrinkage associated with conventional composites.⁴ The incremental technique also has several disadvantages, including the incorporation of voids or contamination between layers and placement difficulty in cavities with limited access, and is clinically time consuming to perform. Bulk-fill composites can be placed in increments of 4 mm and are reported to possess enhanced curing and controlled shrinkage.⁵ The early moisture sensitivity and low physicomaterial properties of glass ionomer cements had been alleviated by fast-setting, highly viscous and reinforced glass ionomers.³ Collectively, bulk-fill composite and glass ionomer restoratives simplify clinical procedures and reduce technique sensitivity, chair time, and stress for both dentists and patients, especially when multiple posterior restorations are required.

Posterior direct tooth-colored restorative material should have adequate strength to resist masticatory and occlusal forces. Tooth-colored restoratives were traditionally evaluated using destructive static compression, tension, or flexure tests. These tests, however, emphasize the only elastic component of materials and provide single-event strength values.⁶ Dynamic methods are now commonly employed to assess mechanical properties of viscoelastic materials in materials science. Dynamic mechanical analysis (DMA) is particularly well suited for viscoelastic materials, such as composites and glass ionomers, as it can determine both elastic and viscous responses of materials.⁷ The test is also able to mimic cyclic masticatory loading that materials are subjected to intraorally.⁸ The nondestructive nature of this test allows for the reexamination of specimens after being subjected to different treatments. In addition,

a wide range of frequency, temperature, and/or amplitude variations is admissible with DMA. DMA and other dynamic tests are superior to static tests, as they provide greater sensitivity to both macroscopic and molecular relaxation.⁹

The physical properties of tooth-colored restorations are affected by their surrounding chemical environment.¹⁰ Direct tooth-colored restoratives have been shown to leach filler and other constituents when stored in distilled water.¹¹ As direct tooth-colored restoratives are constantly being surrounded by saliva, findings obtained from storage in distilled water may be of little clinical relevance.¹² The use of artificial saliva allows for better simulation of the way restoratives interact with human saliva.¹³ Leaching of restorative constituents has been reported to be higher in artificial saliva when compared to distilled water.¹⁴

Studies investigating the viscoelastic properties of bulk-fill tooth-colored restoratives using DMA in different conditioning mediums are still lacking. In addition, no research had been done on novel bulk-fill giomer and reinforced glass ionomer restoratives. Giomers, also known as PRG composites, are based on prereacted glass ionomer (PRG) technology in which acid-reactive fluoride-containing glass is reacted with polyacids in the presence of water, freeze-dried, milled, silanized, ground, and used as fillers. Besides fluoride release and tooth demineralization inhibition, giomers also possess antiplaque formation properties.¹⁵⁻¹⁸ Zircomer and Equia Forte are two recently introduced bulk-fill reinforced highly viscous glass ionomer cements. While Zircomer is reinforced with nanozirconia fillers, Equia Forte is reinforced with ultrafine, highly reactive glass particles forming a glass "hybrid" restorative. Together with the application of a multifunctional monomer layer, "microlaminate" restorations with improved physical and esthetic properties are achieved. A prospective six-year clinical trial using the "microlamination" technique proved the reliability of this restorative approach.¹⁹ Both bulk-fill reinforced glass ionomers had been promoted as amalgam alternatives.

The objectives of this study were to compare the viscoelastic properties of contemporary bulk-fill restorative materials. Variations in storage and loss modulus as well as loss tangent after conditioning in distilled water and artificial saliva were also compared. For the reinforced glass ionomer cements, the effects of resin coating on viscoelastic properties were also evaluated. The null hypotheses were that there were no difference in viscoelastic behavior

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Table 1: *Technical Profiles and Manufacturers of the Materials Evaluated*

Material (Abbreviation)	Manufacturer	Type and Method of Curing	Resin/Liquid	Filler/Powder	Filler Content % by Weight/ % by Volume
Filtek Z350 (ZT)	3M ESPE (St Paul, MN, USA)	Nanohybrid composite (light cured)	Bis-GMA Bis-EMA UDMA TEGDMA	Zirconia/silica cluster, silica nanoparticle	78.5/63.3
Filtek Bulk-Fill (FB)	3M ESPE	Bulk-fill composite (light cured)	Bis-GMA Bis-EMA UDMA Proctylat resins	Zirconia/silica cluster, ytterbium trifluoride	76.5/58.4
Tetric N Ceram Bulk-Fill (TC)	Ivoclar, Vivadent Inc (Amherst, NY, USA)	Bulk-fill composite (light cured)	Bis-GMA Bis-EMA UDMA	Barium glass filler, ytterbium fluoride, spherical mixed oxide	77/55
Beautifil-Bulk Restorative (BB)	Shofu Inc (Kyoto, Japan)	Bulk-fill giomer (light cured)	Bis-GMA UDMA Bis-MPEPP TEGDMA	S-PRG based on F-Br-Al-Si glass	87/74.5
Zirconomer (ZR)	Shofu	Zirconia/reinforced glass ionomer (chemically cured)	Polyacrylic acid solution, tartaric acid	Fluoroaluminosilicate glass, zirconia oxide, pigments, others	Not applicable
GC Equia Forte (EQ)	GC Industrial Co (Tokyo, Japan)	Bulk-fill glass ionomer (chemically cured)	—	Fluoroaluminosilicate glass, polyacrylic acid powder, surface-treated glass	Not applicable
GC Equia Forte Coat (C)	GC Industrial	Nanofilled resin (light cured)	—	Nanofiller	Not available

Abbreviations: Bis-EMA, ethoxylated bisphenol-A-glycidyl methacrylate; Bis-GMA, bisphenol-A glycidyl methacrylate; Bis-MPEPP, bisphenol-A polyethoxy-dimethacrylate; S-PRG, surface-modified prereacted glass; TEGDMA, triethylene glycol dimethacrylate; UDMA, urethane dimethacrylate.

between the various restoratives, conditioning in distilled water, and artificial saliva as well as between resin and non-resin-coated glass ionomers.

METHODS AND MATERIALS

Materials selected for this study included a conventional composite (Filtek Z350 [ZT]), two bulk-fill composites (Filtek Bulk-fill [FB] and Tetric N Ceram [TC]), a bulk-fill giomer (Beautifil-Bulk Restorative [BB]), and two reinforced bulk-fill glass ionomer restoratives (Zirconomer [ZR] and Equia Forte [EQ]). The glass ionomer materials were also assessed with and without a nanofilled resin coating (Equia Forte Coat [C]). Details of the materials used and their technical profiles are shown in Table 1. Test specimens 12 × 2 × 2 mm of the various materials were fabricated using customized stainless-steel molds. The materials were mixed according to the manufacturers’ instructions where applicable and placed in a single increment into the molds. Excess material was removed by compressing the molds between two Mylar strips with glass slides. The top

and bottom surfaces of composite and giomer specimens were subsequently light polymerized with two overlapping irradiation cycles of 10 seconds each using an LED curing light (Demi Plus, Kerr Corp, Orange, CA, USA) with an irradiance of 1330 mW/cm². These restoratives were light polymerized for an additional 10 seconds after removal from their molds. For the glass ionomers, specimens were allowed to set for five minutes before removal from their molds. The specimens were carefully finished using fine contouring/polishing discs (Sof-Lex, 3M ESPE, St Paul, MN, USA). For the resin-coated glass ionomer groups, Equia Forte Coat was applied to the test specimens on all four surfaces and light polymerized in two overlapping irradiation cycles of 10 seconds per surface. All specimens were subsequently measured with a digital caliper (Mitutoyo Corporation, Kawasaki, Japan) to ensure standardized specimens with parallel opposing surfaces.

The specimens were then randomly divided into two groups (n=10) and conditioned in either distilled water or artificial saliva at 37°C for seven days.

Table 2: *Composition of the SAGF Medium*

Components	Concentration (mg L ⁻¹)
NaCl	125.6
KCl	963.9
KSCN	189.2
KH ₂ PO ₄	654.5
Urea	200.0
NaSO ₄ •10H ₂ O	763.2
NH ₄ Cl	178.0
CaCl ₂ •2H ₂ O	227.8
NaHCO ₃	630.8

Composition of the artificial saliva used (SAGF medium²⁰) is shown in Table 2. The pH of the artificial saliva was checked with a digital pH meter (pH 2700, Eutech, Singapore) and adjusted to 6.8. Both conditioning mediums were replaced every two days to minimize changes in pH over time. Specimens were subjected to dynamic mechanical testing (DMA RSA-G2, TA Instruments, New Castle, DE, USA) in distilled water or artificial saliva and flexure three-point bending mode at 37°C with a frequency of 0.1 to 10 Hz. The distance between the supports was fixed at 10 mm, and an axial load of 5 N was employed. Storage modulus, loss modulus, and loss tangent values were obtained for the various bulk-fill restoratives.

Statistical analysis was performed with the SPSS software (version 12.0.1, SPSS Inc, Chicago, IL, USA). Data were checked for normality using the Kolmogorov-Smirnov and Shapiro-Wilk test. Comparisons between materials were performed using one-way analysis of variance and Dunnett's test, while the effects of conditioning medium and resin-coating was appraised using an independent sample *t*-test at a significance level $\alpha = 0.05$.

RESULTS

Mean storage modulus, loss modulus, and loss tangent for the various materials and mediums are shown in Tables 3 through 5. Data were found to be normal, and parametric data analysis was permissible. One-way analysis of variance indicated significant differences in viscoelastic behaviors between bulk-fill materials in both distilled water and artificial saliva. Mean storage modulus ranged from 3.19 ± 0.30 to 7.44 ± 0.28 GPa in distilled water and 3.16 ± 0.25 to 8.98 ± 0.44 GPa in artificial saliva (Table 3). For both mediums, the highest storage modulus was observed with EQ and the lowest with ZRC. With the exception of EQ, storage modulus of

Table 3: *Mean Storage Modulus Values (GPa) of the Various Materials (Standard Deviations in Parentheses)^a*

Materials (Code)	Distilled Water	Artificial Saliva
Filtek ZT (ZT)	5.76 (0.42) A	5.48 (0.57) A
Filtek Bulk-Fill (FB) ^p	5.48 (0.45) AB	6.17 (0.70) A
Tetric N Ceram (TC) ^p	4.77 (0.54) BC	3.63 (0.37) B
Beautiful (BB) ^p	5.27 (0.62) AC	5.91 (0.56) A
Equia Forte without resin coat (EQ) ^p	7.44 (0.28)	8.98 (0.44)
Equia Forte with resin coat (EQC) ^p	4.53 (0.16) c	4.16 (0.27)
Zirconomer without resin coat (ZR)	3.75 (0.36)	3.60 (0.35) B
Zirconomer with resin coat (ZRC)	3.19 (0.30)	3.16 (0.25) B

^a Values with same letters in the same column are not significantly different.
^b Indicates significant differences between distilled water and artificial saliva.

the composite and giomer restoratives was generally higher than that of EQC, ZR, and ZRC in both conditioning mediums. Significant differences in storage modulus were observed between conditioning in distilled water and artificial saliva for FB, TC, BB, EQ, and EQC. Storage modulus of FB, BB, and EQ was significantly larger after conditioning in artificial saliva. Uncoated glass ionomer specimens had a significantly higher storage modulus than their resin-coated counterparts when conditioned in both distilled water and artificial saliva.

Mean loss modulus ranged from 0.24 ± 0.03 to 0.65 ± 0.12 GPa in distilled water and 0.24 ± 0.03 to 0.51 ± 0.09 GPa in artificial saliva (Table 4). TC and FB had the highest loss modulus after exposure to distilled water and artificial saliva, respectively. For both mediums, the lowest loss modulus was observed with ZRC. When conditioned in distilled water, loss modulus of the composite and giomer restoratives was significantly greater than the glass ionomer materials. The same trend was generally observed after conditioning in artificial saliva with the exception of EQ. Significant differences in loss modulus between conditioning in distilled water and artificial saliva were observed for TC, EQ, and EQC. Loss modulus of TC was about 50% lower when exposed to artificial saliva. Unlike TC and EQC, storage in artificial saliva produced higher loss modulus for EQ. For both glass ionomers, resin coating generally resulted in significantly lower loss modulus in artificial saliva.

Loss tangent values of the restoratives ranged from 45.7 ± 7.33 to 134.2 ± 12.36 (10⁻³) in distilled water and 53.7 ± 5.70 to 92.5 ± 9.50 (10⁻³) in

Table 4: Mean Loss Modulus Values (GPa) of the Various Materials (Standard Deviations in Parentheses)^a

Materials (Code)	Distilled Water	Artificial Saliva
Filtek ZT (ZT)	0.47 (0.05) A	0.47 (0.06) A
Filtek Bulk-Fill (FB)	0.45 (0.05) A	0.51 (0.09) A
Tetric N Ceram (TC) ^b	0.65 (0.12)	0.34 (0.06) B
Beautiful (BB)	0.47 (0.08) A	0.44 (0.06) A
Equia Forte without resin coat (EQ) ^b	0.34 (0.06) BC	0.48 (0.05) A
Equia Forte with resin coat (EQC) ^b	0.35 (0.02) B	0.31 (0.03) B
Zirconomer without resin coat (ZR)	0.27 (0.03) CD	0.25 (0.03) C
Zirconomer with resin coat (ZRC)	0.24 (0.03) D	0.24 (0.03) C

^a Values with same letters in the same column are not significantly different.
^b Indicates significant differences between distilled water and artificial saliva.

Table 5: Mean Loss Tangent Values (10⁻³) of the Various Materials (Standard Deviations in Parentheses).^a

Materials (Code)	Distilled Water	Artificial Saliva
Filtek ZT (ZT)	82.7 (5.25) AB	77.3 (24.8) ABCD
Filtek Bulk-Fill (FB)	83.9 (5.97) AB	84.2 (10.41) AB
Tetric N Ceram (TC) ^b	134.2 (12.36)	92.5 (9.50) A
Beautiful (BB) ^b	89.1 (5.76) A	74.7 (5.89) BC
Equia Forte without resin coat (EQ)	45.7 (7.33)	53.7 (5.70) D
Equia Forte with resin coat (EQC)	79.5 (5.87) BC	76.0 (4.06) BC
Zirconomer without resin coat (ZR)	72.9 (3.25) C	70.6 (3.98) C
Zirconomer with resin coat (ZRC)	74.7 (4.55) C	77.4 (7.77) BC

^a Values with same letters in the same column are not significantly different.
^b Indicates significant differences between distilled water and artificial saliva.

artificial saliva (Table 5). For both mediums, the greatest loss tangent was observed with TC and the lowest with EQ. Loss tangent values of the composite and giomer restoratives were higher than the glass ionomer restoratives after conditioning in distilled water. Such trends were not observed in artificial saliva. Significant differences in loss tangent values were observed between mediums for TC and BB. Both these materials exhibited significantly greater loss tangent after exposure to distilled water. While ZR showed no significant difference with resin coating, loss tangent values were significantly greater for EQC in both distilled water and artificial saliva.

DISCUSSION

The viscoelastic properties of contemporary bulk-fill restoratives in distilled water and artificial saliva using DMA were studied. As viscoelastic properties were found to be material and conditioning medium dependent, the null hypotheses were rejected. With DMA, dynamic testing can be performed with a range of temperature, frequency, and amplitude modifications. Temperature was fixed at body temperature (ie, 37°C), while frequency was set at 0.1 to 10 Hz to represent a range from close to “static” testing (0.1 Hz) to the upper limit of normal chewing frequency.²¹ Dimensions for the flexure specimens were based on the work of Yap and others.²² Significant and positive correlations were observed for both flexural strength and modulus between the miniflexural specimens and their lengthier International Organization for Standardization counterparts (25×2×2 mm).²² Besides being clinically more relevant, the miniflexural specimens are also easier

to fabricate and required less material. SAGF medium was used, as its pH, buffering capacity, content, and viscosity mimicked that of natural saliva and has been reported to allow for specification of fluoride release and corrosion behavior of dental biomaterials.²⁰

Storage modulus represents the rigidity or stiffness of the restoratives, while loss modulus indicates their ability to flow. None of the restoratives evaluated had similar or higher modulus than dentin, which is approximately 18 GPa.²³ For both conditioning mediums, EQ was significantly more rigid and will deform less than the other materials under functional stresses, supporting its indication for posterior restorations. This corroborated a recent systematic review that reported comparable failure rate between highly viscous glass ionomers and amalgam in permanent posterior teeth.²⁴ The zirconia-reinforced glass ionomer, however, had the lowest storage modulus regardless of resin coating. This may be attributed to the lack of chemical adhesion between the zirconia fillers and the poly-salt matrix, resulting in areas of stress concentrations.²⁵ Apart from EQ, the composite and giomer materials were generally significantly stiffer than EQC, ZR, and ZRC. Mesquita and others⁷ reported an association between storage modulus and filler weight content. Even with the apparently high percentage of fillers by weight of TC, its storage modulus was still significantly lower than the other polymeric materials. This was due to TC’s low filler volume (notwithstanding its high filler weight), reiterating the greater importance of percentage filler volume in composite characterization. Restoratives with lower modulus have higher elastic

deformation when loaded, leading to possible disruption of restoration–tooth interfacial bonding that is associated with postoperative sensitivity, microleakage, and recurrent caries.²⁶ In both distilled water and artificial saliva, the loss modulus of the composites and giomer was mostly higher than the glass ionomer materials. The polymeric materials thus flowed more than the glass ionomers when subjected to functional loading. Ranking of loss modulus between polymeric materials differed between conditioning mediums. Viscous flow may help reduce or delay fracture, wear, and debonding of restorations.²⁷ Materials with high loss modulus can, however, present with small permanent dimensional changes that may be clinically pertinent.

Loss tangent expresses the energy dissipation capacity of the restoratives and is determined by the ratio of the loss modulus to storage modulus. Mechanical energy is dissipated through conversion into heat by molecular motion and is associated with unrecoverable viscous loss.²⁸ Friction between filler particles and the polymer matrix had been suggested as an important source of energy dissipation during deformation under stress.²⁹ The lower the loss tangent, the quicker the restorative will respond to load and return to its original shape.⁷ In both conditioning mediums, loss tangent values of EQ were significantly lower than the other materials evaluated. Findings were consistent with those of Helvatjoglu-Antoniades and others,³⁰ who reported that composites with the highest filler content and highly viscous glass ionomer exhibited the highest storage modulus and lowest loss tangents. As the same authors also report significant variation of viscoelastic properties with temperature, temperature variations will be taken into consideration for future studies. The small loss tangent values obtained indicate that the restoratives evaluated have a modest viscous component over the frequency range applied, indicating that they were more “elastic-like” in nature.

In the present study, the restoratives were conditioned for seven days and tested in distilled water or artificial saliva at 37°C. Significant differences between conditioning mediums were property and material dependent. For storage modulus, significant differences were observed for all materials with exception of the conventional composite (ZT) and zirconia reinforced glass ionomer (ZR and ZRC). Significant differences in loss modulus were observed between mediums for the bulk-fill composite TC and glass “hybrid” restorative (EQ and EQC), while loss tangent values were significantly different

for TC and the bulk-fill giomer BB. While conditioning in distilled water resulted in better viscoelastic properties for some materials, it reduced storage and loss modulus for others when compared to conditioning in artificial saliva. The interactions between distilled water/artificial saliva and the restoratives are highly complex. For the composite and giomer materials, water sorption from both mediums can result in plasticization and degradation. Absorption of water molecules causes expansion, increasing effective free volume and ease of polymer chain movements, affecting both storage and loss modulus.^{30,31} Degradation from the leaching of fillers and unreacted monomers may be higher in artificial saliva than in distilled water¹⁴ and is anticipated to influence viscoelastic properties. Due to the varied outcomes, the conditioning medium of choice remains equivocal and warrants further investigations.³²

Glass ionomer cements consist of basic fluoroaluminosilicate glasses and acidic copolymers that set chemically by acid–base reactions. Water is the reaction medium into which cement-forming cations are leached and transported to react with polyacids. It also serves to hydrate the cross-link matrix, increasing the cement strength. The final set glass ionomer structure contains a substantial amount of unreacted glass that acts as fillers for the set cement.³³ While previous generations of glass ionomer cements were susceptible to early moisture sensitivity, more recent fast-set and resin-modified cements have improved moisture tolerance. Resin coating is, however, still advocated to improve physicomaterial properties and clinical longevity of highly viscous glass ionomer cements.^{19,34,35} For both EQ and ZR, resin coating did not positively affect their viscoelastic properties. Storage modulus was superior when specimens were not resin coated. For EQ, storage modulus was 1.64 and 2.16 times greater in distilled water and artificial saliva, respectively, when resin coating was omitted. The higher storage modulus observed with conditioning in artificial saliva may be contributed in part by its phosphate content.³⁶ Loss modulus was also generally better when the reinforced glass ionomers were not resin coated. The difference in loss modulus was, however, discrete when compared to storage modulus. Loss tangent was correspondingly lower for EQ without resin coating. The current data supported those of prior studies. Wang and others³⁷ reported that early water exposure did not weaken highly viscous glass ionomer materials, while Pilo and others³⁸ concluded that there was no need to protect

highly viscous glass ionomers from water to improve strength. Other investigators have, however, reported better physico-mechanical properties with resin coating.^{34,39,40} The apparent incongruities could be ascribed to differences in glass ionomers/resin coatings evaluated as well as variances in physico-mechanical properties assessed, testing methodologies, and protocols. Further static and dynamic testing as well as clinical trials are warranted before a definitive inference can be made.

CONCLUSIONS

Within the limitations of this *in vitro* study, the following conclusions may be drawn:

- 1) Viscoelastic properties were found to be material dependent. With the exception of the glass “hybrid” restorative (EQ), the composite and giomer materials generally had higher storage and loss modulus than the reinforced glass ionomer cements.
- 2) As the viscoelastic properties of the giomer bulk-fill restorative were comparable or superior to composites, they could be indicated for posterior restorations.
- 3) Significant differences in viscoelastic properties were observed between conditioning in distilled water and artificial saliva. The variations were again material dependent.
- 4) Resin coating did not positively affect viscoelastic properties and is not required to improve elastic properties.

Acknowledgements

This work was supported by research grant RG531-13HTM, Faculty of Dentistry, University of Malaya. The authors would like to thank 3M EPSE, Shofu Asia, GC Asia, and Ivoclar Vivadent Inc for their material support.

Conflict of Interest

The authors of this article 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.

(Accepted 27 March 2017)

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Queries for odnt-42-04-13

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