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## Effect of preheating on the viscoelastic properties of dental composite under different deformation conditions

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Preheating of dental composites improves their flowability, facilitating successful restorations. However, the flowability of dental composites is affected not only by temperature but also by the deformation conditions. In the present work, the effects of various deformation conditions upon the viscoelastic properties of a preheated dental composite were studied. The rheological properties of Z350 dental composites at 25, 45, and 60°C were measured by a strain-controlled rheometer. When a low strain (0.03%) was applied, the preheated composite exhibited greater shear storage modulus ( $G'$ ) and complex viscosity ( $\eta^*$ ) than a room-temperature composite. Oppositely, when a high strain (50%) was applied,  $G'$  and  $\eta^*$  of a preheated composite were lower than those of a room-temperature composite. Preheating of dental composites might be helpful in clinical practice both to increase the slumping resistance when minimal manipulation is used (e.g., during the build-up of a missing cusp tip) and to increase flowability when manipulation entailing high shear strain is applied (e.g., when uncured composite resin is spread on a dentin surface).

**Keywords:** Deformation condition, Dental composite, Preheating, Viscoelastic properties

### INTRODUCTION

Successful restoration of missing tooth structure by using dental composite relies on its flowability and adaptability, and these characteristics are closely related to the physical characteristic of viscosity. Higher flowability (lower viscosity) has been reported to increase adaptability and decrease microleakage<sup>1-4</sup>.

Preheating dental composite resin is a suggested means of decreasing its viscosity<sup>5-9</sup>, reducing microleakage<sup>10</sup>, increasing the depth of cure<sup>11</sup>, increasing the degree of conversion<sup>12-16</sup>, reduce film thickness<sup>17</sup>, and improving the resin's physical properties<sup>18,19</sup>, which could improve the restoration of lost tooth structure. Repeated and extended preheating have been reported not to damage the monomers<sup>20</sup>.

Viscoelasticity, which is a measurement of flow property, is a function of factors that include time, temperature, and the deformation condition. The temperature dependence of the viscosity of a dental composite involves Brownian thermal energy resulting from the change of the average distance between the constituent molecules. Viscoelasticity is also a function of the deformation condition under which a material is placed, as well as being a function of time. A material's viscosity can be explained in terms of various material behaviors determined by the different measuring conditions. Therefore, when the viscoelasticity of a certain dental material is to be measured, consideration needs to be given to the physical condition under which the dental material is placed during clinical practice. Viscosity should be measured under the same conditions as those in clinical practice, as far as possible. Close

simulation of the clinical conditions under which dental composite is placed would produce more meaningful and relevant results.

In clinical practice, dental composite resin is placed on the tooth structure and applied by using methods that include packing or brushing. During application, the resin experiences various kinds and magnitudes of shear strain. For example, restoration of a missing cusp tip involves positioning an amount of resin composite on the remaining tooth structure, expecting that it will maintain its shape and will not slump. In this situation, the dental composite resin experiences virtually no strain except gravity, since no external force such as packing or brushing is exerted; thus, a composite resin that is stiff and resistant to slumping is desired. This condition can be experimentally simulated by minimizing the strain applied to the dental composite when its viscoelasticity is being measured. On the other hand, when a narrow and deep tooth defect is restored, the dental composite is applied and closely adapted to the tooth structure by manipulating it with instruments, using packing and brushing motions. In this case, success depends on low viscosity and low stiffness of the dental composite, which enable close adaptation of the dental composite to the defect lesion. These brushing and packing motions subject the composite to considerable shearing force. In this situation, it is desirable that the composite resin be less viscous.

Considering both these situations, the ideal rheological properties of dental composite resin include lower viscosity under high shear strain, and higher viscosity or more elasticity under low shear strain. While dental composite resins have been reported to exhibit

higher viscosity at a lower strain and lower viscosity at a higher strain<sup>21</sup>), there have been no reports on the effect of deformation conditions on pre-heated dental composites. To the best of our knowledge, the present work is the first in which the effect of shear strain upon the viscoelasticity of preheated dental composite was studied.

Another aspect worth considering is whether the preheating of dental composite can induce structural changes in it and, if so, whether this structural alteration persists after cooling. In the clinical setting, composite resin is preheated to approximately 60°C in a warming device and is then placed at the prepared tooth cavity. Thereafter, the heated dental composite can be cooled to around 37°C in the oral cavity before polymerization is carried out. The effect of this change in temperature upon the structure of the dental composite is not yet known.

The purpose of this study was to investigate the rheological properties of preheated dental composite under various deformation conditions (*i.e.*, various shear strains). The structural change induced by preheating was also investigated.

## MATERIALS AND METHODS

### Materials

The commercial dental composite used was Filtek Z350 universal restorative material (3M, ESPE, St. Paul, MN, USA). This composite is composed of a zirconia/silica cluster filler having an average cluster particle size of 0.6–1.4 μm and a primary particle size of 5–20 nm, and agglomerated/nonaggregated 20 nm silica filler. The inorganic particles constituted about 78.5 wt% and 59.5 vol% of the composite; the rest of the composite was methacrylate resin, a dental adhesive included to permanently bond the restoration to the tooth structure.

### Rheological measurements

Rheological measurements on the dental composite were performed by using an ARES plate-equipped strain controlled device (TA Instruments, New Castle, DE, USA). Parallel plate geometry with an 8 mm radius and 1 mm gap was used. The experiments were performed in the steady mode and oscillation modes at 25, 45, and 60°C.

#### 1. Dynamic strain sweep test

Viscoelasticity was measured by means of dynamic strain sweep tests conducted at the shear strains of 0.01–100% and the fixed frequency of 1 rad/s, at 25 and 60°C. This test was carried out to investigate the effect of shear strain (from 0.01% to 100%) on the viscoelastic properties of heated and room-temperature dental composite.

#### 2. Dynamic frequency sweep test

Viscoelasticity was measured by means of dynamic frequency sweep tests conducted at the frequencies of

0.1–100 rad/s, using sinusoidal oscillations. The dynamic frequency sweep test was conducted using the fixed strain amplitudes of 0.03% and 50%, which simulated the clinical situations in which very low strain (0.03%) and high strain (50%) were applied to dental composites. The measuring temperatures were 25, 45, and 60°C.

#### 3. Steady rate sweep test

Viscoelasticity was measured at the shear rate of 0.001–100 (1/s). The measuring temperatures were 25, 45, and 60°C. This test simulated the clinical situation when shear strain is applied to dental composite very quickly (*e.g.* 100 1/s) or very slowly (*e.g.* 0.001 1/s).

#### 4. Temperature sweep test

To study the effect of temperature upon the structure of the dental composite, measurements were undertaken while the plate on which specimens were placed was heated from 25 to 60°C and cooled back to 25°C under the fixed strain amplitudes of 0.03% and 50%, respectively, and under the oscillation frequency of 1 rad/s.

### Theory of viscoelasticity and analysis of data

During a dynamic oscillatory shear test, and when the oscillation frequency is indicated as  $\omega$  and the phase difference between stress and strain is indicated as  $\delta$ , the relationship between the strain and the stress can be expressed as follows.

$$\begin{aligned} \text{shear strain } \gamma(t) &= \gamma_0 e^{i(\omega t)} \\ \text{shear stress } \sigma(t) &= \sigma_0 e^{i(\omega t + \delta)} \end{aligned}$$

Then, the complex shear modulus  $G^*$  can be expressed as follows:

$$G^* = \frac{\sigma(t)}{\gamma(t)} = \frac{\sigma_0}{\gamma_0} e^{i\delta} = \frac{\sigma_0}{\gamma_0} (\cos\delta + i\sin\delta) = G' + iG''$$

where  $G'$  is the real (storage) shear modulus and  $G''$  is the imaginary (loss) shear modulus.

$G'$  represents the deformation energy stored in the sample during the shear process, quantifying the elastic behavior of a sample.  $G''$  represents the deformation energy lost to heat during the shear process, quantifying the viscous behavior of a sample. The ratio of  $G''$  to  $G'$ ,  $G''/G'$ , is the loss tangent,  $\tan \delta$ .

Three replicate measurements were made for each sample. The  $G'$ ,  $G''$ ,  $\eta^*$  and  $\tan \delta$  of the specimens were measured under various conditions, and the relationships between these measured values and the changes in temperature and shear strain were investigated.

## RESULTS

The storage modulus ( $G'$ ) of preheated (60°C) or room-temperature (25°C) dental composites were measured during a strain sweep test conducted at the frequency of 1 rad/s over the strain range of 0.01–100% (Fig. 1). For strains below 0.1%, the measured  $G'$  values of preheated composites were higher than those of room-temperature

composites. Preheated composites displayed a more rapid decrease in  $G'$  with increasing strain than room-temperature composites. For the range of strains above 0.1%, preheated composites showed lower  $G'$  results than those of room temperature composites. While determination of the linear range was difficult, 0.03% was selected as the linear strain value.

A frequency sweep test (0.1–100 rad/s) was conducted on dental composites subjected to 0.03% linear strain at 25, 45 and 60°C (Fig. 2). For all temperatures studied,  $G'$  plateaued in the low-frequency region (Fig. 2), indicating the solid-like behavior of dental composite.

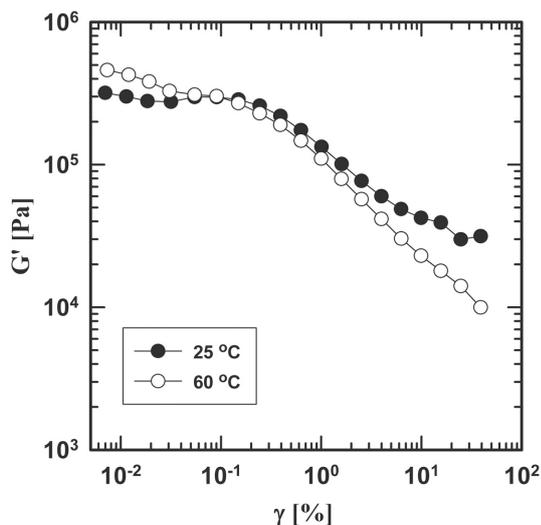


Fig. 1 Strain sweep test ( $\gamma$ : strain,  $G'$ : shear storage modulus)

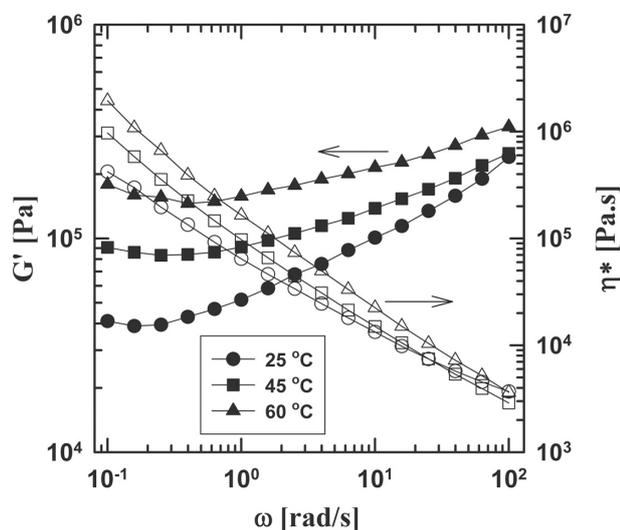


Fig. 2 Frequency sweep test in the linear strain region: shear storage modulus  $G'$  versus frequency ( $\omega$ ), complex viscosity  $\eta^*$  versus frequency ( $\omega$ ).

Under these conditions,  $G'$  (Fig. 2) and complex viscosity ( $\eta^*$ ; Fig. 2) of the dental composites were greatest at 60°C among the three temperatures tested.

$G'$  and  $\eta^*$  of dental composites at 25, 45, and 60°C were measured in the nonlinear (high) strain region (50%) as a function of frequency (0.1–100 rad/s; Fig. 3). The  $\eta^*$  of the composites decreased with increasing frequency for all temperatures studied (Fig. 3).  $G'$  increased at low frequency due to reorientation of particles in the matrix under low-speed oscillation, and decreased at high frequency due to breakdown mediated by high-speed oscillation (Fig. 3). Under these conditions,  $G'$  and  $\eta^*$  of the dental composites were lowest at 60°C among the three temperatures tested. These results gave evidence of the temperature dependence of the viscosity of dental composites filled with inorganic particles.

The effects of the shear rate upon the viscosity of dental composites at 25, 45, and 60°C were also investigated (Fig. 4). The rate sweep test revealed decreased viscosity with increasing shear rate (shear thinning). For all shear rates tested, the composite at 60°C displayed less viscosity than the composites at 25 and 45°C.

A temperature sweep loop test was conducted in which the composite was heated from 25 to 60°C and then cooled back to 25°C (Fig. 5). These tests were carried out using the strain amplitudes of 0.03 and 50% and using the fixed oscillation frequency of 1 rad/s. Under the strain amplitude of 0.03%,  $G'$  increased as the composite was heated to 60°C; then, during cooling,  $G'$  remained similar to its value at 60°C (Fig. 5). Oppositely, under the strain amplitude of 50%,  $G'$  decreased during heating to 60°C and then remained at a similar value during subsequent cooling (Fig. 5).

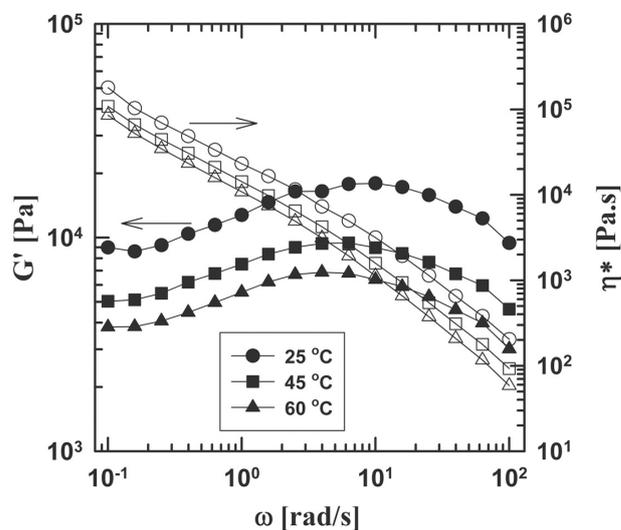


Fig. 3 Frequency sweep test in the nonlinear strain region: shear storage modulus  $G'$  versus frequency ( $\omega$ ), complex viscosity  $\eta^*$  versus frequency ( $\omega$ ).

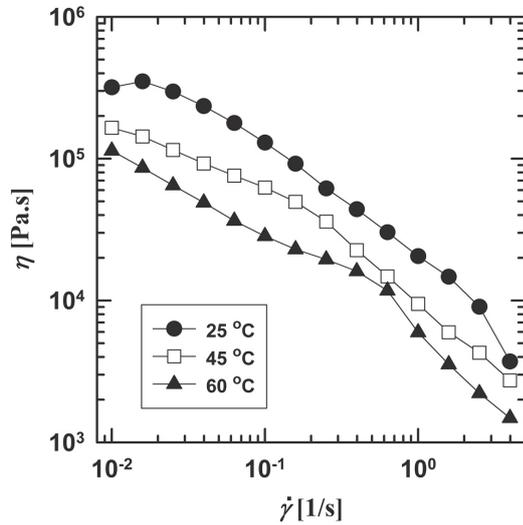


Fig. 4 Rate sweep test. (shear rate:  $\dot{\gamma}$ ;  $\eta$ : viscosity)

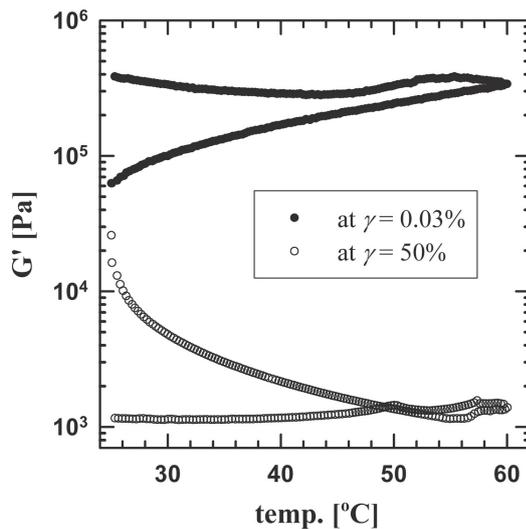


Fig. 5 Temperature sweep loop tests: shear storage modulus  $G'$  during the temperature sweep under strain in the linear strain region and the nonlinear strain region.

## DISCUSSION

When a dental composite is applied to a tooth structure by means of a brushing motion, it is strained; for example, when a composite resin 1.0 mm thick is spread to a width of 0.5 mm, it is placed under the strain amplitude of 50%. To simulate the clinical situation in which the dental composite is placed on the remaining tooth structure without any manipulation, for example, when a missing cusp tip is restored, we evaluated  $G'$  and  $\eta^*$  of the selected dental composite material under a very low strain (0.03%) during a frequency sweep test. Also,

to simulate the clinical situation in which the dental composite is positioned and manipulated by brushing or spreading, we evaluated  $G'$  and  $\eta^*$  of the composite under high strain (50%).

The results of these tests showed the interesting characteristics of the dental composite studied. Under the low strain of 0.03%, preheated composites had higher  $G'$  and  $\eta^*$  than those of room-temperature composites, indicating that a preheated composite placed on a tooth structure without any further manipulation will tend to maintain its shape (*i.e.*, will resist slumping) more than a room-temperature composite will. On the other hand, under the higher strain of 50%, preheated composites had lower  $G'$  and  $\eta^*$  than room-temperature composites, indicating that a preheated composite placed on a tooth structure and spread by a brushing motion will flow and adapt better than a room-temperature composite will. Considering both these observations, the preheating of dental composite appears to improve its rheological properties in two ways. Preheating helps the composite to keep its shape with less slumping when it is placed and when no external manipulation force is exerted. Additionally, preheating allows the composite to flow more readily when it is manipulated.

Under low strain (0.03%), dental composites at 60°C showed higher  $G'$  and  $\eta^*$  than composites at 25°C. This is not a common phenomenon in composite rheology and is regarded as an intrinsic property of the dental composite, arising from the microstructure and composition of the inorganic fillers. It is expected that the heating of a dental composite will increase the dispersibility of particles in the material system. That is to say, preheating increases the mobility of particles (fillers) within the resin matrix, leading to a more compact, homogeneous, and well-dispersed particle system that was evident in the present study as an increased storage modulus.

At the higher strain (50%), dental composites at 60°C displayed lower  $G'$  and  $\eta^*$  than composites at 25°C. This agrees with a previous report indicating that the preheating of composites enhanced their flowability<sup>5)</sup>. This is a common phenomenon in composite rheology. We speculate that preheating of dental composite increases the mobility of the unpolymerized monomer<sup>14)</sup> and allows better distribution of the filler particles. When no external force is applied, the mobile monomers are held by the well-distributed fillers and develop microstructures, leading to the observed increase in  $G'$ . However, when an external strain strong enough to disrupt the developing microstructure is applied, the increased mobility of the preheated unpolymerized monomer allows the composite to move more freely, increasing its flowability compared to that of a room-temperature composite.

In the present work, we observed that preheating altered the structure of composite resins. In the temperature sweep loop test, the shear  $G'$  of a room-temperature dental composite differed before and after the preheating and cooling process. Dental composites

examined at 25°C after the heating and cooling cycle had enhanced physical properties, with a well-dispersed state of the inorganic filler microstructure, resulting in the observation of higher  $G'$  than that observed at 25°C before heating (Fig. 5). These results indicate that the internal structure and filler distribution of dental composites can be improved by heat treatment, and that these improvements persist even after cooling. This observation alleviates concern that a preheated composite could return to its original structure when it cools in the oral cavity.

Under the high strain of 50%, the trend in  $G'$  was markedly different from that evident under the strain of 0.03%. Under the strain of 50%,  $G'$  decreased with increasing temperature and then was maintained during cooling back to 25°C (Fig. 5). That is to say,  $G'$  at 25°C after a temperature sweep test to 60°C and then back to 25°C was smaller than  $G'$  for the initial state at 25°C. The best explanation for our observations is that the microstructure of dental composites is intrinsically altered by the high deformation strain applied, and does not recover during the temperature sweep. The differences observed between the temperature sweep loop test results conducted under low and high strains, but with the same thermal history, is likely to be explained by the hydrodynamic force generated by the high strain, which leads to rupture of the microstructure in a manner similar to thermal degradation.

### CONCLUSION

Within the scope of this study, we suggest that preheating of dental composites might be helpful in clinical practice, as it can increase the slumping resistance when the manipulation force is not applied. At the same time, it can increase the unpolymerized composite's flowability and adaptability when a manipulation force is applied to shape it. Finally, the observation that preheated composites display improved rheological properties even after cooling to room temperature may be beneficial to dental practice, because preheated dental composites cool in the oral cavity after their application.

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### CONFLICTS OF INTEREST

The authors declare no conflicts of interest.

### REFERENCES

- 1) Opdam NJ, Roeters JJ, Joosten M, Veeke O. Porosities and voids in Class I restorations placed by six operators using a packable or syringable composite. *Dent Mater* 2002; 18: 58-63.
- 2) Unlu N, Krakaya S, Ozer F, Say EC. Reducing microleakage in composite resin restorations: an in vitro study. *Eur J Prosthodont Restor Dent* 2003; 11: 171-155.
- 3) Li Q, Jepsen S, Albers HK, Eberhard J. Flowable materials as an intermediate layer could improve the marginal and internal adaptation of composite restorations in Class-V-cavities. *Dent Mater* 2006; 22: 250-257.
- 4) Leevailoj C, Cochran MA, Matis BA, Moore BK, Platt JA. Microleakage of posterior packable resin composites with and without flowable liners. *Oper Dent* 2001; 26: 302-307.
- 5) Blalock JS, Holmes RG, Rueggeberg FA. Effect of temperature on unpolymerized composite resin film thickness. *J Prosthet Dent* 2006; 96: 424-432.
- 6) Freedman G, Krejci I. Warming up to composites. *Compend Contin Educ Dent* 2004; 25: 371-374, 76; quiz 78.
- 7) Trushkowsky R. A new technique for placement of posterior composite. *Dent Today* 2002; 21: 20-21.
- 8) Deliperi S, Bardwell DN. Preheating composite resin: a clinical perspective. *Pract Proced Aesthet Dent* 2007; 19: 161-164.
- 9) Knight JS, Fraughn R, Norrington D. Effect of temperature on the flow properties of resin composite. *Gen Dent* 2006; 54: 14-16.
- 10) Wagner WC, Asku MN, Neme AM, Linger JB, Pink FE, Walker S. Effect of pre-heating resin composite on restoration microleakage. *Oper Dent* 2008; 33: 72-78.
- 11) Munoz CA, Bond PR, Sy-Munoz J, Tan D, Peterson J. Effect of pre-heating on depth of cure and surface hardness of light-polymerized resin composites. *Am J Dent* 2008; 21: 215-222.
- 12) Prasanna N, Pallavi Reddy Y, Kavitha S, Lakshmi Narayanan L. Degree of conversion and residual stress of preheated and room-temperature composites. *Indian J Dent Res* 2007; 18: 173-176.
- 13) Conditt M, Leinfelder K. Improving the polymerization of composite resins. *Pract Proced Aesthet Dent* 2006; 18: 169-171.
- 14) Daronch M, Rueggeberg FA, De Goes MF, Giudici R. Polymerization kinetics of pre-heated composite. *J Dent Res* 2006; 85: 38-43.
- 15) Daronch M, Rueggeberg FA, De Goes MF. Monomer conversion of pre-heated composite. *J Dent Res* 2005; 84: 663-667.
- 16) Trujillo M, Newman SM, Stansbury JW. Use of near-IR to monitor the influence of external heating on dental composite photopolymerization. *Dent Mater* 2004; 20: 766-777.
- 17) Dionysopoulos D, Tolidis K, Gerasimou P, Koliniotou-Koumpia E. Effect of preheating on the film thickness of contemporary composite restorative materials. *J Dent Sci* 2014;9:313-319
- 18) Bausch JR, de Lange C, Davidson CL. The influence of temperature on some physical properties of dental composites. *J Oral Rehabil* 1981; 8: 309-317.
- 19) Awliya WY. The influence of temperature on the efficacy of polymerization of composite resin. *J Contemp Dent Pract* 2007; 8: 9-16.
- 20) Daronch M, Rueggeberg FA, Moss L, de Goes MF. Clinically relevant issues related to preheating composites. *J Esthet Restor Dent* 2006; 18: 340-350; discussion 51.
- 21) Lee I, Chang J, Ferracane J. Slumping resistance and viscoelasticity prior to setting of dental composites. *Dent Mater* 2008; 24: 1586-1593.