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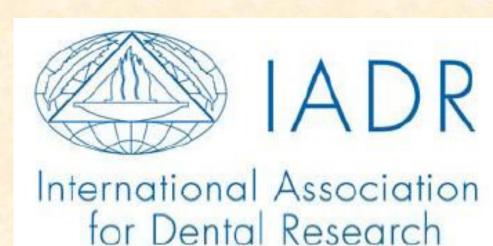
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RHEOLOGY TESTS ON CORE BUILD-UP MATERIALS AND CLINICAL IMPLICATIONS

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INTRODUCTION Reconstruction of endodontically treated posterior teeth use now dual or chemical cure core build-up materials. Core build-up materials have mechanical properties still unknown. These properties have consequences on survival of reconstruction and on the tooth. For the same material family, the operator feelings may be very different depending on resin flowability. The aim of this study was to evaluate the viscosity (processing properties) and some mechanical properties (in service properties) of different resins. For example:

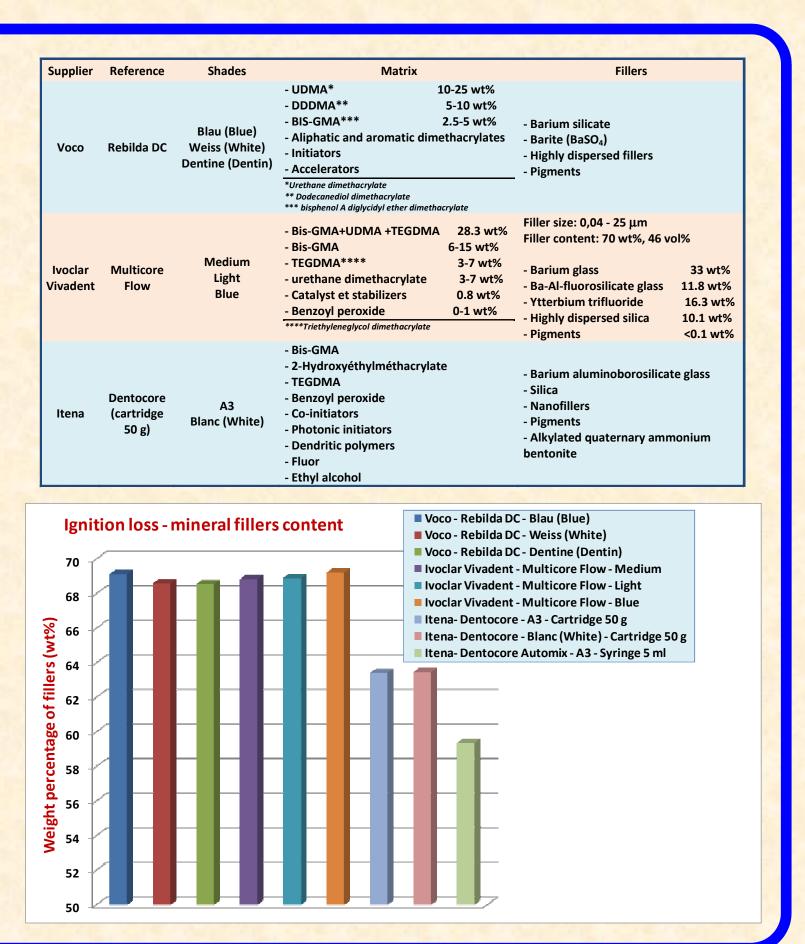
- → If the resin is very viscous it is difficult to fill the root canal
- → If the resin is very flowable, injection is facilitated but what about the mechanical properties (stiffness, yield stress, hardness, ...)?
- The resins properties were obtained from shear rheometry, flexural vibration analysis, micro-indentation and micro-hardness.

MATERIALS Several dental dual cure resin composites were studied. They were supplied by ITENA (Dentocore - A3, White), Ivoclar Vivadent (Multicore Flow - Medium, Light an Blue), Voco GmbH (Rebilda DC - Blau (blue), Weiss (white) and Dentine (dentin)), DMG (LuxaCore Automix dual - A3), Bisico (Best Core DC - A3) and Coltène Whaledent (ParaCore Automix - dentin). They were selected owing to their different behaviours (especially "flowability") during core build-up processing. All these resins are available in 50 ml cartridges. Data about the composition (list of organic and inorganic ingredients) of the resins are available from the suppliers' instructions for use or(and) Safety Data Sheets (see Table on the right).

All are based on mixtures containing monomers such as Bis-GMA ("high" viscosity about 700 Pa.s at 25 °C) and diluents monomers such as TEGDMA (about 8 mPa.s at 25 °C) or DDDMA (about 15 mPa.s at 20 °C). Voco and Ivoclar Vivadent contain also UDMA monomer ("intermediate" viscosity about 7 Pa.s at 23 °C). For instance, addition of TEGDMA tends to decrease notably the viscosity of the resin composite. But drawbacks are usually higher shrinkage and higher water absorption.

The inorganic fraction contains different nature of fillers which act as viscosity and mechanical properties enhancers, or for radio opacity.

The filler content of each resin composite was measured by ignition loss. About 0.4 g of each resin is pyrolised at 625 °C for 3 h. The fraction left is assumed to be only composed of mineral fillers. Other procedures such as thermogravimetric analysis are known to give the same results [Sabbagh, 2004]. For instance, the results (see Fig. on the right) show no difference between Voco and Ivoclar Vivadent (about 69 wt%). For Itena the result is about 63.5 wt%. Moreover, for Ivoclar Vivadent resin composites the value matches the supplier's data (70 wt%).



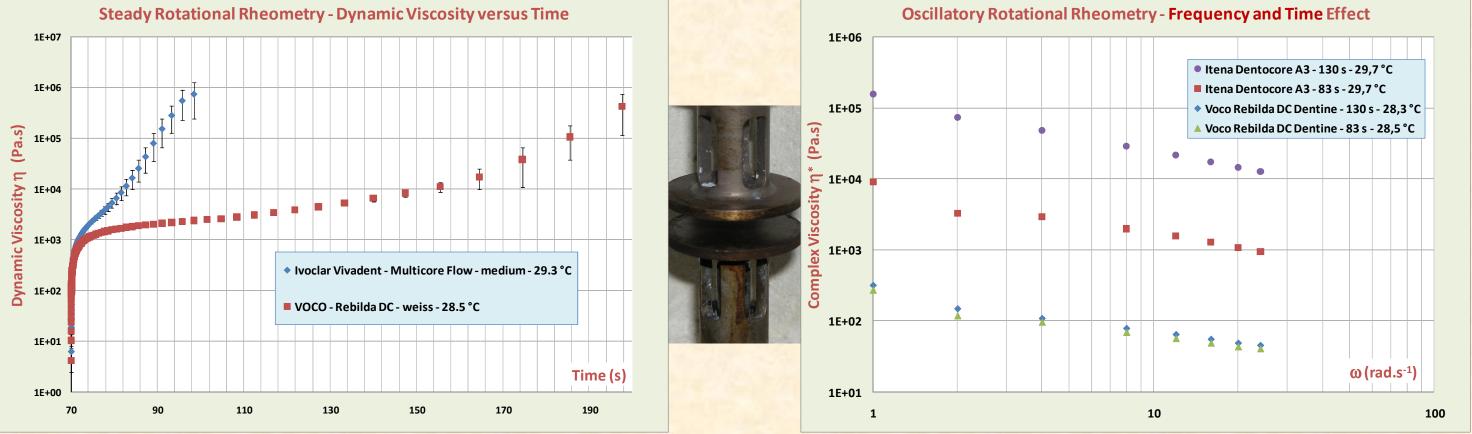
EXPERIMENTAL

RHEOLOGY Rotational rheometry experiments were carried out with a TA Instruments ARES rheometer. A parallel plates (25 mm diameter, 1 mm gap) geometry was used. For the whole tests materials were conditioned for 45 min at ambient before being extruded through a static mixing tip from 50 ml cartridges with a dispenser gun type 3. The delay before the first measurement is about 70 s. The materials undergo only chemical curing during the test.

Two kinds of experiments are carried out:

. Steady: time sweep (shear rate 0.05 s⁻¹). The dynamic viscosity is estimated for 2 materials. Ivoclar Vivadent Multicore Flow medium, assumed to be "viscous" and Voco Rebilda DC weiss, assumed to be "flowable". Results are given below (each curve is the mean of 3 experiments; standard deviations are indicated by vertical bars). Time in abscissa represents the delay from the deposit of the material onto the geometry just after mixing the two components. Ivoclar and Voco resins have clearly different behaviours. The increase in viscosity is much faster for Ivoclar than for Voco. This is a result that confirms the operator feelings. Moreover it is consistent with the working times of the different resins (120 s for Voco Rebilda DC and 90 - 120 s for Ivoclar Vivadent Multicore Flow).

No definitive explanation can be given except the weight fraction of viscous monomer Bis-GMA is much higher in Multocore Flow (about 15 %) than in Rebilda DC (2.5 - 5 %) whereas the filler content is the same (69 wt%).



2. Oscillatory: frequency sweep (1 - 24 rad.s⁻¹). Effect of delay from mixing is also estimated (83 and 130 s). The strain amplitude is 1 %. Two resins were tested: Itena Dentocore A3 and Voco Rebilda DC Dentine. Results (complex viscosity) are given above. Both materials are shear-thinning, which is quite usual for this kind of mineral filled materials [Lee, 2006] [Beun, 2009]. Moreover these ones reveal very different behaviours:

- → At the same time Itena is much more viscous than Voco,
- → The complex viscosity of Itena varies much more rapidly than viscosity of Voco.

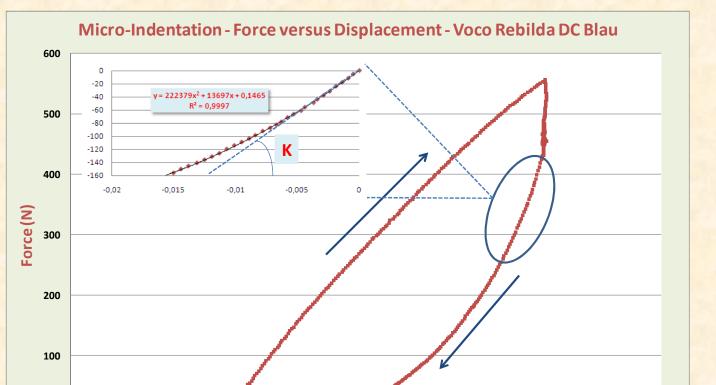
MICRO-INDENTATION Three resins from Voco (blau, weiss and dentine shades) were characterized by mean of micro-indentation experiments. Thus information is given about Young's modulus of the material.

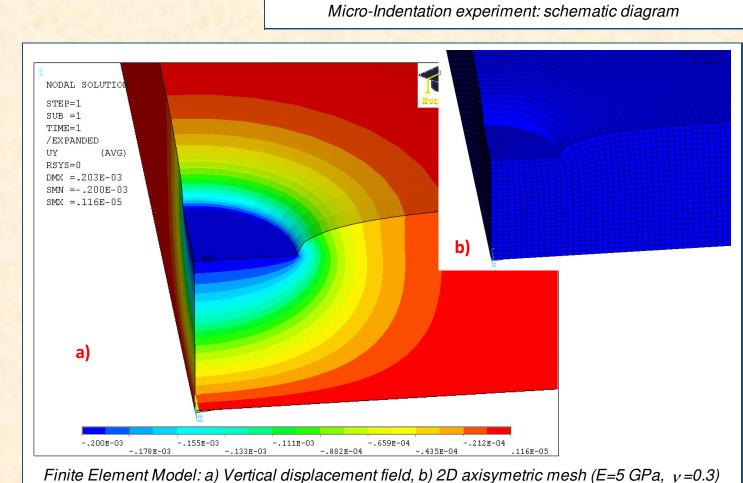
The disk-shaped sample is 1 mm thick and 25 mm in diameter. The test is composed of 3 steps: 1. Loading: a compression is applied by the punch (1 mm diameter) at a controlled speed of 0.1 mm.min⁻¹. The maximum displacement and force are respectively 0.2 mm and 100 N

2. Relaxation: constant displacement (0.2 mm) for 2 min 3. Unloading: the punch is driven back to its initial position

The tangential stiffness K is measured at the beginning of the unloading

(polynomial regression on the 30 first points) (see Fig. below).





Flat cylindrical

Displacement Δl

Then the Young's modulus of the material is estimated thanks to a 2D axisymetric Finite Element Model (FEM) (see Fig. above) under the assumptions:

Displacement (mm)

(1) the material is purely elastic (Hooke's law), (2) the sample lays on a perfectly rigid substrate, (3) the punch and the sample are coaxial, (4) The boundary conditions are: -0.2 mm displacement along

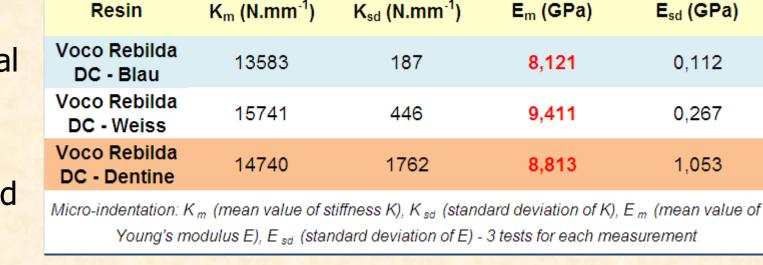
the contact punch/sample and no vertical displacement in contact with the substrate. Finally the relation between the Young's modulus E (GPa) and the stiffness K (N.mm⁻¹) is $E = \frac{K - 0.0095}{1672.5}$

Results: the results are given in the table below. They are rather consistent with values obtained from dynamical tests.

Several causes of errors could be identified: 1. The FEM model used is simplified. The material

is supposed elastic whereas an elastoplastic

strain is likely to occur 2. The stiffness of the material could be increased at a strain level about 20 %



FLEXURAL VIBRATION ANALYSIS

characterize the elastic behaviour of structures or simple geometry samples (bars, plates, beams, ...). In this study several resin composites from Voco (Rebilda DC -Weiss and Dentin) were characterized. Beams 80x2.3x2.5 mm³ sized were molded and light cured according to the supplier's

indications then chemical cured for 15 more minutes. Method: the sample is fixed as a cantilever on an exciter which induces vertical oscillations at a variable frequency (see Figures on the right).

→ Hypotheses:

- The material is elastic, homogeneous and isotropic (Hooke's law), - The deflection of the beam is governed by the Bernoulli condition,

- The damping of the material is neglected.

The equation of motion [Chevalier, 2002] is $\frac{\partial^4 y}{\partial x^4} + \frac{\rho S}{EI} \frac{\partial^2 y}{\partial t^2} = 0$ [1] (x: longitudinal axis, I: flexural inertia, y(x,t): vertical deflection, E: Young's modulus, t: time). If the solution is written as: $y(x,t)=Y(x)e^{i\omega t}$ [2], then equation of motion gives $\frac{d^4Y}{dx^4}-\frac{\beta^4}{L^4}Y=0$ [3].

β is the adimensional wave number given by: $\beta^4 = \frac{\rho SL^4\omega^2}{FI}$ [4] and $\omega = 2\pi f$ (f: frequency). The general solution is then: $Y(x) = Ach(\beta x) + Bsh(\beta x) + Ccos(\beta x) + Dsin(\beta x)$ [5].

Taking account of boundary conditions $(y(0)=0, \frac{dy}{dx}(0)=0)$ clamping, $\frac{d^2y}{dx^2}(0)=0$ null bending moment,

 $\frac{d^3y}{dx^3}(0)=0$ null vertical shear force) gives: $ch(\beta)\cos(\beta)+1=0$ [6] which gives an infinite set of solution. Each solution is associated with an amplitude resonance of the beam, an eigen mode and an eigen frequency. For each eigen mode the Young's modulus E can be deduced from: $E = \frac{\rho SL^4 \omega^2}{I\beta^4} = C \frac{\rho f^2 L^4}{h^2}$ [7].(

 $C = \frac{48\pi^2}{\beta^4}$, $\rho = \frac{m}{V}$: material's density, $f = 2\pi\omega$: resonance frequency of the first eigen mode, L: vibrating length of the sample, h: height of the sample). In the first mode: $\beta_1 = 1,875$

In each test the cantilever sample is excitated with an increasing frequency until the first eigen mode

is reached. Then the value of E is calculated according to equation [7]. Results: the results are given in the table below.

Frequency E (GPa) ρ (kg.m⁻³) L (m) Sample 9,066 ± Voco Rebilda DC - Blau 1838 0,050 330 0,725 $9,094 \pm$ Voco Rebilda DC - Weiss 0,050 330 1844 0,728

MICRO-HARDNESS Cylindrical-shaped samples were prepared with metallic molds

(4 mm diameter- 2 mm thickness). Five samples were prepared for each experimental group (n=5). Dual-cured resin-composites were inserted into the molds and covered with acetate strips (Hawe-Neos Dental, Bioggio, Switzerland). A 1 mm thick glass slide was placed over the acetate strips and manual

pressure was applied to extrude excess material. Light curing of the materials was carried out using a quartz tungsten halogen (Elipar Trilight: 3M/Espe company) operating at 800 mW.cm⁻² with the tip of the light curing unit positioned in direct contact

with the glass slide. The specimens were positioned centrally beneath the indenter of a micro-hardness tester (MVK H2 from Mitutoyo company) and a 500 gf load was applied through the indenter with a dwell time of

20 seconds. One reading was performed for each sample. Dual-cured resin materials were light cured for different exposure times: 10, 20, 40 and 60 seconds. Microhardness readings were delayed for 24 hours.

			90		,						
	Time (s)>		10		20		40		60		
Supplier	Reference	Shade	m (sd)		m (sd)		m (sd)		m (sd)		
Bisico	Best Core DC	А3	46,06 (0,56)	С	46,02 (0,58)	С	45,32 (0,67)	b	46,40 (0,68)	b	NS
Itena	Dentocore	А3	26,62 (1,55)	d2	28,78 (0,46)	d2	30,46 (1,01)	c2	34,66 (0,77)	с1	p=0,0006
DMG	Luxacore	А3	52,58 (0,51)	а	53,20 (0,60)	а	52,96 (0,81)	а	53,38 (1,35)	а	NS
Ivoclar Vivadent	Multicore Flow	Medium	50,06 (0,80)	b2	51,70 (0,58)	b1	51,76 (0,98)	a1	51,46 (0,88)	a1	p=0,05
Coltène Whaledent	Paracore	Dentin	44,68 (1,10)	c2	54,18 (0,55)	a1	55,08 (1,52)	a1	54,18 (1,57)	a1	p=0,008
Voco	Rebilda DC	Dentine	45,32 (1,05)	с1	47,16 (0,48)	c2	46,66 (0,38)	b2	46,96 (0,85)	b2	p = 0,01
		n <0 0001		n<0.0001		n<0.0001		n<0.0001			

specimens were stored dry in lightproof containers at 37 °C. After 24 hours samples were submitted to Vickers hardness testing.

Results (mean and standard deviations and statistical analysis) are given in the table above. A statistical analysis was carried out with Kruskal-Vickers micro-hardness at 24 h versus exposure time (or energy)

Wallis and Games-Howell tests (p-value of less than 0.05). Analysis results determine three types of behaviours for the different resins: → Resins with final hardness independent of light

Light curing unit with light densities

of 8 J.cm⁻² (10 s), 16 J.cm⁻² (20 s),

32 J.cm⁻² (40 s), and 48 J.cm⁻²

(60 s) were used to light-cure the

specimens. After light curing, the

exposure time: LuxaCore® (DMG, Hamburg, Germany) and BestCore (Bisico, Lançon de Provence, France)

→ DentoCore (Itena resin, Paris, France) seems to reveal a hardness evolution close to a linear function of time. Moreover its final hardness is the lowest among the different tested materials

→ Some resins MultiCore Flow (Ivoclar Vivadent, Schaan, Liechtenstein) Paracore (Coltène Whaledent, Altstätten, Switzerland) and Rebilda DC (Voco, Cuxhaven, Germany) need at least 20 s light exposure time to reach their final hardness value. That is a typical behaviour of dual cure resins.

CONCLUSION The various tests showed differences between the resins. All this Core build-up materials belong theoretically to the same family of materials and should have similar physical properties.

- → Flowability is clearly different from one resin to another
- > Final Vickers micro-hardness at 24 h and its dependence on the light exposure time is also clearly different for the various tested resins. It can induce different behaviours in dental milling and operator filling during core build-up

These differences show that clinically endodontically treated reconstructions may not have the same rheological behaviour, mechanical properties and impact on quality and longevity of reconstructions. The overall analysis of the results suggests the establishment of a manufacturers common standard to dispose equivalent materials in this clinical application.

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BIBLIOGRAPHY

composites used in this work.