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Correlation of filler content and elastic properties of resin-composites

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ABSTRACT

Objectives. The aim was to determine the Young's modulus (E), bulk modulus (B), shear modulus (G) and Poisson's ratio (ν) of a series of composite restorative materials and to correlate them with their filler volume-fractions.

Methods. Twelve model resin-composite formulations, with systematically varied volume-fraction (Tokuyama), a flowable resin-composite (Point 4 flowable, Kerr) and two hybrid resin-composites (Filtek Supreme XT, 3M-Espe & X-tra Fil, Voco) were investigated. Twelve cylindrical specimens (5 mm × 6 mm) were prepared from each material. Six were free to expand radially under axial compressive loading, and were used to calculate the Young's modulus (E). The other six were radially constricted in a rigid stainless steel ring during loading, from which the bulk modulus (B) was calculated. Compression loading was performed at 1 mm/min. The Young's and bulk moduli were determined using equations of elasticity. Poisson's ratio from $\nu = 0.5 - (E/6B)$ and shear modulus from $G = E/2(1 + \nu)$.

Results. Young's moduli ranged from 2.19 to 7.15 GPa, bulk moduli from 12.79 to 22.43 GPa and shear moduli from 0.74 to 2.47 GPa. Poisson's ratio ranged from 0.45 for the stiffer to 0.47 for the more compliant composites. Statistically significant differences (ANOVA and Bonferroni at $p = 0.05$) were found depending on filler volume-fraction.

Significance. Elastic moduli varied significantly and a positive correlation existed between elastic moduli and filler volume-fraction (r^2 : 0.905–0.992 and 0.940–1.000 for Young's and bulk moduli, respectively).

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1. Introduction

The introduction of nanotechnology opens up new vistas in the formation and evolution of improved materials. The mechanical performance of nanometer-size reinforcements may in some circumstances be superior to their micron-size counterparts [1]. Dental polymeric restorative materials are now incorporating nanofillers (5–100 nm) and/or organic–inorganic hybrids. The aim has been to improve

mechanical properties, surface smoothness and gloss, polymerization shrinkage and biocompatibility [2].

Filler size is only one of several parameters affecting the overall properties of a resin-composite. The filler type, shape and amount, as well as the efficient coupling of fillers and resin matrix, contribute to the material performance. Properties such as compressive or flexural strength, hardness and Young's modulus improve as the filler content increases. At the same time polymerization shrinkage decreases. However, in

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some cases an optimum value can be reached at a certain filler volume-fraction and then declines with any further increase of the volume-fraction [3].

Understanding structure–properties relationships is valuable in assessing existing materials and developing new ones. Elastic moduli are key parameters in describing the deformation behavior of materials. Poisson's ratio is essential in determining other properties such as biaxial flexure strength and indentation modulus [4]. Moduli and Poisson's ratio are also basic input parameters for all components in computational Finite Element Analysis (FEA) of dental structures [5,6].

In many restorative materials the assumption of linear elasticity can be applied if they are uniform, homogeneous and isotropic [7,8]. Particulate resin-composites are considered isotropic materials as the filler particles are homogeneously embedded in the matrix and linked with the matrix phase through the coupling agent [9]. This link should be strong enough to allow sufficient stress transfer from the matrix to the filler particles. Different moduli, such as Young's, shear or bulk, arise from different modes of stress application. However, in the case of linear elasticity, these are inter-related via Poisson's ratio, the ratio of lateral to axial strain [5]. A full comprehension of elastic behavior requires the values of all the aforementioned moduli.

According to linear elasticity theory Young's (E), bulk (B), shear (G) moduli and Poisson's ratio (ν) are inter-related via the following equations [7,8]:

$$\nu = 0.5 - \frac{E}{6B} \quad (1)$$

$$\nu = \frac{3B - 2G}{6B + 2G} \quad (2)$$

$$G = \frac{E}{2(1 + \nu)} \quad (3)$$

$$\frac{1}{E} = \frac{1}{3G} + \frac{1}{9B} \quad (4)$$

$$B = \frac{E}{3(1 - 2\nu)} \quad (5)$$

Thus, experimentally obtained values of only two parameters are sufficient to permit the calculation of the other two, by using these equations.

Several different experiments have been used to determine Young's modulus and also Poisson's ratio. Some of these procedures are static tests [3–7,10–20] and other dynamic tests [21–32].

Some mathematical models based on, but also improving, the older Voigt's and Reuss/Angew's Rules of Mixtures, have also been developed to predict Young's modulus of composite materials [9,33,34]. However, data existing in the literature, for bulk and shear moduli of dental polymeric restorative materials are incomplete.

In this study a static method was adopted, which has the advantage of the concurrent determination of both Young's and bulk moduli. It was previously proposed and used by Warfield et al. [35] and Chabrier et al. [7]. The aim was to determine the full elastic behavior of several polymeric

Table 1 – Material composition according to the manufacturers

Material code	wt. %	vol. %	Filler size (nm)	Filler shape	Filler type	Matrix	Manufacturer
A 55	55	40	80	Spherical	SiO ₂ -TiO ₂ (2:1)	Bis-GMA/TEGDMA 60:40 (w/w)	Tokuyama, Tokyo, Japan
A 60	60	45					
A 65	65	50					
A 67	67	52					
B 55	55	38	150	Spherical	SiO ₂ -ZrO ₂ (2:3)	Bis-GMA/TEGDMA 60:40 (w/w)	Tokuyama, Tokyo, Japan
B 60	60	43					
B 65	65	48					
B 67	67	50					
C 55	55	37	450	Spherical	SiO ₂ -ZrO ₂ (2:4)	Bis-GMA/TEGDMA 60:40 (w/w)	Tokuyama, Tokyo, Japan
C 60	60	42					
C 65	65	47					
C 67	67	49					
Point 4 flowable	70	48	400	Spherical	Ba Si glass	TEGDMA, EBPADMA	Kerr, Orange CA, USA
Filetek Supreme XT (hybrid mini-fill)	78.5	59.5	20 (600–1400)	Aggregates or spherical	SiO ₂ and ZrO ₂ /SiO ₂ nanocluster aggregates	Bis-GMA, UDMA, TEGDMA, bis-EMA	3M-Espe, St. Paul, MN, USA
X-tra fil (hybrid midi-fill)	86	70.1	2000–3000	Irregular	Ba B Al Si glass	Bis-GMA, UDMA, TEGDMA	Voco, Cuxhaven, Germany

Fig. 1 – Mould used for specimen fabrication.

restorative materials. The specific objectives were (i) to measure experimentally Young's and bulk moduli and (ii) to calculate Poisson's ratio and shear modulus using the above equations. The null hypotheses tested were (i) that there were no differences among the elastic moduli of the materials and (ii) that there was no correlation between the elastic moduli and the filler volume-fraction.

2. Materials and methods

2.1. Materials

Twelve model composite systems with a systematically varied volume-fraction and size of fillers and three commercial ones were studied (Table 1). The latter were a flowable resin-composite, a hybrid mini-filled resin-composite and a hybrid midi-filled resin-composite.

Twelve cylindrical specimens 5 mm × 6 mm were prepared from each material by injecting them in a stainless steel divisible mould. Two microscopic glass slides, covered with transparent polystyrene matrix strips, were used to compress the material in the mould (Fig. 1). The samples were initially irradiated for 60 s from each surface with a halogen light curing unit (Elipar Trilight, 3M-ESPE, Seefeld, Germany) operated in standard mode and emitting 550 mW/cm² irradiance. This was as measured with the incorporated radiometer and calibrated with a Molectron™ power meter. The small excess of material was removed by using a stainless steel blade applied parallel to the mould's surfaces. After the specimen removal from the mould an additional 7 min light cure under vacuum was performed in a curing chamber (Visio Beta Vario, 3M-Espe, MN, USA). Finally, specimens were sonicated in an ultrasonic water bath for 2 min to remove any remaining debris. A digital caliper was used to measure the dimensions to ±5 μm before and after loading. The samples were stored dry at 23 °C and loaded 24 h after fabrication.

2.2. Methods

A Zwick/Roell Z020 Universal Testing Machine (Zwick GmbH & Co. KG, Ulm, Germany) was used for loading the specimens. The compliance of the machine was measured prior to the performance of the actual tests by running a loading cycle using exactly the same attachments and adjustments which would

Fig. 2 – Experimental arrangement for determination of bulk modulus.

be used at the real experiment, but without placing samples. The stress–strain curves (correction curves) that resulted from this procedure were uploaded to the software during the real test and the compliance of the machine was automatically subtracted to generate the corrected stress–strain curves from the overall measured displacement. Two correction curves were produced, one for the arrangement used for the determination of the Young's moduli and the other for the bulk moduli measurements.

Specimens of each material were divided into two groups. Group A ($n=6$) were loaded, totally unconstrained, with a gradual load up to 1000 N to obtain Young's modulus. The crosshead speed was set at 1 mm/min. Group B ($n=6$) were loaded between pistons at the same rate with a gradual load up to 10,000 N, but this time radially constrained in a close-fitting rigid stainless steel ring, to obtain bulk modulus. The ring bore was only slightly wider (0.05 mm) than each specimen diameter, to allow their insertion in the ring but not their expansion (Fig. 2).

Young's and bulk moduli were obtained from the slope of the stress/strain curve at the higher loads for the unconstrained and constrained specimens respectively, using graphics software (Sigmaplot 8.0) (Fig. 3a and 4a).

The statistical analysis was performed by using the SPSS (13.0.0) statistical software. The mean values of the experimentally obtained moduli of each material were analyzed by using one-way ANOVA at a level of significance 0.05, in order to accept or reject the null hypothesis. The Bonferroni *post hoc* multiple comparison test was used to define the statistically significant differences among the groups. Moreover, a regression and correlation analysis was performed to investigate any possible correlation between the filler volume-fraction and the elastic moduli.

3. Results

Fig. 3a is a representative stress/strain plot of the compression of a radially unconstrained specimen (of each material, but in this case of Filtek Supreme XT). This plot was used for the determination of Young's modulus. To confirm that the elastic deformation region was not exceeded, a regression and correlation analysis was performed for the data at the higher

Fig. 3 – (a) Representative stress–strain graph of a radially unconstrained sample (Filtek Supreme XT—specimen 2). (b) Magnification of the final part of the previous graph which was used for the calculation of the Young's modulus.

loads. For those data, since the coefficient of determination was almost 1 ($r^2 = 0.999$), essentially all the measured points were on the same line, so the final part of the graph was linear. Thus, the tangent of the angle formed between the regression line and the horizontal axis in Fig. 3b, corresponded to the Young's modulus (E) for the unconstrained specimens. For this particular specimen of Filtek Supreme XT, E was 5.88 GPa.

Similarly, Fig. 4a is a representative stress/strain plot of the compression of a radially constrained specimen (in this case, of B 55). This plot was used for the determination of bulk modulus (B). On these stress/strain plots for the constrained specimens, there was a fairly abrupt change in gradient (arrowed). This region corresponds to the transition where the specimen becomes completely pressed against the constraining walls, preventing further radial expansion. The transition-region was not completely abrupt, probably due to the combined effects of specimen barreling and microscopic roughness of the specimen perimeter. From that transition, the gradient represented the bulk modulus. Definitive values for the bulk moduli were taken from a region some way past that transition regime, as illustrated in Fig. 4a. Accordingly the tangent of the angle formed between the regression line and the horizontal axis in Fig. 4b corresponded to the bulk modu-

Fig. 4 – (a) Representative stress–strain graph of a radially constrained sample (B 55—specimen 5). The arrow indicates a transition-region beyond which the gradient represents the bulk modulus. (b) Magnification of the final part of the previous graph which was used for the calculation of the bulk modulus.

lus for the constrained specimen. For this particular specimen of B 55, B was 12.84 GPa.

The mean values of Young's and bulk moduli of all the materials tested are presented in Table 2. Young's moduli ranged from 2.19 ± 0.26 GPa for B 55 to 7.15 ± 0.36 GPa for X-tra Fil and bulk moduli from 12.79 ± 0.29 GPa for A 55 to 22.43 ± 0.42 GPa again for X-tra Fil. Analysis of variance revealed statistically significant differences among the groups. A general trend was observed: materials with higher filler load had higher Young's modulus. A similar tendency was observed for bulk moduli.

Poisson's ratio for each material was derived by substituting in Eq. (1) the mean values of Young's and bulk moduli. Similarly, the shear modulus was calculated by substituting in Eq. (3) the mean value of the Young's modulus and Poisson's ratio for each material (Table 3). Poisson's ratios ranged from 0.45 for the stiffer to 0.47 for the more compliant composites. Shear moduli ranged from 0.74 GPa for B 55 to 2.47 GPa for X-tra Fil. As the filler volume-fraction increased the shear moduli also increased, whilst Poisson's ratio decreased.

A strong positive correlation was found between Young's moduli and filler volume-fraction of each series (r^2 was between 0.905 and 0.992) (Fig. 5a–d). Bulk moduli also corre-

Table 2 – Mean Young's and bulk modulus (standard deviation) of the materials tested

Material	Young's modulus (S.D.) (GPa)	Bulk modulus (S.D.) (GPa)
A 55	2.46 (0.09) abc	12.79 (0.29) A
A 60	3.22 (0.12) abcde	13.26 (0.53) AB
A 65	4.25 (0.59) ef	14.83 (0.32) DEF
A 67	4.77 (0.25) fg	14.91 (0.53) EF
B 55	2.19 (0.26) a	12.86 (0.42) A
B 60	2.91 (0.63) abcd	13.77 (0.30) ABCD
B 65	3.41 (0.81) bcde	14.45 (0.52) CDEF
B 67	3.27 (0.58) abcde	15.14 (0.29) EF
C 55	2.36 (0.22) ab	13.01 (0.37) A
C 60	2.87 (0.47) abcd	13.54 (0.84) ABC
C 65	3.63 (0.35) de	14.86 (0.52) EF
C 67	3.91 (0.98) def	15.45 (0.59) F
Point 4 flowable	3.52 (0.87) cde	14.27 (0.74) BCDE
Filtek Supreme XT	5.76 (0.39) g	18.58 (0.60) G
X-tra Fil	7.15 (0.36) h	22.43 (0.42) H

Different letters denote statistically significant differences among the groups.

Table 3 – Poisson's ratio and shear modulus of the materials tested

Materials	Poisson's ratio, $\nu = 0.5 - (E/6B)$	Shear modulus (GPa), $G = E/2(1 + \nu)$
A 55	0.47	0.84
A 60	0.46	1.10
A 65	0.45	1.46
A 67	0.45	1.65
B 55	0.47	0.74
B 60	0.47	0.99
B 65	0.46	1.17
B 67	0.46	1.12
C 55	0.47	0.80
C 60	0.47	0.98
C 65	0.46	1.24
C 67	0.46	1.35
Point 4 flowable	0.46	1.21
Filtek Supreme XT	0.45	1.99
X-tra Fil	0.45	2.47

Fig. 5 – (a) Correlation of filler volume-fraction of the A series and Young's modulus. (b) Correlation of filler volume-fraction of the B series and Young's modulus. (c) Correlation of filler volume-fraction of the C series and Young's modulus. (d) Correlation of filler volume-fraction of Point 4 flowable/Filtek Supreme XT/X-tra Fil and Young's modulus.

Fig. 6 – (a) Correlation of filler volume-fraction of the A series and bulk modulus. (b) Correlation of filler volume-fraction of the B series and bulk modulus. (c) Correlation of filler volume-fraction of the C series and bulk modulus. (d) Correlation of filler volume-fraction of Point 4 flowable/Filtek Supreme XT/X-tra Fil and bulk modulus.

lated with filler volume-fraction of each series (r^2 was between 0.940 and 1.000) (Fig. 6a–d).

4. Discussion

All resin-composites used in this study incorporated in the resin matrix small filler particles, in the nanometer range. Some materials had only fillers of one size (A, B, C series and Point 4 flowable), while others a mixture of more than one size (Filtek Supreme XT and X-tra Fil). The shape of the fillers also varied, as most of the materials had spherical fillers (A, B, C series, Point 4 flowable and Filtek Supreme XT) while one had irregular fillers (X-tra Fil).

The combination of relatively small and varied size fillers allows a more dense packing, which in turn increases the possible filler volume-fraction of the resin-composites. Moreover, the spherical shape, especially in a mixture of different sizes, favors geometrically the incorporation of more inorganic fillers in the resin matrix [18,36]. It also improves the

fracture strength of the materials because stresses tend to develop preferably at sharp edges of the fillers [36]. Nevertheless, the reduction of the size cannot be unlimited for several reasons. Technical difficulties exist in the production of very small fillers which must be fabricated by pyrolytic processes rather than by grinding down larger fillers. The very small fillers, by providing a bigger surface for the same volume, also tend to increase significantly the viscosity of the material. This happens due to the formation of more secondary polar bonds with the resin matrix. The mixing with the resin matrix is also affected and becomes more difficult. Finally the very small particles, usually silica, tend to agglomerate and form larger aggregates of about 0.04–0.4 μm in size [37].

Special care was given to ensure sufficient curing of the materials. Incomplete polymerization may produce a heterogeneous specimen which in turn may affect the stress distribution during loading [4]. Before testing, specimens were stored dry for 24 h to provide enough time for any post-polymerization and the development of optimum mechanical properties. Young's modulus can increase substantially in

some cases upon storage due to the post-polymerization procedure [23]. Contrary, wet storage was shown to decrease Young's modulus even after only 24 h [28]. This decline was attributed to the water plasticizing effect on the resin matrix that may also degrade the filler–matrix interface. Wet storage can also allow the elution of unreacted components and affect the magnitude of the elastic moduli [4]. Another factor that can greatly decrease the mechanical properties and the elastic moduli is the presence of any pores in the samples [21,29,38].

As it is evident from Table 2 and Fig. 5a–d, Young's modulus showed a strong correlation with filler volume-fraction ($r^2=0.905\text{--}0.992$). It increased progressively and almost linearly more than three times (2.19–7.15 GPa) from the less filled (38 vol.%) to the more filled materials (70.1 vol.%). This observation is in agreement with the conclusions of many other studies [3,4,9,11,23].

Materials with similar filler volume-fraction had similar, and not significantly different, Young's moduli. Among these materials a weak trend was noticed, that materials with larger filler sizes tended to have slightly higher Young's moduli. By this observation it can be inferred that the filler size, as a sole factor, cannot affect greatly Young's modulus, particularly when the size range of fillers is relatively small. However, it may affect it indirectly if the filler fraction is increased by using small and variant size distributions of fillers [9,11].

Concerning bulk modulus, the differences among the materials were smaller. The highest value registered (22.43 GPa) was less than two times greater than the lowest (12.79 GPa). Bulk moduli were rather more strongly correlated with the filler volume-fraction than Young's moduli (Fig. 6a–d).

It was not possible to define a definite relation between either the composition or the shape of the fillers and the two moduli. Materials including hard glasses, like ZrO_2 , were ranked both low and high in terms of elastic moduli. The moduli also seemed to be independent of filler shape. These two factors were assumed to indirectly influence the elastic moduli by influencing the maximum filler loading [11].

Many different experimental methods have been used for the determination of the elastic moduli of resin-composites. Some are static and some dynamic. Some mathematical models also exist which have been used for the prediction of the elastic moduli. In most static experiments, uniaxial stress is applied to the material and Poisson's ratio is calculated from the resulting transverse and axial deformation. In some dynamic measurements, an ultrasound (resonance) is commonly applied to the materials and Poisson ratio is computed from the velocity or natural frequency of the transverse and longitudinal waves generated [4]. Factors such as the test method (compression, tension, flexure or shear), the strain rate, the efficiency of polymerization, etc. have a considerable impact on the values of the elastic moduli [39]. Generally the value of Young's modulus decreases with decreasing strain rate, or by using lower frequencies in dynamic ultrasound tests [13,40]. The direct comparison of the values obtained by different methodologies would be incorrect. The elastic moduli measurements must be evaluated along with the experimental parameters [4,7,21].

All techniques have advantages and disadvantages. Several dynamic methods are simple and non-destructive but need

special equipment and are sensitive to the correct positioning of the sample on the supports as well as to the geometry, porosity and density of the samples [4,29]. Static methods have their own complications especially because small strains are difficult to record [29] and require high stresses to produce sufficient deformation [4].

The results of this study are comparable with results of other studies using static methodologies. Boyer et al. [12] used compression at a crosshead speed of 0.5 mm/min, to evaluate some mechanical properties of 10 conventional and micro-filled resin-composites. Young's moduli varied from 2.81 to 8.90 GPa. Chabrier et al. [7] also performed a compression test at low strain rate (crosshead speed 0.2 mm/min). This study, along with one by Warfield et al. [35], constituted the basis for the present study and had similar methodology. Chabrier et al. found Young's moduli from 5.88 to 7.09 GPa for the resin-composites tested, bulk moduli between 10.99 and 16.52 GPa and Poisson's ratios between 0.40 and 0.44. These values are in the range obtained from the present study. Minor differences may be attributed mainly to the wider range of filler-loading in the materials studied (Table 1) and partly to the fivefold greater strain rate used presently. Chung et al. [4] used a tensile test at a crosshead speed of 0.5 mm/min and they found Poisson's ratios between 0.30 and 0.39, using strain gauges for measuring transverse and axial deformation of their samples. Three-point flexure tests were employed by Braem et al. [40], Kim et al. [16,18] and Beun et al. [36]. Braem et al. quoted Young's moduli between 2.88 and 14.00 GPa, Kim et al. [16] values between 1.69 and 4.01 GPa, Kim et al. [18] values between 3.40 and 15.00 GPa and Beun et al. values between 2.60 and 14.20 GPa, always depending on the particular parameters and the materials used in each study.

The dynamic ultrasound methods produce higher values of Young's moduli and consequently lower Poisson's ratios, which are attributed to differences in the deformation rate of the specimens [4,7,13,40]. The high frequencies used in these studies are equivalent to very high strain rate deformation of materials in static methods. Braem et al. [23] reported Young's moduli between 5.44 and 25.12 GPa. A year later the same group of researchers [40] published the results obtained by two different dynamic techniques; the one used in their previous study which was the Fundamental Period Technique (FPT) and another, Dynamic Mechanical Thermal Analysis (DMTA). By the FPT they found Young's moduli from 6.09 to 25.12 GPa, while with DMTA they reported frequency-dependent results. For exactly the same materials they reported values ranging from 2.83 to 14.13 GPa at 0.1 Hz, from 3.29 to 14.79 GPa at 1 Hz and from 3.84 to 15.49 GPa at 10 Hz. Especially these results highlight the significance of the *dynamic frequency* of Young's modulus determination, due to the viscoelastic components of behavior. Further studies have employed dynamic procedures: Willems et al. [26] reported Young's moduli between 5.44 and 27.38 GPa; Labella et al. [27] reported values between 4.53 and 22.35 GPa; Abe et al. [28] reported values between 13.3 and 19.7 GPa; Beun et al. [36] reported values between 6.3 and 26.3 GPa. However, intra-oral deformation rates are low, circa 1 Hz. Thus, the most appropriate values may be those from either static or low-frequency dynamic-mechanical measurements.

5. Conclusions

- The elastic moduli of the materials tested did vary significantly. Thus, the first null hypothesis was rejected
- A strong positive correlation was demonstrated between Young's and bulk moduli and the filler volume-fractions. Thus, the second null hypothesis was rejected

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