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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

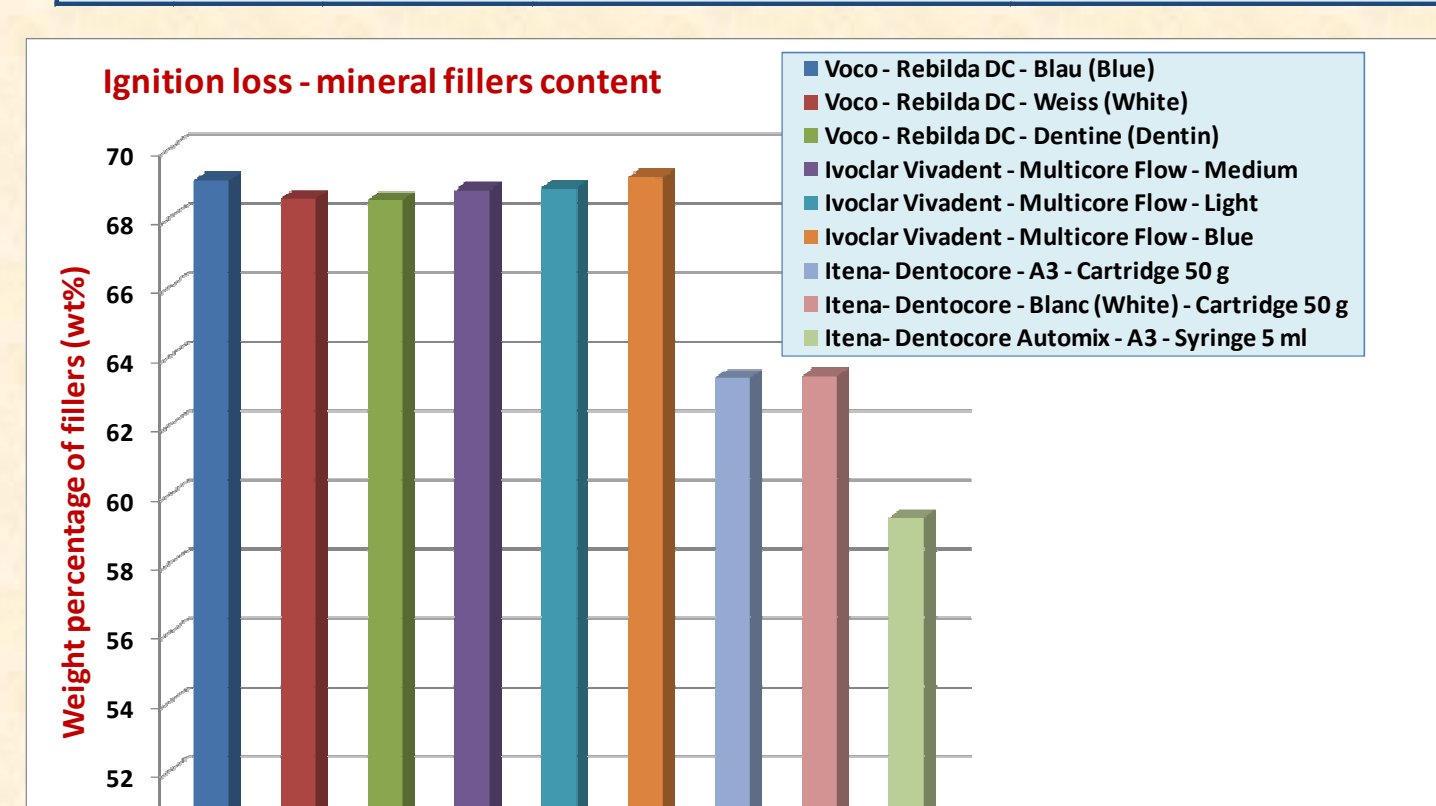
Several dental **dual cure** resin composites were studied. They were supplied by ITENA (Dentocore - A3, White), Ivoclar Vivadent (Multicore Flow - Medium, Light and Blue), Voco GmbH (Rebilda DC - Blau (blue), Weiss (white) and Dentine (dentine)), DMG (LuxaCore Automix dual – A3), Bisico (Best Core DC – 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 pyrolysed 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%).

Supplier	Reference	Shades	Matrix	10-25 wt%	Fillers
Voco	Rebilda DC	Blau (blue) Weiss (white) Dentine (dentine)	-UDMA* -DDMA** -Bis-GMA*** -Aliphatic and aromatic diacrylate/polyacrylate -Carboxylic acid -Photoinitiators	5-20 wt% 5-15 wt%	-Barium silicate -Barite (BaSO <sub>4</sub> ) -Highly dispersed fillers -Fillers
Ivoclar Vivadent	Multicore Flow	Medium Light Blue	-Bis-GMA -UDMA -TEGDMA -Urethane dimethacrylate -Carboxylic acid -Benzoyl peroxide	26-3 wt% 6-25 wt% 2-7 wt% 0.2 wt% 0.1 wt%	-Barium glass -Zirconium silicate -Highly dispersed silica -Fillers -Filler size: 0.04 - 25 µm -Filler content: 70 wt%, 44 wt%
Itena	Dentocore (cartridge 50 ml)	A3 Blanc (White)	-Bis-GMA -UDMA -TEGDMA -Urethane dimethacrylate -Carboxylic acid -Photoinitiators -Diethyltoluene diisocyanate -Ethyl acrylate	26-3 wt% 6-25 wt% 2-7 wt% 0.2 wt% 0.1 wt%	-Barium silicate -Barite -Alumina -Silica -Highly dispersed amorphous boron -Fillers -Filler content: 63.5 wt%



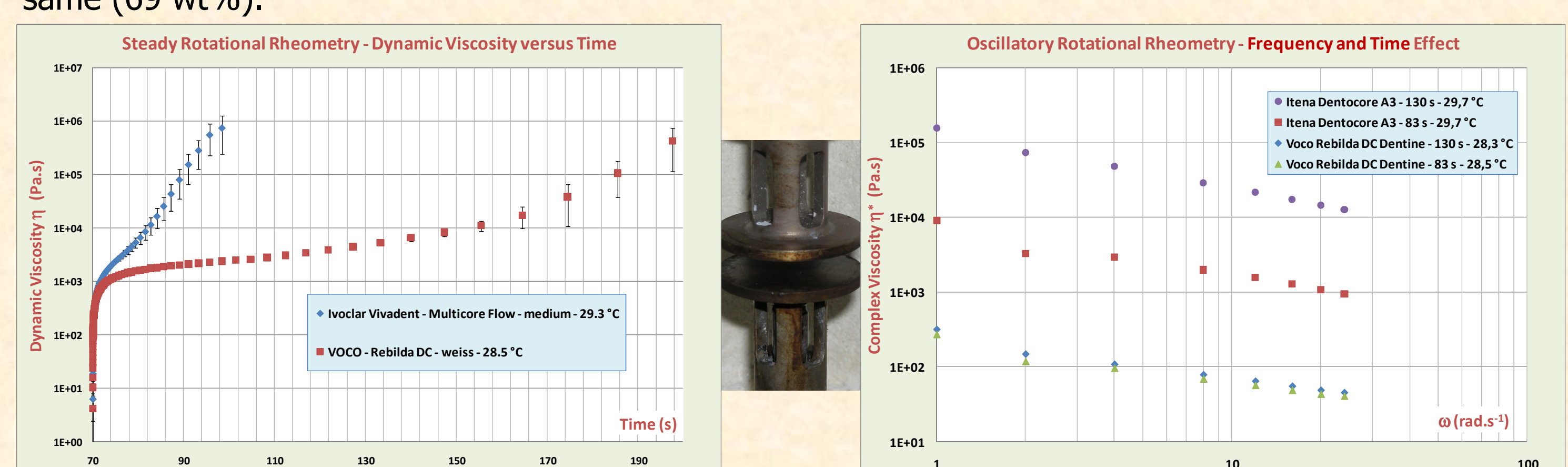
## EXPERIMENTAL

### 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:

**1. Steady:** time sweep (shear rate 0.05 s<sup>-1</sup>). 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 Multicore 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<sup>-1</sup>). 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.

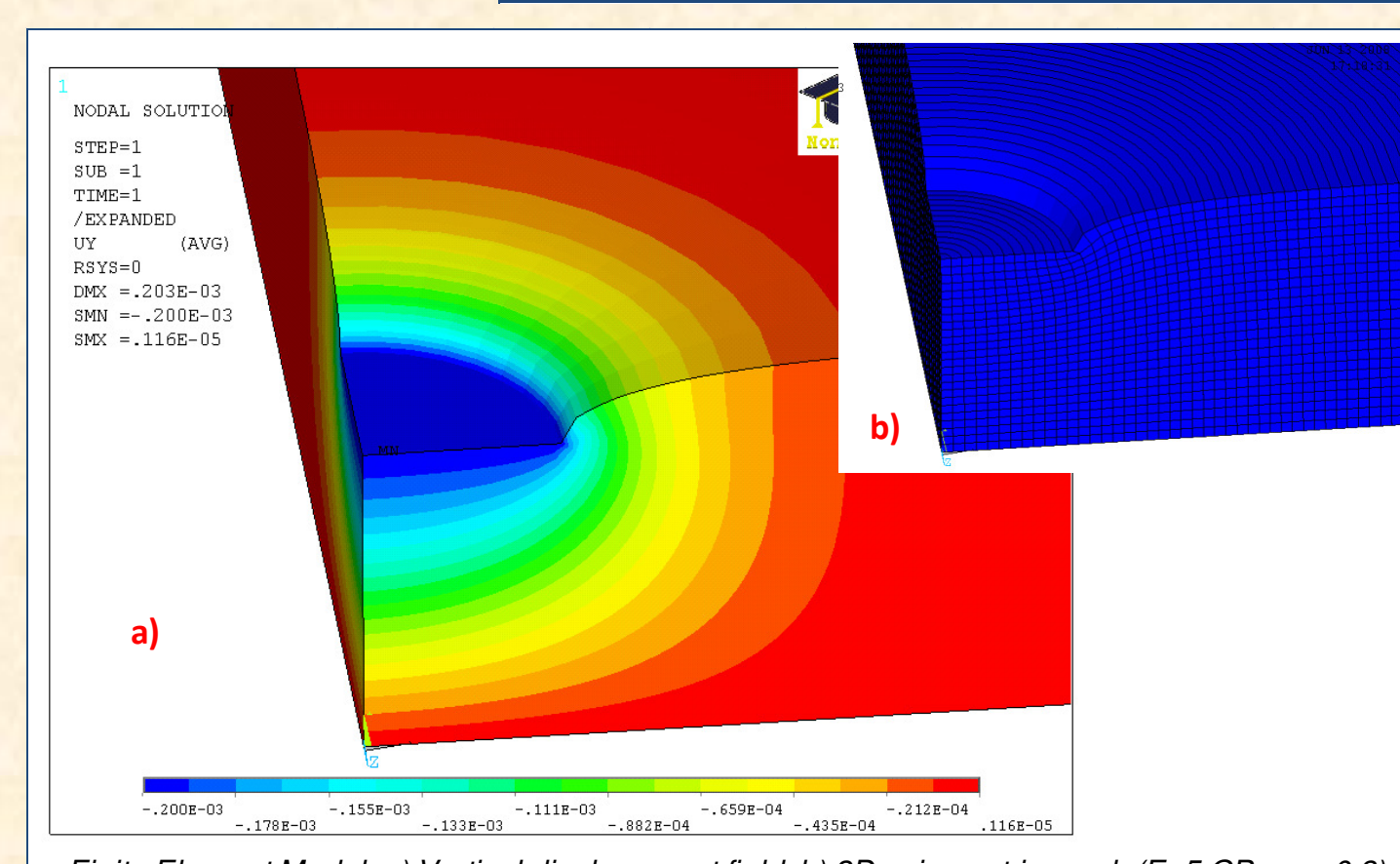
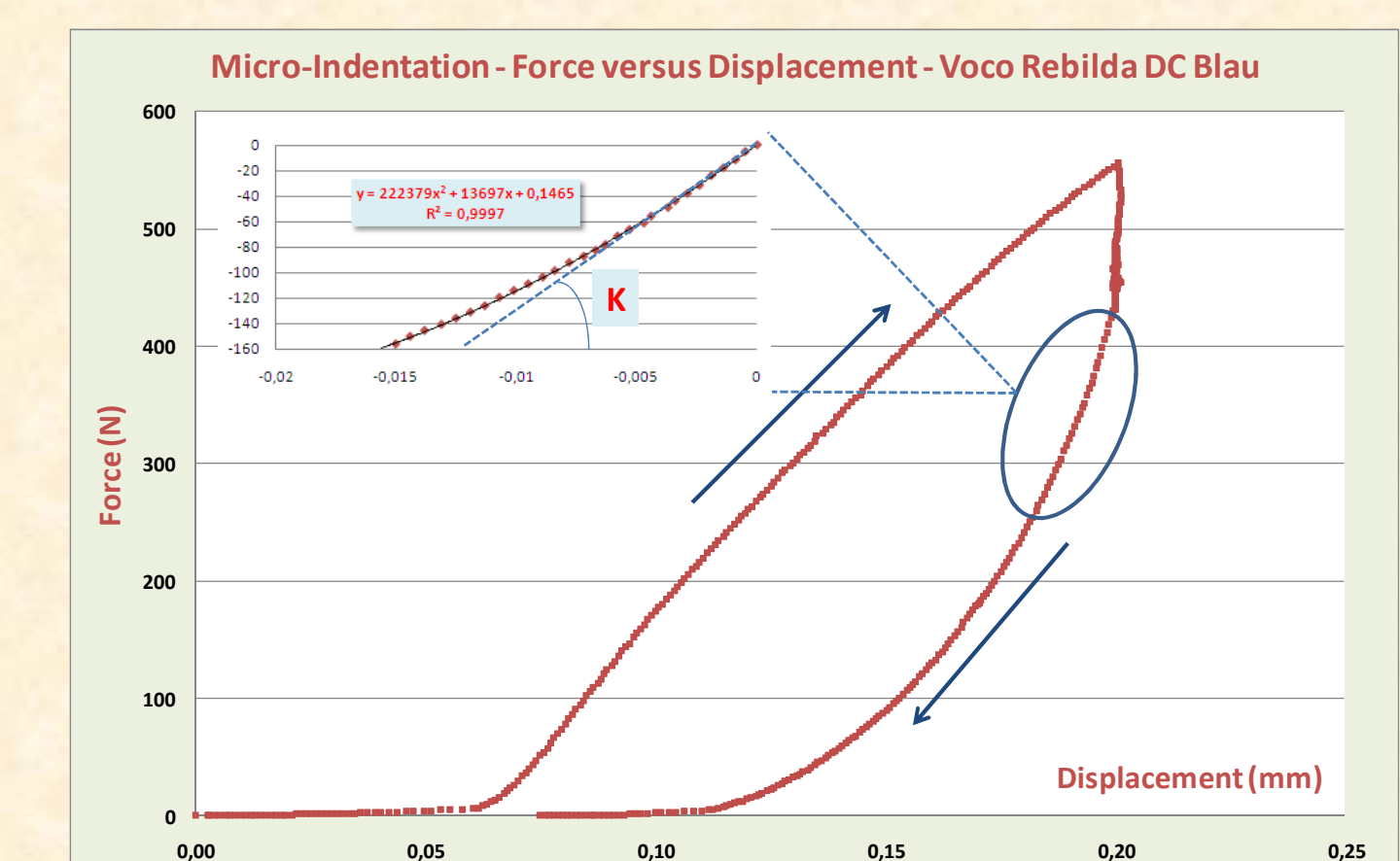
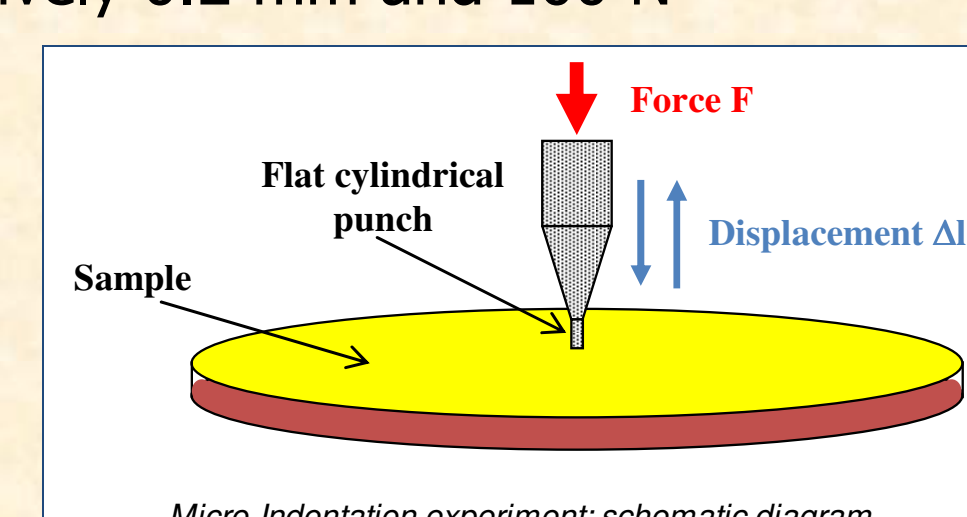
### MICRO-INDENTATION

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<sup>-1</sup>. 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).



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

- (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<sup>-1</sup>) 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 %

Resin	K <sub>0</sub> (N.mm <sup>-1</sup> )	K <sub>20</sub> (N.mm <sup>-1</sup> )	E <sub>0</sub> (GPa)	E <sub>20</sub> (GPa)
Voco Rebilda DC - Blau	13583	187	8.121	0.112
Voco Rebilda DC - Weiss	15741	446	9.411	0.267
Voco Rebilda DC - Dentine	14740	1762	8.813	1.053

Micro-indentation: K<sub>0</sub> (mean value of stiffness K), K<sub>20</sub> (standard deviation of K), E<sub>0</sub> (mean value of Young's modulus E), E<sub>20</sub> (standard deviation of E) - 3 tests for each measurement

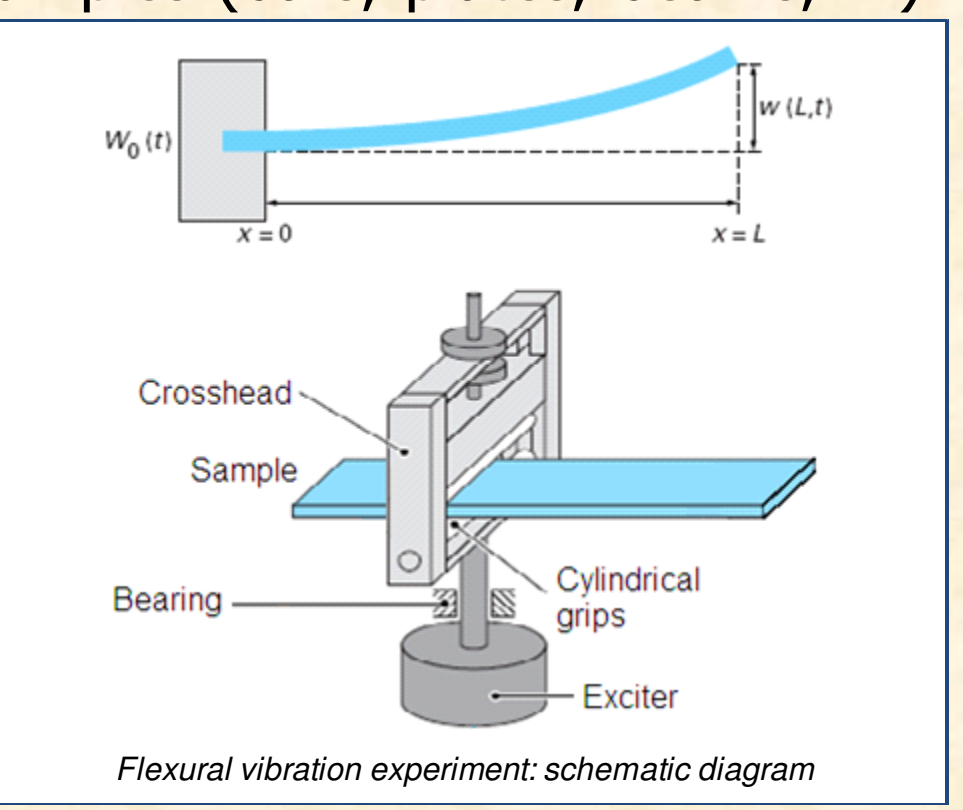
### FLEXURAL VIBRATION ANALYSIS

Flexural vibration is used to 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 mm<sup>3</sup> 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 [Chevallier, 2002] is  $\frac{d^4 y}{dx^4} = \frac{\rho S \omega^4}{EI} y = 0$  [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  $\frac{d^4 Y}{dx^4} - \beta^4 Y = 0$  [3].

$\beta$  is the dimensional wave number given by:  $\beta^4 = \frac{\rho S L^4 \omega^4}{EI}$  [4] and  $\omega = 2\pi f$  (f: frequency). The general solution is then:  $Y(x) = A \cosh(\beta x) + B \sinh(\beta x) + C \cos(\beta x) + D \sin(\beta x)$  [5]. Taking account of boundary conditions ( $y(0) = 0$ ,  $\frac{dy}{dx}(0) = 0$  clamping,  $\frac{d^2 y}{dx^2}(0) = 0$  null bending moment,  $\frac{d^3 y}{dx^3}(0) = 0$  null vertical shear force) gives:  $\cosh(\beta L) \cos(\beta L) + 1 = 0$  [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:  $E = \frac{\rho S L^4 \omega^4}{\beta^4 h^2} = C \frac{\rho^2 L^4}{h^2}$  [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 excited 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.

Sample	$\rho$ (kg.m <sup>-3</sup> )	L (m)	Frequency (Hz)	E (GPa) SD
Voco Rebilda DC - Blau	1838	0,050	330	9,066 ± 0,725
Voco Rebilda DC - Weiss	1844	0,050	330	9,094 ± 0,728

### MICRO-HARDNESS

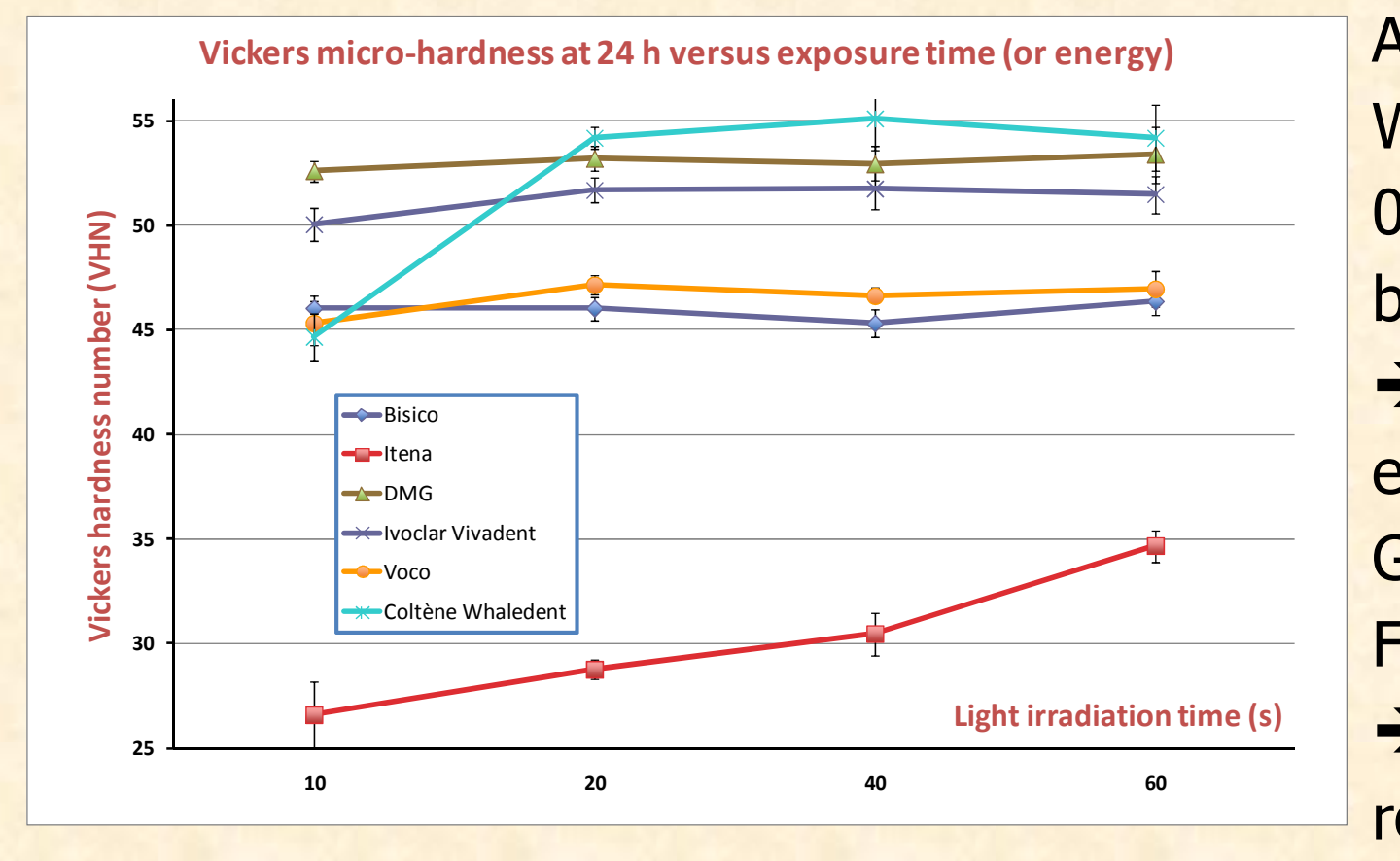
Cylindrical-shaped samples were prepared with metallic molds (4 mm diameter- 2 mm thickness). Five samples were prepared for each experimental group (n=5). Dual-cured resin-composites were inserted into the molds and covered with acetate strips (Hawe-Neos Dental, Bioggio, Switzerland). A 1 mm thick glass 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<sup>-2</sup> 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.

Supplier	Reference	Shade	10	20	40	60
Bisico	Best Core DC	A3	46.06 (0.56)	46.02 (0.58)	45.32 (0.67)	46.40 (0.68)
Itena	Dentocore	A3	26.62 (1.88)	28.78 (0.46)	30.46 (1.01)	34.66 (0.77)
DMG	LuxaCore	A3	52.58 (0.51)	53.20 (0.60)	52.96 (0.81)	53.88 (1.35)
Ivoclar Vivadent	Multicore Flow	Medium	50.96 (0.80)	51.70 (0.58)	51.70 (0.98)	51.46 (0.88)
Coltène Whaledent	Paracore	Dentine	44.68 (1.16)	44.18 (0.95)	45.08 (1.52)	44.18 (1.57)
Voco	Rebilda DC	Dentine	45.32 (1.05)	47.16 (0.48)	46.66 (0.38)	46.96 (0.85)

p < 0.0001, p < 0.0001, p < 0.0001, p < 0.0001

Light curing unit with light densities of 8 J.cm<sup>-2</sup> (10 s), 16 J.cm<sup>-2</sup> (20 s), 32 J.cm<sup>-2</sup> (40 s), and 48 J.cm<sup>-2</sup> (60 s) were used to light-cure the specimens. After light curing, the 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-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 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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