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Rheological properties of flowable resin composites and pit and fissure sealants

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ABSTRACT

Objectives. The purpose of this research was to determine the viscoelastic properties of commercially available flowable resin composites and resin-based pit and fissure sealants. The weight percentage of filler particles and the morphology of the filler particles were also investigated.

Methods. Eight flowable resin composites (Admira Flow, Filtek Flow, FlowLine, Grandio Flow, Point-4 Flowable, Revolution Formula 2, Tetric Flow and X-Flow) and four pit and fissure sealants (Clinpro, Delton FS+, Estiseal F and Guardian Seal) were tested. Rheological measurements were performed using a dynamic oscillation rheometer. The filler weight content was determined by thermogravimetric analysis (TGA) and the morphology of the particles was investigated by scanning-electron microscopy (SEM).

Results. Flowable resin composites are non-Newtonian, shear-thinning materials. As the shear rate increased, the complex viscosity decreased drastically. They all showed elasticity even at the lowest frequencies. They also all showed thixotropy. Pit and fissure sealants are non-Newtonian, very low-viscosity fluids. No correlation was found between the rheological properties and the filler weight content or the particles' shape.

Significance. Huge differences are observed in the viscosity and flow characteristics of flowable resin composites that can have a potential influence on their clinical behavior during handling and thus on their clinical indications. Pit and fissure sealants show very different rheological properties from one another.

1. Introduction

Since resin composite restorations were developed as an esthetic and more conservative alternative to silver amalgams, many efforts have been made to improve their physical and mechanical properties by the development of new monomers, new filler particles and surface treatments as well as new photoinitiation systems and many other important topics [1–8].

Resin composites are more time-consuming and by far more technique sensitive than silver amalgam restorations. Thus, the handling characteristics are of major importance for the final quality, success rate and life span of composite restorations. Indeed, the consistency and flow characteristics of the material are directly related to the ability of the resin composite to be correctly placed and shaped in the cavity without the formation of gaps between the material and the cavity margins or between successive layers of therestoration.

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Resin composites should adhere to tooth tissues and not to instruments [9–14].

The first generation of flowable resin composites was introduced in late 1996. They were created mainly by retaining the same small particle size of traditional hybrid composites, but reducing the filler content and consequently the viscosity of the mixture [15]. These materials can be manipulated using a syringe and a loading tip and injected where traditional instruments' access is difficult or impossible, because of the low viscosity of these materials.

Throughout the years, various techniques were used as an attempt to characterize the rheological properties of resin composites. Ferracane et al. [9] used a rotational cone and plate viscometer to determine the viscosity characteristics and working-time of chemically cured composites. Opdam et al. [16] compared the consistency of several resin composites by a press method. Lee et al. [12] characterized the viscoelastic properties of resin composites using a dynamic oscillatory rheometer and, more recently, using a vertical oscillation rheometer [14]. Lee et al. studied the rheological properties of resin composites with variations in monomer and filler composition using a dynamic oscillatory rheometer [13].

The consistency and flow-characteristics of flowable resin composites as well as pit and fissure sealants are the most obvious, since their handling is mainly based on the controlled flow of the material in the cavity or in the pits and fissures of teeth. However, the rheological properties of flowable resin-based materials are complex and it is known that there are large differences in the consistency of flowable resin composites from one manufacturer to another [15]. Therefore, there is a need for a classification of these materials as a function of their clinical indications. Indeed, there is an obvious interest to use very low-viscosity materials to seal the pits and fissures of the occlusal anatomy but one may prefer to use resin composites with higher consistency in order to achieve the restoration of class V cavities.

The purpose of this study was to understand the structure and the dynamic behavior of commercially available flowable resin composites and pit and fissure sealants by means of dynamic rheological measurements as well as morphological analysis of their inorganic fraction in order to determine precisely their clinical indications as a consequence of their flow characteristics. Rheological measurements were performed using a dynamic oscillatory rheometer. The inorganic fraction was analyzed by thermogravimetric analysis (TGA) to determine the weight percentage of filler particles and by scanning-electron microscopy (SEM) to investigate the morphology of the particles.

2. Materials and methods

2.1. *Resin composites and pit and fissure sealants*

Eight flowable resin composites and four pit and fissure sealants were tested in this study. They are listed in Table 1. It is to be noted that Admira Flow has a peculiarity in the fact that it emanates from Ormocer (organically modified ceramics) technology.

2.2. *Rheological measurements*

Rheological measurements on all the materials tested in this study were performed using an Advanced Rheometric Expansion System (ARES, TA Instruments Inc., New Castle, DE, USA) at a temperature of 23 °C.

2.3. *Flowable resin composites*

A parallel plates viscometer module with a diameter of 25 mm was used to measure the rheological properties of the flowable resin composites tested. The gap between the plates was 1 mm. Strain sweep tests were performed to check the range of the uniform output signal so that all the following measurements were carried out in the linear range of deformation of each material. A time sweep test was then made for 7200 s at a frequency of 0.1 rad/s after materials had undergone a pre-shear at a rate of $1\,s^{-1}$ for a complete 360° revolution of the lower plate in order to destroy the internal structure of the samples and to reveal any potential thixotropic behavior. Then, a frequency sweep test was performed from 100 rad/s to 0.01 rad/s to determine the variation of the complex viscosity (η^*), storage (elastic) shear modulus (G'), loss (viscous)

Table 1 – List of the materials tested

Material	Manufacturer	Shade	Batch
Resin composites			
Admira Flow	Voco (Cuxhaven, Germany)	A2	500244
Filtek Flow	3M-ESPE (St. Paul, MN, USA)	A3	4GJ
FlowLine	Heraeus-Kulzer (Wehrheim, Germany)	A2	010103
Grandio Flow	Voco (Cuxhaven, Germany)	A3	450086
Point-4 Flowable	Kerr (Orange, CA, USA)	A3	408419
Revolution Formula 2	Kerr (Orange, CA, USA)	A3	4-1336
Tetric Flow	Ivoclar-Vivadent (Schaan, Liechtenstein)	A3	G05246
X-Flow	Dentsply-De Trey (Konstanz, Germany)	A3	501000570
Pit and fissure sealants			
Clinpro	3M-ESPE (St. Paul, MN, USA)	N/A	5FG
Delton FS$^+$	Dentsply-De Trey (Konstanz, Germany)	N/A	040825
Estiseal F	Heraeus-Kulzer (Wehrheim, Germany)	N/A	010027
Guardian Seal	Kerr (Orange, CA, USA)	N/A	403412

shear modulus (G'') and loss tangent ($\tan\delta$) as a function of the frequency.

2.4. *Pit and fissure sealants*

Due to the very low viscosity of pit and fissure sealants, a cone and plate viscometer module with a diameter of 50 mm and a cone angle of 0.02 rad was chosen. The sequence used for the rheological measurements on the pit and fissure sealants was the same as the one used for the flowable resin composites except that no time sweep tests were performed and the frequencies analyzed in the frequency sweep tests ranged from 100 rad/s to 0.1 rad/s. Indeed, due to the very low viscosity of pit and fissure sealants, no satisfactory signal was recorded between 0.1 rad/s and 0.01 rad/s.

2.5. *Weight percentage of filler particles*

The weight percentage of filler particles was determined using thermogravimetric analysis (TGA/SDTA861^e, Mettler-Toledo, Greinfensee, Switzerland). Weight changes were evaluated as a function of time and temperature during a thermal program ranging from 30 °C to 900 °C at the rate of 10 °C/min in normal atmosphere followed by air cooling to room temperature. The calculated ratio between the final weight of the sample and its nominal weight is assigned to the inorganic fraction. Three samples of each material were analyzed.

2.6. *Morphology of the filler particles*

The filler morphology was determined using scanning-electron microscopy (SEM). Unpolymerized monomers were removed by a washing technique: 0.5 g of each material was dissolved in 4 ml of acetone and centrifuged for 5 min at 700 × *g*. This process was repeated three times using acetone, followed by three others with chloroform for a further washing to ensure complete elimination of the unpolymerized resin. The remaining fillers – including prepolymerized fillers – were suspended in 2.5 ml of absolute ethanol, smeared on a glass slide (7.5 cm × 2.5 cm) and dried at 37 °C for 6 h. After gold coating, fillers were observed by SEM (Leica Stereoscan S-260, Cambridge, UK) at 5000 and 10,000 magnifications.

3. Results

3.1. *Rheological measurements*

3.1.1. *Flowable resin composites*

The evolution of the complex viscosity at 0.1 rad/s of the flowable composites, tested as a function of the time after destruction, is presented in Table 2. Fig. 1 shows the viscosity as a function of the time reported to the viscosity without any deformation (i.e. at rheological equilibrium, relative viscosity = 1) of all the materials tested. The complex viscosity at t = 0 s is much lower than the one at t = 7200 s and increases gradually for all the materials tested except for Filtek Flow whose complex viscosity shows a huge overshoot phenomenon (i.e. the viscosity is higher than when at rest) immediately after deformation and then returns progressively to equilibrium after around 2000 s.

Table 2 – Complex viscosity (η^*) as a function of the time after destruction of the flowable resin composites tested

Material	Complex viscosity (Pa s) at 0.1 rad/s		Time constant, τ
	t = 0 s	t = 7200 s	
Admira Flow	304.73	939.18	1822.16
Filtek Flow	30286	28344	N/A
FlowLine	3692.43	14718.4	2536.14
Grandio Flow	86.977	740.16	2492.21
Point-4 Fl.	6817.75	10792	2562.59
Revolution F.2	832.75	2757	2431.67
Tetric Flow	141.53	968.06	4098.86
X-Flow	706.79	2420.4	583.77

Right column shows the calculated time constant of each material.

As an attempt to characterize the evolution of the complex viscosity after destruction of the resin composites, the results presented above were modeled in order to isolate the speed of return to equilibrium. To obtain this, the complex viscosity was expressed as follows:

$$\eta_{\mathrm{rel}} = \frac{\eta_{7200\,\mathrm{s}} - \eta_{\mathrm{t}}}{\eta_{7200\,\mathrm{s}}}$$

on a logarithmic scale. The curve fitting gives a straight line whose equation is written as follows:

$$y = a\,\exp\left(\frac{-x}{\tau}\right)$$

where τ is the time constant of the material. This is presented in Fig. 2. Only FlowLine, Point-4 Flowable and Revolution Formula 2 were represented to keep the figure easily understandable. It is to be noted that the three slopes are very close to each other. Table 2 presents the time constant of each resin composite as calculated using this method.

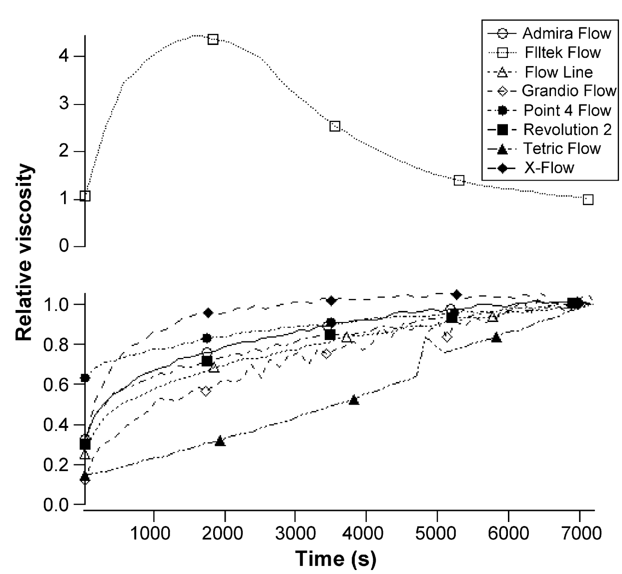

Fig. 1 – Relative viscosity of the flowable resin composites as a function of the time after deformation (the 1 value is the viscosity of the sample without any deformation).

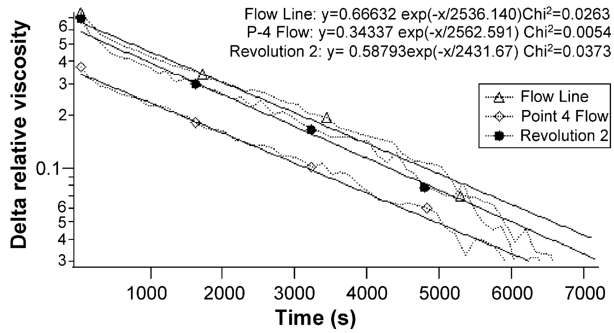

Fig. 2 – Modeling of the return to equilibrium of the flowable resin composites as a function of the time after deformation.

The complex viscosities versus frequency of the eight resin composites tested are presented in Fig. 3. They all decreased as frequency increased, indicating strong non-Newtonian behavior and no hint of a Newtonian plateau even at the lowest frequencies. Globally, the difference in the complex viscosity of the materials between low and high frequencies lies between two and three decades. Filtek Flow has the highest viscosity whatever the frequency. Grandio Flow shows the lowest complex viscosity at low frequencies and Admira Flow shows the lowest complex viscosity at high frequencies. However, Tetric Flow has the lowest complex viscosity at intermediate frequencies. At very high frequencies, Revolution Formula 2 has the lowest complex viscosity.

Fig. 4 shows the storage moduli of the flowable resin composites. The low slopes at low frequencies (except for Admira Flow, Grandio Flow and X-Flow) indicate very strong elasticity of the materials. Confirming the viscosity results, no transition to terminal behavior (i.e. simple liquid flow) can be observed even at the lowest frequencies.

The storage shear modulus of all the flowable resin composites tested increases as the frequency increases. Filtek Flow has the highest storage shear modulus whatever the frequency. Tetric Flow has the lowest storage modulus, except at low frequency where Grandio Flow has the lowest.

For clarity purpose, the values of the loss modulus and the loss tangent are not presented because they did not give additional information on the rheological properties of the materials tested.

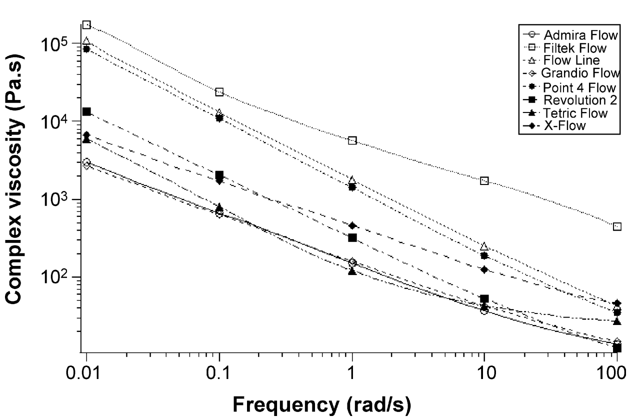

Fig. 3 – Complex viscosity (η^*) vs. frequency for the flowable resin composites tested.

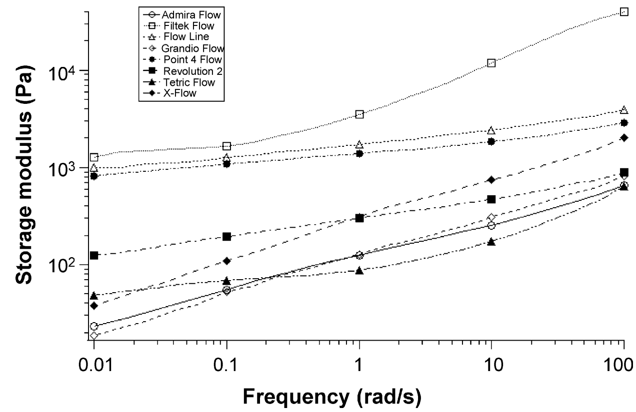

Fig. 4 – Storage modulus (G') vs. frequency of the flowable resin composites tested.

3.1.2. *Pit and fissure sealants*

The complex viscosities versus frequency of the four pit and fissure sealants tested are shown in Fig. 5. They very slightly decreased as frequency increased except for the Estiseal F which showed higher variation of complex viscosity. They were consequently non-Newtonian. The difference in the complex viscosity at low and high frequencies lay around half a decade except for Estiseal F, which it lay at around one decade. As for the flowable resin composites, no Newtonian plateau at the lowest frequencies can be observed.

Fig. 6 presents the storage moduli of the pit and fissure sealants tested. They all increased as the frequency increased. Again, no sign of a transition to terminal behavior is observed.

3.2. *Weight percentage of filler particles*

The mean values and standard deviations of the weight percentage of fillers of all the materials tested are shown in Fig. 7. Averages for the flowable resin composites ranged from 45.0% for the Revolution Formula 2 to 77.8% for the Grandio Flow. For the pit and fissure sealants, averages varied from 6.5% for the Clinpro to 54.3% for the Delton FS+.

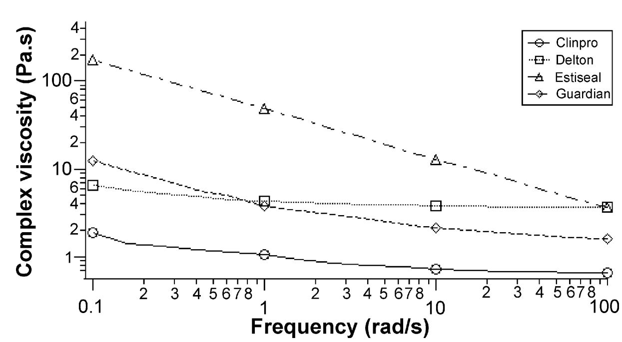

Fig. 5 – Complex viscosity (η^*) vs. frequency of the pit and fissure sealants tested.

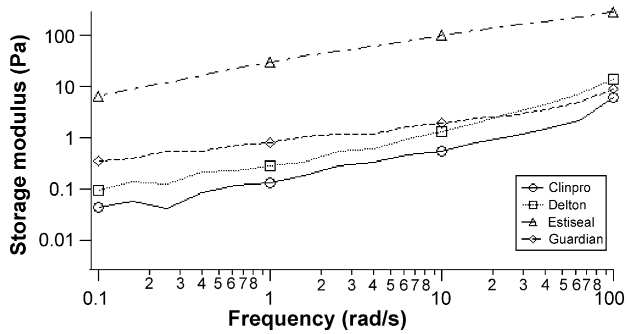

Fig. 6 – Storage modulus (G′) vs. frequency of the pit and fissure sealants tested.

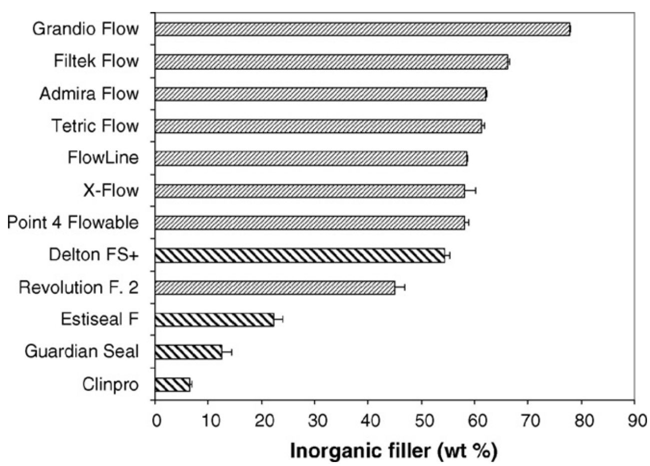

Fig. 7 – Mean values and standard deviations of the percentage of fillers by weight of all the materials tested.

3.3. *Morphology of the filler particles*

The SEM microphotographs of the filler particles that are included in the flowable resin composites are shown in Fig. 8. Inserts are twice the magnification of the main pictures. Filtek Flow and Point-4 Flowable showed rather spherical particles. More irregular shaped particles were found in Admira Flow, FlowLine, Grandio Flow, Revolution Formula 2, Tetric Flow and X-Flow.

Fig. 9 presents the filler particles that can be found in the pit and fissure sealants. It is to be noted that for an unknown reason, no satisfying image of the particles of the Clinpro could be obtained. Delton FS+, Estiseal F and Guardian Seal showed irregular shaped particles. Delton FS+ showed bigger particles than the two others.

4. Discussion

All the flowable resin composites tested in this study are viscoelastic materials. They are all non-Newtonian and show a shear-thinning behavior, i.e. their complex viscosity decreases as the frequency increases. This is in accordance with the results obtained by Lee et al. [12–14]. The fact that there is no hint of a Newtonian plateau for the complex viscosity of the flowable composite even at the lowest frequencies in Fig. 2, combined with the fact that there is no drop of the storage modulus in Fig. 3 at low frequency indicates a solid-like behavior at rest.

From the results of the complex viscosity and the storage modulus versus frequency, three groups of flowable resin composites can be observed. The first one, including Filtek Flow, FlowLine and Point-4 Flowable, has higher viscosities and storage moduli than the others. In addition, the slope of their storage moduli at low frequencies is close to the horizontal, indicating a very strong elasticity and thus a strong solid-like behavior. At high frequencies, Filtek Flow shows a higher viscosity than FlowLine and Point-4 Flowable. The second group (Revolution Formula 2 and Tetric Flow) shows materials that have a lower viscosity than the previous one, but whose storage moduli at the lowest frequencies show similar properties than for the first group, i.e. low slopes and thus strong solid-like behavior. The third group, including Admira Flow, Grandio Flow and X-Flow, has rheological properties similar to those of the second group except that their storage moduli show higher slopes at low frequencies, indicating less solid-like behavior than for the other flowable resin composites tested.

Clinically, this means that a composite that belongs to the third groups has better ability to flow by itself in small cavities or fissures than the others. On the contrary, those belonging to the first or the second group stay better in place when manipulated and are less subject to creep flow. But this has to be put in balance with the fact that, as demonstrated in Fig. 1, their viscosity decreases dramatically when submitted to an important destruction. Indeed, flowable resin composites are most often handled using a syringe and the flow of the materials through the syringe tip strongly affects their internal structure and thus highly reduces their viscosity. Further studies are needed to better understand their behavior during the seconds and minutes after strong destruction.

Except for Filtek Flow, the complex viscosity of all the resin composites tested returns to equilibrium while their internal structure builds up again. This follows an exponential law, as shown in Fig. 2. Globally, the rate remained pretty much the same whatever the material. The overshoot observed with Filtek Flow suggests a jamming phenomenon indicating that its inorganic fraction must be very close to the maximum packing limit of filler particles (in this case small spherical particles). From a clinical point of view, since resin composites are photopolymerized immediately after being handled, they are frozen in a state that is far from equilibrium. It is thus quite simple to imagine that this could have consequences on the shrinkage stress, the post-shrinkage phenomenon, or even on the physical and mechanical properties of the materials. Again, further studies are needed.

The use of thermogravimetric analysis is a good method to determine the filler particle weight content of dental resin composites, as demonstrated by Sabbagh et al. [17] and the values obtained with this method are highly correlated with those obtained by the more traditional ashing in air technique.

No correlation could be made between the weight percentage of filler particles and the complex viscosity of the flowable resin composites. This is in accordance with the results obtained by Lee et al. [12]. Grandio Flow, which has the highest filler particle weight content (77.8%), the lowest viscosity at low frequencies and one of the lowest at both midrange

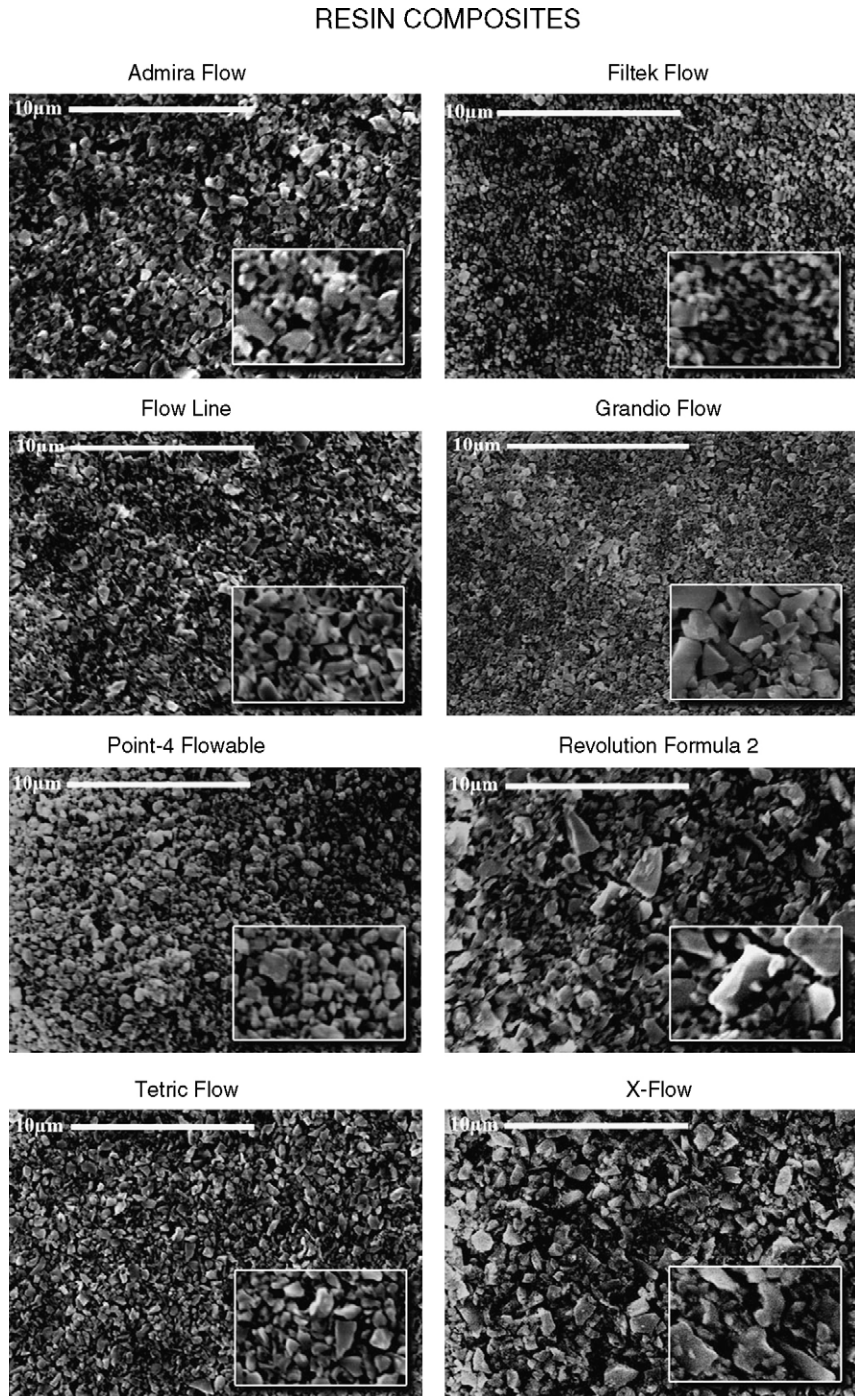

Fig. 8 – Fillers of the flowable resin composites by scanning-electron microscopy at 5000× magnification (10,000× in the inserts).

and high frequencies. This is of major interest since this material shows mechanical properties that are comparable to those of universal hybrid composites [18] while traditional flowable composites have lower properties [19]. Adding nanoparticles in resin composites could consequently allow the production of flowable materials with both higher mechanical properties and better flow characteristics than previously. The third most filled material, Admira Flow (62.1%), shows the lowest viscosity at high frequencies. The explanation may at least partially reside in the fact that it is actually an Ormocer and that part of its inorganic content does probably not influence its flow characteristics.

All the pit and fissure sealants tested in this study are non-Newtonian. They are low-viscosity materials but show an elastic behavior at low frequencies. As for the flowable resin composites, no correlation could be made between the complex viscosities and the filler weight content of the filler particles. Estiseal F has a higher viscosity and higher stor-

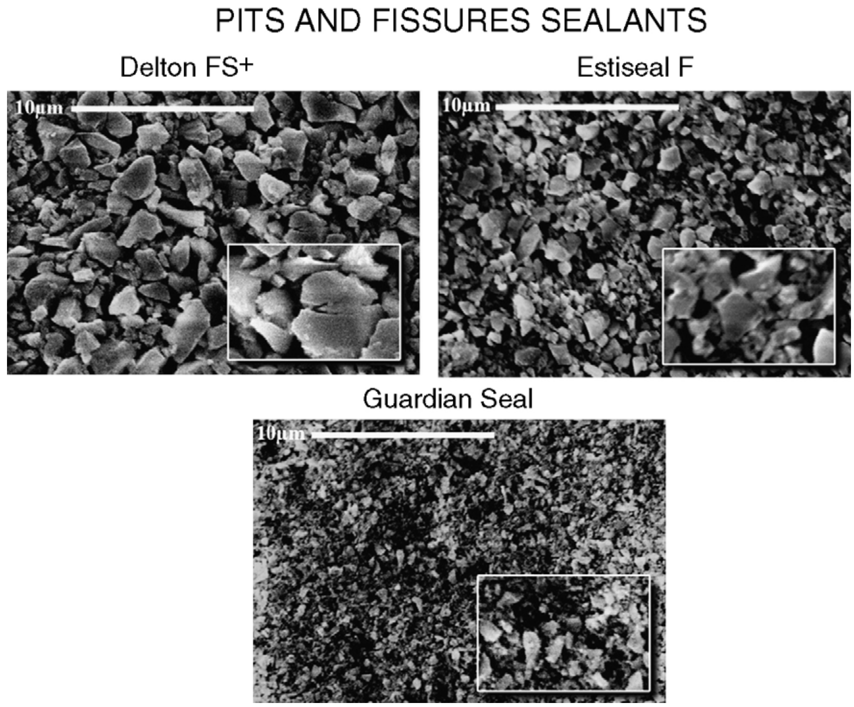

Fig. 9 – Fillers of the pit and fissure sealants by scanning-electron microscopy at 5000× magnification (10,000× in the inserts).

age modulus than all the other pit and fissure sealants even though Guardian Seal has more than twice its filler weight content (22.2% and 54.3%, respectively). No measurement of the evolution of the complex viscosity after destruction could be made because the values recorded were below the measurement threshold of the rheometer.

Most filler particles of the flowable resin composites are of irregular shape. Again, no strong correlation could be made between the particles' morphology and the rheological properties of the resin composites. However, spherical particles are found in Filtek Flow and Point-4 Flowable which both belong to the first group. A spherical shape is known to have many advantages such as allowing an increased filler load in composites [20] but also to enhance their fracture strength since mechanical stresses tend to concentrate on the angles and protuberances of the filler particles [21]. Irregular shaped particles were found in all the other composites tested. Admira Flow, Revolution Formula 2 and X-Flow showed bigger particles than the others. As for the flowable resin composites, no correlation could be made between the flow characteristics of the pit and fissure sealants and the morphology of their filler particles.

The viscosity of a resin composite is influenced by both the organic and the inorganic fraction. The type and amount of each monomer used are directly responsible for the viscosity of the organic matrix. This one is then strongly influenced by the amount, shape, size and surface treatment of the filler particles that are added to the composite. Since no correlation was found between the viscosity and the shape and weight content of filler particles, research should be undertaken to investigate the influence of various surface treatments of the particles that can influence their surface energy and consequently the physical and chemical interactions between particles and the organic fraction.

Since the filler particles content of pit and fissure sealants is pretty low, it is possible that their viscosity is much influenced by the composition of the organic fraction (much more than for the flowable resin composites), which differs considerably from one material to another. Unfortunately, only very poor information is available on the exact nature and content of the organic fraction of these materials.

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