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Dental Materials 18 (2002) 64–71

dental
materials

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Dynamic and static moduli of elasticity of resin-based materials

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Received 20 June 2000; revised 19 December 2000; accepted 30 January 2001

Abstract

Objectives: The purpose of this study was to assess and compare the elastic moduli of 34 resin-based materials using a dynamic and a static method. The effect of water storage was also studied up to 6 months.

Methods: Five samples of each material were prepared according to ISO-4049. The dynamic moduli were first determined non-destructively from the fundamental period of the vibrating specimen, then the static moduli were determined by a three-point bending test. The percentages of fillers by weight were determined by ashing in air at 900°C.

Results: Low values were obtained with flowable composites as well as with two packable resin composites. Correlations were found between the static and the dynamic modulus of elasticity ($r = 0.94$; $p = 0.0001$) as well as between the weight percentage of fillers and the moduli of elasticity ($r = 0.82$; $p < 0.05$ for static modulus and $r = 0.90$; $p < 0.05$ for the dynamic modulus) both at 24 h. Water storage significantly affected both static and dynamic moduli of elasticity (ANOVA two factors; $p < 0.05$).

Significance: The low moduli of the flowable composites do not allow their use in posterior cavities under high stress. However, this does not exclude their use for minimally-invasive Class I cavities when the opposing tooth is stabilized to a large amount on the natural enamel.The Grindosonic method is very useful and simple for determining the dynamic moduli although it gives higher values than the static one. The elastic modulus evolution of resin-based materials after water storage is unpredictable since different patterns were observed as a function of time. © 2002 Academy of Dental Materials. Published by Elsevier Science Ltd. All rights reserved.

Keywords: Resin-based materials; Modulus of elasticity; Water storage

1. Introduction

Never before in restorative dentistry, have so many different materials been presented as alternatives for cavity restorations [1]. First introduced as anterior restorative materials, resin composites are being used, more and more, for posterior restorations. In the last few years, along with the development of resin modified glass-ionomer formulations and polyacid-modified resin composites known as 'compomers', two new categories of resin composites have been developed, that are packable and flowable composites.

Elastic modulus describes the relative stiffness or rigidity of a material. It is measured by the slope of the elastic region of the stress–strain diagram [2]. Moreover, it is a function of the whole sample rather than of a weak part or of the surface only [3].

The elastic modulus and other mechanical properties as tensile strength, diametral tensile strength, fracture toughness etc., are important in determining the resistance to occlusal forces.

Elastic modulus as well as adhesive properties play an important role in preventing microleakage, secondary decay and/or filling dislodgement. For cervical cavities (Class V) composites must have a low modulus of elasticity to allow the composite to flex during tooth flexure. For occluso-proximal cavities (Class I and II) the modulus must be high to withstand deformations and cusp fractures [4].

Many dynamic and static methods have been used to measure the elastic modulus of resin composites. The three-point bending test is the most used among the static methods. It is described by the ISO-4049 [5]. Compressive modulus of elasticity has also been determined by drawing the compressive stress–strain curves [6,7] but values were lower than those obtained by three-point flexural test [7]. In 1961, Spinner and Tefft [8] described the mechanical resonance frequencies technique. Its main drawback was the contact of the driver and the pick-up with the sample, which interfered with the resonance frequency. Later, Braem et al. [3] used the fundamental period of the vibrating specimens. The samples were set in flexural instead of longitudinal vibration because it is

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0109-5641/02/$22.00 + 0.00 © 2002 Academy of Dental Materials. Published by Elsevier Science Ltd. All rights reserved.
PII: S0109-5641(01)00021-5

Table 1
List of materials and percentage of fillers by weight determined by ashing in air. VLC: Visible light cured, CC: chemically cured, CS: composite, CM: compomer, Flow: flowable, Pack: packable, Univ: universal, Hyb: hybrid

Material	Classification	Manufacturer	Batch and shade	% Fillers by weight
Aeliteflo	VLC Hyb Flow CS	Bisco, Inc Itasca, IL, USA	039317 (A3)	54.9
Amelogen	VLC Hyb Univ CS	Ultradent products, UT, USA	2CPM (A2)	72.9
Arabesk	VLC Hyb Univ CS	Voco, Cuxhaven, Germany	70500 (A3)	71.6
Arabesk-Flow	VLC Hyb Flow CS	Voco, Cuxhaven, Germany	82777 (A3)	61.8
Arabesk-Top	VLC Hyb Univ CS	Voco, Cuxhaven, Germany	81594 (A3)	71.5
Ariston-pHc	VLC ‘Smart Material’	Vivadent, Schaan, Liechtenstein	A00001 (–)	74.8
Brilliant-Dentin	VLC Hyb Univ CS	Coltène, Whaledent, Switzerland	GE931 (A3)	75.6
Brilliant-Enamel	VLC Hyb Univ CS	Coltène, Whaledent, Switzerland	GE902 (A3)	75.4
Charisma-F	VLC Hyb Univ CS	Heraeus Kulzer, Wehrheim, Germany	23 (A20)	76.4
Charisma-PPF	CC Hyb Univ CS	Heraeus Kulzer, Wehrheim, Germany	2 (A10)	68.3
Clearfil Photo Post	VLC Hyb Univ CS	Kuraray, Osaka, Japan	0035A (UL)	84.7
Clearfil Photo Ant.	VLC Hyb Univ CS	Kuraray, Osaka, Japan	0024C (A3)	59.9
Coltène-SE	VLC Hyb Univ CS	Coltène, Whaledent, Switzerland	FBJ01 (A3)	71.3
Concise	CC Conventional CS	3M, St. Paul, MN, USA	19970303 (U)	80.2
Dyract-Flow	VLC Flow CM	Dentsply De Trey, Konstanz, Germany	9809000103 (A2)	55.4
Elan	VLC CM	Sybron/Kerr, Orange, USA	805872 (A3,5)	71.2
EXI-119 (Z-250)[a]	VLC Hyb Univ CS	3M, St. Paul, MN, USA	030998 (A3,5)	77.4
EXI-120 (P-60)[a]	VLC Hyb Pack CS	3M, St. Paul, MN, USA	030998 (A3,5)	78.9
F-2000	VLC CM	3M, St. Paul, MN, USA	19970905 (A3)	80.5
Glacier	VLC Hyb Univ CS	Southern Dental Industries, Australia	60506 (B3)	78.2
Metafil-CX	VLC Microfine CS	Sun Medical, Shiga, Japan	71201 (A3,5)	41.7
Pertac-II	VLC Hyb Univ CS	Espe, Seefeld, Germany	00634764 (A3)	70.0
Polofil-Molar	VLC Hyb Univ CS	Voco, Cuxhaven, Germany	63596 (U)	78.5
Quadrant Anterior	VLC Microfine CS	Cavex Haarlem, Holland	22C (A2)	58.6
Quadrant Posterior	VLC Hyb Pack CS	Cavex Haarlem, Holland	30C (A2)	65.2
Revolution	VLC Hyb Flow CS	Sybron/Kerr, Orange, USA	710669 (A3)	53.9
Silux-Plus	VLC Microfine CS	3M, St. Paul, MN, USA	6DH (U)	54.8
Solitaire	VLC Hyb Pack CS	Heraeus Kulzer, Wehrheim, Germany	26 (A30)	64.3
Spectrum	VLC Hyb Univ CS	Dentsply De Trey, Konstanz, Germany	9608244 (A3)	75.3
Surefil	VLC Hyb Pack CS	Dentsply De Trey, Konstanz, Germany	980818 (A2)	79.4
Tetic-Ceram	VLC Hyb Univ CS	Vivadent, Schaan, Liechtenstein	900513 (A3)	75.7
Tetric-Flow	VLC Hyb Flow CS	Vivadent, Schaan, Liechtenstein	901232 (A3)	64.0
Wave	VLC Hyb Flow CS	Southern Dental Industries, Australia	80608 (A3)	60.7
Z-100	VLC Hyb Univ CS	3M, St. Paul, MN, USA	19960229 (UD)	79.6

[a] The P-60 (Posterior composite) and the Z-250 (Antero-Posterior composite), marketed by 3M were included in this study as experimental composites EXI-120 and EXI-119.

easier to excite flexural vibration, especially in thin specimens. The Dynamic Mechanical Thermal Analysis (DMTA) [9,10] and the ultrasonic method [11] have also been used. DMTA requires a complicated set-up, and the elevated range of temperature used can cause post-cure reaction, thus affecting the results. Nevertheless, glass-transition temperature can be determined by this method. A new technique for determining the elastic modulus during polymerization has been described by Meredith [12]. The authors measure the variations in resonance frequency of a system as the material undergoes polymerization.

The aim of this study was to measure the elastic moduli of some resin-based materials including flowable and packable composites, using a dynamic and a static method, and to establish a correlation between the percentages of fillers by weight and the moduli of elasticity. The effect of water storage and materials was studied up to 6 months.

2. Materials and methods

Thirty-four materials were tested in this study. They included 26 hybrid composites (16 universal, five flowable, four packable, one ‘smart’ restorative material), three microfine composites, two chemically-cured composites, and three compomers (two conventional and one flowable).

All the materials and their specifications are listed in Table 1. For both tests, five barshape specimens ($25 \times 2 \times 2$ mm) were prepared in metallic moulds. After filling the mould to excess, the material surface was covered with a mylar strip and a glass slide, then pressure was applied to extrude excess material. Specimens were light-cured by overlapping, as recommended in ISO-4049. Two hand-held curing lights (XL-3000, 3M; tip diameter 8 mm) were simultaneously used in order to enlarge the irradiated surface. Each overlap was light cured for 60 s. Then specimens were stored in distilled water at room temperature prior to testing for 24 h, 7, 28 and 180 days. The weight, width, length and height of each specimen were measured before testing.

2.1. Dynamic modulus

The elastic modulus was first measured non-destructively by the impulse excitation technique [3]. Each sample was set in flexural vibration by a light mechanical impulse. The signal produced was captured via a microphone located underneath the sample by a special signal analyser, the Grindosonic® (Lemmens Elektronics, Haasrode, Belgium). Fundamental frequency under flexure is displayed on the screen of the apparatus. Modulus of elasticity was calculated as follows:

$$E_d = 0.9465(mf_t^2/b)(L^3/t^3)T_1$$

where m is the mass of the bar, b is the width of the bar, L is the length of the bar, t is the thickness of the bar, f_t is the fundamental frequency of the bar in flexure and T_1 is a correction factor for fundamental flexural mode to account for finite thickness of the bar, Poisson's ratio and other constants. Poisson's ratio is defined as the absolute value of the ratio of transverse strain to the corresponding axial strain resulting from uniformly distributed axial stress below the proportional limit of the material [13]. Its value varies between 0.25 and 0.35. In this study 0.3 as the mean value has been chosen for all the materials.

2.2. Static modulus

The static modulus of elasticity was then measured by the three-point bending test, according to the ISO–4049 [5]. Samples were loaded in an Instron machine at a speed rate of 0.75 mm/min until fracture and modulus value was determined as $E_s = Fl^3/4bh^3d$ where F is the load, l is the distance between the support, b is the width of the bar, h is the thickness of the bar and d is the deflexion corresponding to load F.

2.3. Percentage of fillers by weight

The percentages by weight of inorganic fillers of all the materials were determined by the ashing technique. A porcelain crucible containing approximately 0.5 g of material was heated at 900°C for 1 h in a furnace (Heraeus K 1272, Germany, range of heating between 200 and 1125°C). The percentages of fillers by weight were determined by calculating the difference of weight of the crucible before and after ashing in air.

2.4. Statistical analysis

The mean values of the moduli of elasticity of the different materials were compared using a one-way ANOVA and post-hoc Scheffe's tests at $p < 0.05$ level. This was performed separately for each of the different storage time. Simple regression analysis was performed to study the relationship between both elastic moduli, and exponential regression analyses were performed to study the relationship between elastic moduli and percentage of fillers by weight. A two-way ANOVA (independent variables: materials and duration of storage) was used to assess the effect of storage.

3. Results

The mean values and the standard deviations of the dynamic moduli of elasticity of the materials tested are shown in Table 2. They ranged from 3.0 to 28.6 GPa.

Considerable differences were found between materials of the same category. The dynamic moduli of packable composites ranged between 8.3 GPa (Quadrant-Posterior) and 20.3 GPa (P-60), while for flowable composites they ranged between 4.6 GPa (Revolution) and 10.9 GPa (Tetric-Flow).

For the static moduli of elasticity of the materials tested, results are shown in Table 3. Values ranged from 1.4 to 18.5 GPa. They also showed differences between materials of the same category as packable composites showed elastic moduli ranging between 2.1 GPa (Quadrant-Posterior) and 11.9 GPa (Surefil).

Higher values were recorded for all the tested materials by the Grindosonic method (Fig. 1). The differences varied between 31 and 72% more than the static modulus. One-way ANOVA analysis showed, that Clearfil-Posterior had significantly the highest dynamic and static moduli of elasticity among the tested materials. The lowest values were obtained essentially with the flowable composites, microfine as well as with two packable composites.

The dynamic modulus of elasticity correlated significantly with the static modulus ($r = 0.94$; $p = 0.0001$) (Fig. 2).

The percentages of fillers by weight of the materials used in this study are shown in Table 1. High exponential correlation was found from regression analysis, between percentages of fillers and moduli of elasticity (Fig. 3) (static moduli $r = 0.82$; $p \leq 0.05$ and dynamic moduli $r = 0.90$; $p \leq 0.05$ at 24 h).

A significant materials–storage time interaction indicated that storage time influenced both static and dynamic moduli of elasticity of the tested materials (Table 4). Different patterns were observed between one day and 180 days, that made it unable to determine a uniform behavior for the materials. Water storage did not affect the correlations between the dynamic and static moduli of elasticity (linear regression with $p < 0.05$), neither the correlations between percentage of fillers by weight and both moduli (exponential correlation with $p < 0.05$).

4. Discussion

Understanding the mechanical properties of human teeth is important for the development of 'tooth-like' restorative materials [14]. According to Craig and Peyton [15], human

Table 2
Dynamic modulus of elasticity (GPa) expressed as mean values with standard deviation (SD). Values designed with the same letters are not significantly different (Anova-one factor)

Materials	Dynamic modulus of elasticity (GPa)			
	1 day	7 days	28 days	180 days
Aeliteflo	7.3 (0.2)^{a,b,c,d}	7.3 (0.3)^{a,b,c}	7.2 (0.4)^{a,b,c,d}	7.2 (0.1)^{b,c,d,e}
Amelogen	13.5 (0.3)^{h,i}	14.0 (0.2)^{g,h}	13.6 (0.4)^{g,h}	14.2 (0.3)^{i,j,k,l}
Arabesk	15.0 (0.6)^{i,j}	16.0 (0.4)^{i,j,k,l}	15.9 (0.4)^{h,i,j,k,l}	16.1 (0.4)^{j,k,l,m,n}
Arabesk Flow	9.7 (0.3)^{e,f,g}	10.0 (0.2)^{d,e}	10.4 (0.3)^{e,f}	9.5 (0.4)^{d,e,f,g}
Arabesk-Top	14.3 (0.4)^{i,j}	14.6 (0.3)^{h,i}	14.5 (0.3)^{g,h,i,j}	13.7 (0.2)^{h,i,j}
Ariston-pHc	16.3 (0.2)^{j,k,l}	16.8 (0.6)^{j,k,l}	16.4 (0.3)^{i,j,k,l,m}	15.1 (0.7)^{i,j,k,l,m}
Bril-Dent	16.2 (1.3)^{j,k,l}	16.9 (0.7)^{k,l}	17.3 (0.4)^{k,l,m,n}	17.5 (0.2)^{m,n}
Bril-Enam	16.1 (0.6)^{j,k,l}	16.4 (0.3)^{i,j,k,l}	17.1 (0.6)^{j,k,l,m,n}	17.0 (0.6)^{k,l,m,n}
Charisma-PPF	9.5 (0.5)^{d,e,f,g}	9.6 (1.0)^{d,e}	9.0 (1.2)^{c,d,e}	10.0 (0.2)^{e,f,g}
Charisma-F	14.3 (0.6)^{i,j}	14.8 (0.4)^{h,i,j}	14.5 (0.2)^{g,h,i,j}	15.0 (0.6)^{i,j,k,l,m}
ClearF P Ant	11.8 (0.3)^{g,h}	12.1 (0.2)^{f,g}	12.2 (0.3)^{f,g}	12.3 (0.5)^{g,h,i}
ClearF P Post	27.5 (0.2)^{o}	28.4 (0.6)^{q}	28.1 (0.7)^{r}	28.6 (1.0)^{q}
Coltène-SE	14.9 (0.8)^{i,j}	15.8 (0.6)^{h,i,j,k}	16.1 (0.3)^{h,i,j,k,l}	15.2 (0.7)^{i,j,k,l,m}
Concise	22.9 (0.8)^{n}	23.9 (0.6)^{p}	24.2 (0.4)^{q}	24.4 (0.6)^{p}
Dyract-Flow	9.1 (0.3)^{c,d,e,f}	8.7 (0.2)^{c,d}	8.7 (0.4)^{c,d,e}	9.6 (0.7)^{d,e,f,g}
Elan	16.6 (0.4)^{j,k,l}	18.6 (0.4)^{l,m,n}	17.0 (0.2)^{k,l,m,n}	17.3 (0.3)^{l,m,n}
F-2000	21.0 (1.3)^{n}	22.7 (0.8)^{p}	22.4 (0.3)^{p,q}	23.2 (1.5)^{p}
Glacier	14.4 (0.2)^{i,j}	14.4 (0.2)^{h,i}	14.2 (0.6)^{g,h,i}	13.9 (0.8)^{i,j,k}
Metafil-CX	6.9 (0.1)^{a,b,c}	6.4 (0.4)^{a,b}	6.7 (0.3)^{a,b,c}	6.5 (0.6)^{b,c,d}
P-60	19.9 (0.4)^{m,n}	20.3 (0.3)^{o}	19.6 (0.4)^{n,o}	18.5 (0.9)^{n}
Pertac-II	16.3 (0.4)^{j,k,l}	17.1 (0.7)^{k,l,m}	16.9 (0.4)^{j,k,l,m}	16.9 (0.5)^{j,k,l,m,n}
Polofil-Molar	18.1 (0.3)^{l,m}	19.2 (0.5)^{n,o}	18.5 (0.5)^{l,m,n}	18.7 (0.7)^{n}
Quad Ant	7.3 (0.3)^{a,b,c,d,e}	7.4 (0.3)^{a,b,c}	7.4 (0.4)^{a,b,c,d}	7.7 (0.4)^{b,c,d,e,f}
Quad Post	8.8 (0.3)^{b,c,d,e,f}	8.3 (0.5)^{b,c,d}	8.4 (0.4)^{b,c,d,e}	6.0 (0.9)^{a,b,c}
Revolution	5.0 (0.4)^{a}	5.6 (0.4)^{a}	5.5 (0.4)^{a}	4.6 (0.6)^{a,b}
Silux-Plus	8.9 (0.3)^{b,c,d,e,f}	9.2 (0.2)^{c,d,e}	8.6 (0.4)^{b,c,d,e}	8.8 (0.2)^{c,d,e,f}
Solitaire	8.7 (0.4)^{b,c,d,e,f}	9.1 (0.4)^{c,d,e}	9.3 (2.0)^{d,e}	8.6 (0.2)^{c,d,e,f}
Spectrum	15.4 (0.6)^{i,j,k}	16.0 (0.3)^{h,i,j,k,l}	17.3 (0.6)^{k,l,m,n}	16.0 (1.7)^{j,k,l,m,n}
Surefil	17.7 (0.7)^{k,l,m}	18.9 (0.3)^{m,n,o}	20.0 (0.9)^{o,p}	19.2 (0.5)^{n,o}
Tet-Ceram	14.3 (1.0)^{i,j}	15.4 (0.4)^{h,i,j,k}	15.7 (0.4)^{h,i,j,k}	15.1 (0.8)^{i,j,k,l,m}
Tet-Flow	10.7 (0.4)^{f,g}	10.8 (0.5)^{e,f}	10.9 (0.5)^{e,f}	10.6 (0.5)^{f,g,h}
Wave	6.5 (0.3)^{a,b}	5.4 (0.6)^{a}	6.1 (0.2)^{a,b}	3.0 (1.3)^{a}
Z-100	22.0 (0.4)^{n}	22.4 (0.3)^{p}	22.0 (0.4)^{o,p,q}	22.1 (0.2)^{o,p}
Z-250	18.5 (0.6)^{l,m}	19.1 (0.5)^{m,n,o}	19.0 (0.4)^{m,n}	18.0 (0.5)^{m,n}

dentin modulus is 18 GPa. This corresponds to an imaginary volume percentage of 60% of inorganic fillers [16]. Watts [17] reported a modulus of elasticity for dentin of 13 GPa determined in compression at body temperature. In a recent study, Xu et al. [14] measured the elastic modulus of human enamel and dentin by an indentation method; they obtained a mean value of 19 GPa for the dentin. Young's modulus of enamel exhibited a significant dependence on tooth orientation; for the occlusal section it was 94 GPa, while for the axial section it decreased to 80 GPa. Human enamel has anisotropic properties, that means that the values of measured physical properties differ upon the location of enamel prism. This could also affect the susceptibility of these crystallites to acid etch [18].

An optimal restoration should mimic structural, mechanical and physical characteristics of dentin and enamel, which could only be achieved if different restorative materials are combined.

Although there is no constant value in the literature for enamel and dentin modulus, it is obvious that the values obtained with the tested materials are far from those of enamel. Only Clearfil-Posterior showed a static modulus approaching that of dentin (18.5 GPa); the other universal hybrid composites had lower values with wide variations of both moduli of elasticity.

A composite material with a high modulus of elasticity, if combined with high fracture parameters such as flexural strength, diametral tensile strength and fracture toughness, would be able to resist occlusal loads and provide support at the interface.

As opposed to the high values obtained with some materials, low moduli were recorded with flowable composites. Those materials are hybrid composites, with lower filler load [19]. Their moduli values were 50 to 85% less than other hybrids, depending upon the selected material. In the present study, two couples of resin composites, issued respectively from the same manufacturer, a universal versus a flowable (Tetric Ceram/Flow and Arabesk Top/Flow) were tested. Supposing that those materials have a similar chemistry, the main difference would be their filler load that is reduced by

Table 3
Static modulus of elasticity (GPa) expressed as mean values with standard deviation (SD). Values designed with the same letters are not significantly different (Anova-one factor)

Materials	Static modulus of elasticity (GPa)			
	1 day	7 days	28 days	180 days
Aeliteflo	3.4 (0.2)^{a,b,c,d}	3.8 (0.2)^{a,b,c}	4.0 (0.4)^{a,b,c,d,e}	4.1 (0.1)^{a,b,c,d,e}
Amelogen	5.9 (0.3)^{b,c,d,e,f,g,h,i,j}	7.9 (0.5)^{d,e,f,g,h,i}	7.3 (0.5)^{d,e,f,g,h,i,j}	8.1 (0.7)^{f,g,h,i,j}
Arabesk	8.3 (0.5)^{h,i,j,k,l,m,n}	7.5 (1.2)^{d,e,f,g,h}	9.2 (0.5)^{g,h,i,j,k}	9.0 (2.3)^{f,g,h,i,j}
Arabesk Flow	4.3 (0.2)^{a,b,c,d,e,f,g}	5.5 (0.4)^{a,b,c,d,e}	5.9 (0.3)^{b,c,d,e,f,g,h}	5.4 (0.3)^{b,c,d,e,f}
Arabesk-Top	6.4 (0.3)^{c,d,e,f,g,h,i,j,k}	7.0 (0.7)^{c,d,e,f,g}	7.7 (0.5)^{f,g,h,i,j}	6.5 (0.3)^{d,e,f,g,h,i}
Ariston-pHc	6.6 (0.7)^{d,e,f,g,h,i,j,k}	7.6 (0.5)^{d,e,f,g,h}	8.0 (0.6)^{f,g,h,i,j}	6.6 (1.2)^{d,e,f,g,h,i}
Bril-Dent	8.7 (1.2)^{i,j,k,l,m,n}	9.7 (0.5)^{f,g,h,i,j}	9.4 (0.6)^{h,i,j,k}	10.8 (0.9)^{j,k}
Bril-Enam	11.1 (2.8)^{m,n,o}	9.5 (0.2)^{f,g,h,i,j}	10.7 (0.7)^{j,k,l}	9.5 (0.7)^{h,i,j}
Charisma-PPF	4.7 (0.6)^{a,b,c,d,e,f,g,h}	4.9 (0.9)^{a,b,c,d}	4.5 (0.4)^{a,b,c,d,e,f}	5.7 (0.3)^{b,c,d,e,f,g}
Charisma-F	7.9 (0.6)^{g,h,i,j,k,l,m,n}	6.9 (0.2)^{c,d,e,f,g}	7.0 (0.6)^{d,e,f,g,h,i}	7.7 (0.5)^{e,f,g,h,i,j}
ClearF P Ant	7.7 (0.4)^{g,h,i,j,k,l,m,n}	7.6 (0.6)^{d,e,f,g,h}	7.7 (0.3)^{f,g,h,i,j}	8.2 (0.5)^{f,g,h,i,j}
ClearF P Post	18.0 (1.2)^{p}	17.7 (0.9)^{l}	18.5 (1.4)^{n}	15.2 (1.7)^{m}
Coltène-SE	7.4 (1.8)^{e,f,g,h,i,j,k,l,m}	8.6 (0.5)^{e,f,g,h,i}	8.1 (0.5)^{f,g,h,i,j}	9.1 (0.9)^{f,g,h,i,j}
Concise	11.1 (1.0)^{m,n,o}	12.3 (1.6)^{j,k}	12.7 (1.0)^{k,l,m}	16.4 (1.1)^{m}
Dyract-Flow	4.4 (0.3)^{a,b,c,d,e,f,g}	4.7 (0.2)^{a,b,c,d}	5.0 (0.4)^{a,b,c,d,e,f}	4.1 (0.2)^{a,b,c,d,e}
Elan	8.4 (0.6)^{h,i,j,k,l,m,n}	10.2 (0.9)^{g,h,i,j,k}	10.8 (0.9)^{j,k,l}	9.3 (1.1)^{g,h,i,j}
F-2000	11.3 (0.6)^{n,o}	12.4 (1.8)^{j,k}	13.2 (3.3)^{l,m}	14.6 (1.8)^{l,m}
Glacier	7.6 (0.8)^{f,g,h,i,j,k,l,m,n}	6.1 (0.4)^{b,c,d,e,f}	6.2 (0.3)^{c,d,e,f,g,h}	6.7 (0.5)^{d,e,f,g,h,i}
Metafil-CX	2.6 (0.3)^{a,b}	2.6 (0.1)^{a,b}	2.6 (0.1)^{a,b}	2.1 (0.1)^{a,b}
P-60	9.4 (0.8)^{j,k,l,m,n,o}	9.3 (1.0)^{f,g,h,i,j}	10.8 (0.8)^{j,k,l}	8.5 (0.9)^{f,g,h,i,j}
Pertac-II	9.1 (0.7)^{j,k,l,m,n}	10.0 (1.1)^{g,h,i,j,k}	10.0 (0.3)^{i,j,k,l}	11.3 (0.7)^{j,k,l}
Polofil-Molar	10.5 (0.9)^{l,m,n,o}	10.2 (0.9)^{g,h,i,j,k}	10.5 (0.6)^{i,j,k,l}	11.2 (0.4)^{j,k,l}
Quad Ant	3.7 (0.2)^{a,b,c,d,e}	3.6 (0.1)^{a,b,c}	3.8 (0.3)^{a,b,c,d}	3.5 (0.1)^{a,b,c,d}
Quad Post	2.7 (0.2)^{a,b,c}	2.9 (0.6)^{a,b}	3.2 (0.2)^{a,b,c}	2.2 (0.3)^{a,b,c}
Revolution	1.4 (0.2)^{a}	2.1 (0.2)^{a}	2.2 (0.2)^{a}	2.1 (0.2)^{a,b,c}
Silux-Plus	5.0 (0.3)^{a,b,c,d,e,f,g,h,i}	5.5 (0.4)^{a,b,c,d,e}	5.6 (0.1)^{a,b,c,d,e,f,g}	5.4 (0.4)^{b,c,d,e,f}
Solitaire	3.8 (0.2)^{a,b,c,d,e,f}	2.8 (0.3)^{a,b}	2.8 (0.2)^{a,b,c}	3.6 (0.4)^{a,b,c,d}
Spectrum	10.4 (0.1)^{k,l,m,n,o}	9.5 (1.6)^{f,g,h,i,j}	10.7 (0.6)^{j,k,l}	9.3 (1.0)^{g,h,i,j}
Surefil	9.4 (1.0)^{j,k,l,m,n,o}	10.9 (0.1)^{h,i,j,k}	12.0 (0.4)^{k,l,m}	9.6 (0.6)^{i,j,k}
Tet-Ceram	6.9 (0.5)^{d,e,f,g,h,i,j,k,l}	8.0 (0.4)^{d,e,f,g,h,i}	7.5 (0.5)^{e,f,g,h,i,j}	7.7 (0.5)^{e,f,g,h,i,j}
Tet-Flow	4.3 (0.3)^{a,b,c,d,e,f,g}	5.1 (0.3)^{a,b,c,d,e}	5.3 (0.2)^{a,b,c,d,e,f}	5.8 (0.4)^{c,d,e,f,g,h}
Wave	1.9 (0.1)^{a}	1.9 (0.1)^{a}	2.1 (0.2)^{a}	1.4 (0.3)^{a}
Z-100	13.2 (1.2)^{o}	13.6 (1.8)^{k}	14.6 (0.9)^{m}	13.2 (0.6)^{k,l,m}
Z-250	10.7 (1.2)^{l,m,n,o}	11.5 (0.3)^{i,j,k}	10.8 (0.8)^{j,k,l}	9.4 (0.7)^{g,h,i,j}

about 15% for the flowable composites (Table 1). As expected from the correlation curves (Fig. 3), flowable composites had lower moduli of elasticity (nearly 20–30% less) than universal ones for both static and dynamic moduli.

Concerning flowable composites, no clinical trials have yet been published. However, manufacturers claim many advantages for these new materials. Their low moduli of elasticity and increased flow capacity might provide more contraction stress relaxation, and could reduce the frequency of marginal microleakage and possible de-bonding. Some practitioners advise their use in the deep parts of class II cavities under hybrid resin composites or as shock absorbers (e.g. abfraction lesions) [20]. On the other hand, Labella et al. [21] recently demonstrated that these materials tend to shrink more than the non-flowable composites. The authors also studied the elasticity of some resin-based materials at 24 h, including Aeliteflo, Revolution, Tetric-Flow, Z-100 and F-2000. Elastic moduli were determined using the Grindosonic, and the results were very similar to those found in the present investigation. For Silux-Plus, the data were in accordance with those of Irie and Nakai [22].

Table 4
Two-way ANOVA with materials and storage time as independent variables for both dynamic and static moduli. p-value <0.05 is significantly different

Source	df	p-value	
		Dynamic modulus	Static modulus
Materials (A)	33	0.0001	0.0001
Storage (B)	3	0.0001	0.0001
Interaction (A × B)	99	0.0001	0.0001

The two chemically-cured composites showed wide variations between their static and dynamic moduli. The moduli of Concise was nearly 2.5 times higher than that

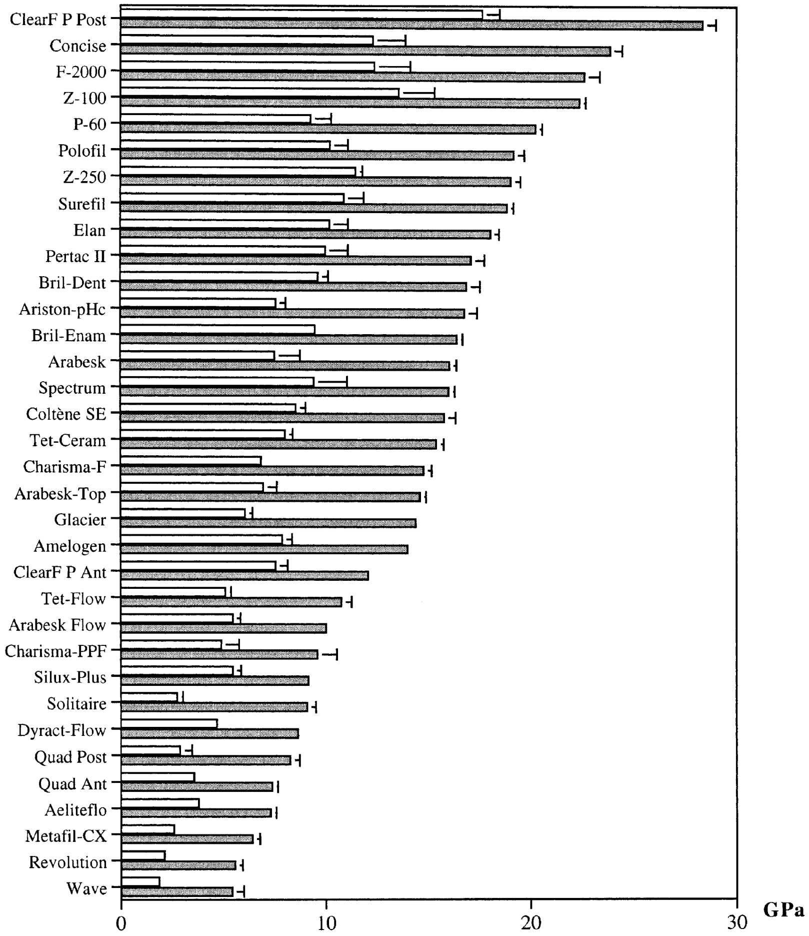

Fig. 1. Bar chart showing differences between the static (☐) and the dynamic (▨) modulus of elasticity. Materials are ranked in decreasing order according to their dynamic modulus of elasticity at 7 days.

of Charisma-PPF. Again this wide variation can be correlated to the wide difference of percentages of fillers between the two materials (Table 1).

Packable resin composites, except Solitaire and Quadrant Posterior, had moduli of elasticity comparable to universal hybrid composites. According to the manufacturer, those two materials have a high percentage of fillers by volume (both 90%). However, in this study they showed a low percentage by weight (Solitaire 64.3% and Quadrant Posterior 65.2%). This high percentage of fillers by volume may be due to the structure of the fillers used because these two materials contain highly porous SiO_2-glass fillers that could act like voids or defects. As expected by the high positive correlations found between the percentage of fillers by weight and moduli of elasticity, those materials showed low dynamic and static moduli (Tables 2 and 3). This correlation observed between these parameters, is in accordance with Braem et al. [3], and highlights the influence of fillers on physical properties.

Static modulus of elasticity was highly correlated to the dynamic one ($r = 0.94$; $p < 0.0001$; $y = 0.629x - 1.586$) (Fig. 2). All the materials tested showed a higher dynamic than static modulus of elasticity.

The high values obtained with the dynamic method are due to the high strain rate applied on the specimen during testing [3]. However, the Grindosonic method has several advantages and differences when compared to the static loading technique. The test method is non-destructive and can be used for specimens prepared for other tests and for those with complex geometry. The modulus of elasticity is measured at or near the origin of the stress–strain curve, with the minimum possibility of fracture and there is no requirement for a complex set-up [13]. The data obtained with the Grindosonic have low standard deviations because the frequency is recorded only when three consecutive and similar (lying within 1% of each other) values are obtained. Although having rather the same standard deviations (Tables 2 and 3), the coefficients of variation are lower for the dynamic modulus.

When looking at the Young's modulus as a function of time, it can be noticed that the dynamic moduli undergo small variations compared to the static ones. Statistical analysis (ANOVA two-factors) showed that materials and water storage are factors affecting significantly both dynamic and static moduli of elasticity ($p < 0.05$).

Most materials showed an increase of their moduli

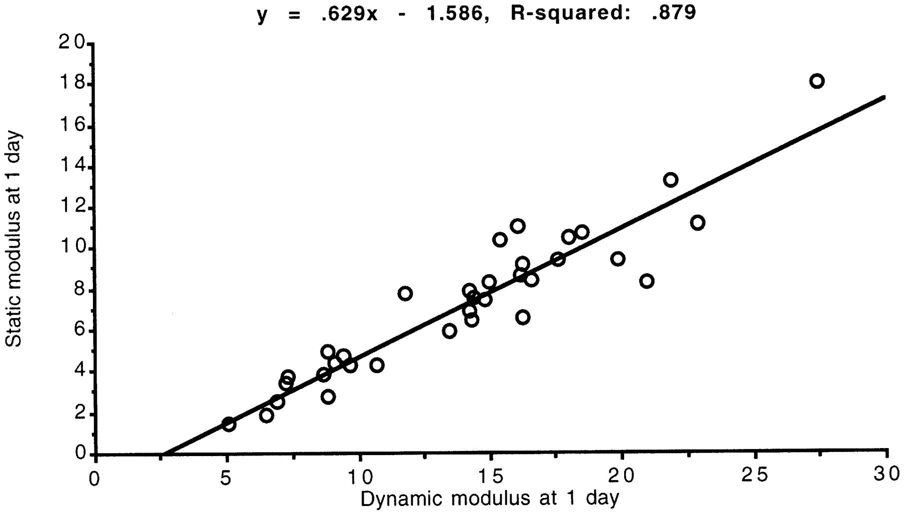

Fig. 2. Correlation between static and dynamic moduli of elasticity (GPa) at 1 day. Linear regression ($r = 0.94$; $p = 0.0001$).

between 24 h and 1 month and a relative stability up to 6 months.

In the literature, authors do not always agree about the effect of water storage as there were conflicting trends of increase and decrease over time. According to Gladys et al. [23] the Young's modulus increased slightly during the first month and then showed a slight decrease to reach a steady state, while for Ferracane et al. [24,25] composites experienced a slight but significant decrease in their modulus after aging in water for 30 days and approached a plateau at 6 weeks. Ferracane et al. [24,25] found that Z-100 did not change significantly, whereas Gladys et al. [23] reported a decrease in its modulus. Drummond et al. [26] noticed a decrease in the modulus of elasticity after aging in water up to 12 months, but the aging influence was only significant between 0 and 6 months.

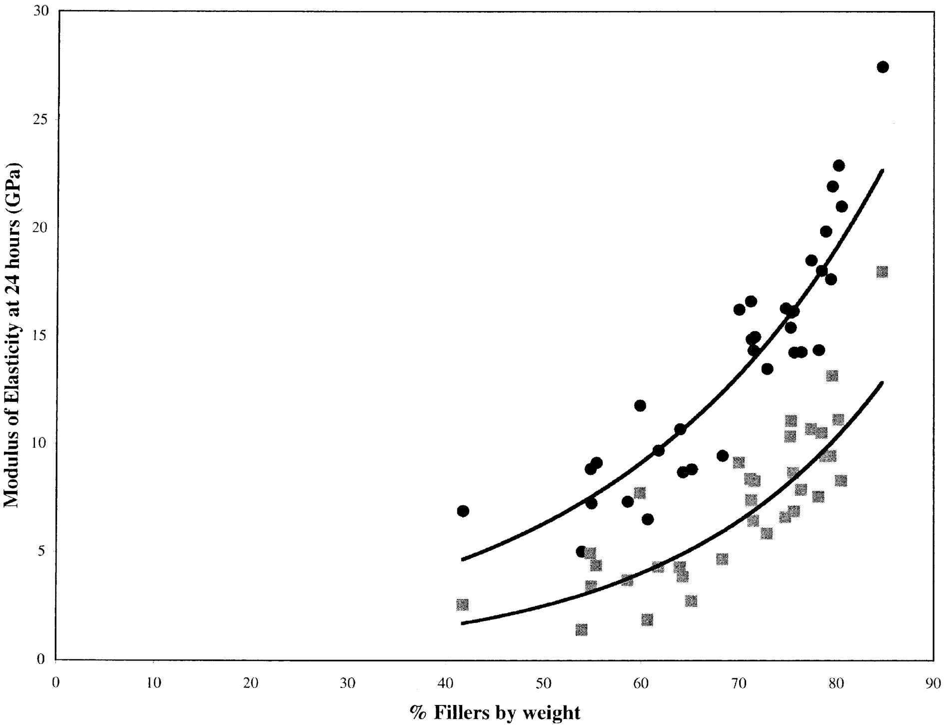

Fig. 3. Correlations between percentages of fillers by weight and modulus of elasticity at 1 day. Exponential regression curves showed the following equation respectively for the dynamic modulus (●) and static modulus (■): $Y_d = 0.875e^{0.0391x}$, with $r = 0.9$ and $p \leq 0.05$ and, $Y_s = 0.2791e^{0.0457x}$ with $r = 0.82$ and $p \leq 0.05$.

However, all agree that the reason for these variations is mainly due to water uptake that is a diffusion controlled process [22–27]. Water seems to have a plasticizing effect on the matrix, and causes degradation of the filler–matrix interface [27]. After 6 months, the modulus reaches a stable state and no further reduction in properties occurs as the polymer network is saturated with water.

In the present study, compomers showed behavior similar to that of resin composites, except F-2000 that showed a slight increase (statistically non significant) of its elastic modulus up to 6 months. According to the manufacturers, the continuous increase in the acid–base reaction or maturation process for up to 6 months indicates that the process may continue for some time. However, it has been observed that compomers have no self-curing mechanism [28]. Those materials may contain one or both of the essential components of glass-ionomer cements but at a level too low to promote the acid–base reaction [29], so their main cure system is light activated.

5. Conclusion

This in vitro study compared two ways of measuring modulus of elasticity of different categories of resin-based materials. A wide range of moduli of elasticity were observed among these categories of tested materials. Flowable and microfine composites as well as flowable compomer, showed the lowest values of dynamic and static modulus of elasticity.

The Grindosonic method, although giving higher values, is simple, reliable and non-destructive, so the sample can be examined more than once. Correlations were observed between both static and dynamic moduli of elasticity. Percentages of fillers were highly correlated with both elastic moduli, and water storage significantly affected all the tested materials.

Acknowledgements

The authors are very grateful to Professor Paul Lambrechts for reviewing this paper. They also thank the manufacturers for providing materials.

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