

**1273** Hot-pressing of biodegradable composites based on hydroxyapatite and poly-L-lactic acid. N. WAKAMATSU and Y. MORIWAKI (Dept. of Dent. Mater. & Tech., Sch. of Dent., Asahi Univ., Gifu, Japan)

Synthetic hydroxyapatite (HAP) closely resembles bone mineral and have been reported to show superior biocompatibility. Poly-L-lactic acid (PLA) is one of the bioresorbable polymers, essentially non-toxic and the lactic acid yielded after hydrolysis is a normal intermediate of carbohydrate metabolism without accumulation in vital organs. Therefore, the combination of the bioactive HAP and the bioresorbable PLA is expected to result in a biodegradable material with mechanical properties, such as Young's modulus and fracture energy, similar to those of compact bone. The aim of this preliminary study was to produce pore-free HAP/PLA composites by using hot-pressing. The HAP powder prepared in a aqueous medium was mixed with the PLA powder (M.W.: 10,000) (20, 30, 40, 50, 60wt%PLA). The mixtures were prepressed uniaxially at 50MPa and then isostatically pressed at 400MPa. The powder compacts were hot-pressed uniaxially at 150°C.

Contents of PLA (wt%)

	20	30	40	50	60
Bulk density (g/cm <sup>3</sup> )	1.66(0.02)	1.73(0.01)	1.84(0.01)	1.81(0.03)	1.87(0.04)
Relative density (%)	68.4	79.4	92.9	99.9	100

It was concluded that the hot-pressing of powder compacts was effective to make pore-free HAP/PLA composites. It was revealed that the composites hot-pressed were composed of the glassy PLA matrix and randomly oriented HAP crystals by X-ray diffraction technique.

**1275** The fundamental properties of the nickel-titanium endodontic instruments. M. Kubota, R. Terata, K. Minami, and Y. Kudou (Dept. of Operative Dentistry and Endodontics, Iwate Medical University, Morioka, Iwate, Japan)

The purpose of this study was to investigate the fundamental properties of the nickel-titanium endodontic instruments (Mac-file<sup>®</sup>, McSpadden).

The composition of Mac-file<sup>®</sup> was analyzed with the electron probe x-ray microanalyzer. The cross sectional shape and the permanent deflection were observed with the scanning electron-microscope. The length of the blade was measured with the profile projector. The micro Vickers hardness was measured with the micro hardness tester. Fortyfive degrees bending torque, breaking torque and breaking angular deflection were determined in conformity to the ISO Standards, 3630-1:1992. The three stainless steel reamers (MANI, GC, BEUTELROCK) were used as control.

The composition of Mac-file<sup>®</sup> was 49.7 atom.% Nickel and 50.3 atom.% Titanium. The length of the blade of Mac-file<sup>®</sup> was not in conformity to the ISO Standards. The cross sectional shape of #10 Mac-file<sup>®</sup> was square, #15 was triangle, the other sizes (#20, #25, #30, #35, #40) were the shapes like "S". The hardness of Mac-file<sup>®</sup> was significantly lower than stainless steel reamers' (t-test, p=0.01). The 45 degrees bending torque and breaking torque of Mac-file<sup>®</sup> satisfied the ISO Standards. The permanent deformation by the fracture of Mac-file<sup>®</sup> was less than the controls.

**1277** Impact Analysis by FEM on Shock Absorbing Effect of Periodontium. T. ITO\* and S. TSUTSUMI (Research Center for Biomedical Engineering, Kyoto University, Kyoto, JAPAN).

In this study, an impact stress analysis at the final stage of the chewing cycle, using 3D finite element modeling (FEM), was performed to examine the shock-absorbing effect of the periodontal membrane. A 3D FEM model of a lower second premolar with its supporting tissues (periodontal membrane, compact and sponge bones) was prepared. DYNA3D (Lawrence Livermore National Laboratory) was used for impact stress analyses. The minimum principal stress in the alveolar wall adjacent to the root after a horizontal impact loading of 10 N over in 1.0 ms was -1.45 MPa at 18 ms after impact which was two times as high as that due to static loading of the same magnitude. The stress induced by a vertical impact loading of the same magnitude of the horizontal loading was 1/6 as low as that due to the horizontal loading. The minimum principal stresses in the alveolar wall around the ankylosed tooth root model after a horizontal impact loading showed high concentration (about -3.8 MPa at 3, 12 and 20 ms) which may lead to bone and root resorption. In that model, the stress induced by a vertical loading was 1/3 as low as of that due to the horizontal loading. However, it was six times as high as that in the normal model under vertical loading. Thus, the shock absorbing effect of the periodontal membrane, though only several hundred microns thick, was significantly confirmed. Especially, the periodontium has the biomechanical optimality opposing to the vertical loading of occlusal forces.

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**1279** Differential Photocalorimetry (DPC) of 3,3,5-Trimethylcyclohexane Based Dimethacrylates For Dental Composites. B. M. CULBERTSON\*, A. TIBA and J. XU (The Ohio State Univ., Columbus, Ohio 43210, USA)

As part of a composites study, we are focusing on the synthesis, characterization, photopolymerization, etc., of new dimethacrylates. Objectives of this study were to evaluate the photopolymerization properties of dimethacrylates based on the phenol adduct of 3,3,5-trimethylcyclohexan-1-one, compared to 2,2-bis[4(3-methacryloxy-2-hydroxy-propoxy) phenyl] propane (Bis GMA) control. We earlier reported on the DPC of several dimethacrylates for dental composites (IADR 1994, #203 and IADR 1995, #1364). Dimethacrylates explored in this study were 1,1-bis [4(methacryloxy) phenyl]-3,3,5-trimethylcyclohexane (PWL 2791), 1,1-bis [4(2-methacryloxyethoxy) phenyl]-3,3,5-trimethylcyclohexane (PWL 3503) and 1,1-bis [4(2-methacryloxy propoxy) phenyl]-3,3,5-trimethylcyclohexane (PWL 3502), supplied by Bayer AG (Leverkusen, Germany). PWL 2791 and 3503 were difficult to work with due to lack of appreciable miscibility with reactive diluents such as triethylene glycol dimethacrylate (TEGDMA), whereas PWL 3502 readily blended with TEGDMA. Bis GMA:TEGDMA and PWL 3502:TEGDMA (50:50, wt:wt) were combined with 0.5 wt% camphorquinone and 1.0 wt% N,N-dimethylaminoethyl methacrylate, providing visible light curable (VLC) resins. The VLC characteristics of the systems were evaluated by DPC, using a TA Instruments 930 unit, equipped for VLC use. Samples of each neat resin were exposed to light for 30-60 sec, under N<sub>2</sub>, with the heat of reaction (enthalpy, ΔH) calculated. The ΔH values for the two systems, respectively, were 108.5 J/g and 107.7 J/g. Cured samples were conditional for 1 wk in water at 37° for glass transition (T<sub>g</sub>) studies. Using a TA Instruments TMA 2940 unit, the wet T<sub>g</sub> values of the VLC systems were observed as 55 and 67°C, respectively. PWL 3502 formulations undergo the VLC process equivalent to the control. PWL 3502 composites also retain a higher T<sub>g</sub> than the control on exposure to water. Thus, PWL 3502 may possibly be used to formulate VLC restoratives with improved properties, due to reduced water sorption and higher wet T<sub>g</sub> values.

**1274** New method for reproducible and standardized cavity preparation of class II lesions. B. HUGO\*, A. STASSINAKIS, P. HOTZ. (University of Bern, School of Dental Medicine, Bern, Switzerland).

The purpose of this study was to develop a new method for cavity preparations, and to evaluate the margin quality and the reproducibility of these preparations. For different sizes of class II lesions five diamond coated tips for oscillating "air-scalers" were designed. Two different "air-scaler" tips (diamond coating 40µm) preparing slightly divergent proximal boxes with a faciolingual width of the cervical extension of 2.5mm were tested. The first tip (G1) produces a 90-degree cavosurface angle, the second (GA1) a short bevel of the proximal and the gingival wall with a 45-degree cavosurface angle. With each method ten proximal box cavities were prepared in vitro on a model simulating clinical conditions. The margins were evaluated quantitatively in the SEM. The statistical comparison of the two methods was performed by non-parametric test procedures. The method with the short bevel (GA1) showed a significantly better finishing with 91.8% perfect margins (G1 53.8%)(p<0.05). Unacceptable margins with large chips were found in 3.8% (G1) and in 22.9% (GA1). The differences were statistically significant (p<0.05). In a second experiment the reproducibility of the cavities was tested indirectly by measuring the mean thickness of a silicon-layer between the inserted tip in the prepared cavity. 20 cavities were prepared with the GA1 tips and the mean thicknesses were determined on sections in 6 locations: faciolingual bevel 19.4±11.6µm, cervical bevel 12.1±6.3µm, faciolingual wall 94.3±40.4µm, cervical wall 12.8±7.7µm, palpal wall 12.9±7.2µm. Except for the faciolingual wall the results demonstrate a very small deviation from the original dimensions of the instrument. We conclude that the presented oscillating preparation method allows the production of highly precise beveled cavities with excellently finished margins and the potential of adapting preformed inserts.

**1276** Numerical Comparison of Artificial Saliva Recipes. V.W.-H. Leung\* and B.W. Darvell (Prince Philip Dental Hospital, University of Hong Kong, Hong Kong).

An artificial saliva (AS) of defined composition is necessary for testing the performance of materials which serve in the mouth, as natural saliva is too variable. The chemistry involved is critically important. Many AS recipes can be found in the literature, but the stability of tooth material, i.e. hydroxyapatite (HAP), in most of these has not been addressed. In fact, only few contain all major ionic components with concentrations in the physiological range. The aim of the present study was (a) to compare the stability of hydroxyapatite in various reported AS, and (b) to investigate the individual effects of major ionic components, i.e. calcium, magnesium, phosphate, ammonium, citrate, urea, urate, lactate, carbonate, thiocyanate, within the reported ranges, on the stability of hydroxyapatite. A computer algorithm, RAMESES (Leung and Darvell, *Talanta* 37: 425-429, 1990), for solving multiple equilibrium equations, was employed for the calculation of all species distributions and solubility isotherms. The positions of the solubility isotherms are compared. These show (a) that there is a marked difference between two groups, i.e. those with and without the presence of Ca, those with Ca are super saturated with respect to HAP in the physiological pH range, the saturation pH ranging from 4.4 to 5.8; the solubility of HAP in those without Ca has a 150-fold range at pH 7. From (b), there is also a 150-fold range in solubility at pH 7, due to the individual effects of major ions, i.e. phosphate, carbonate, and citrate. Acetate, lactate, and sulphate show smaller effects, and others have no appreciable effect. It is concluded that the composition of an AS must take into account all ions (such as carbonate and citrate) that affect HAP solubility and that closer attention must be paid to Ca/phosphate/pH values if stability with respect to HAP is to be attained.

**1278** The Quantitative Evaluation of Vital Bleaching. H. MATSUBA\*, N. KOSUGI, T. TOKO, T. SUZUKI and H. HISAMITSU (Department of Operative Dentistry, Showa University, Tokyo, Japan)

The effect of a bleaching agent, Hi-Lite (Shofu), was investigated by measuring the colors of the teeth directly in vivo before and after bleaching. Vital anterior teeth of 19 persons were bleached after measuring the colors of the teeth using a colorimeter (Minolta). After cleaning of the teeth, Hi-Lite was applied to the teeth and was light activated with Griplight II (Shofu) for 3 minutes. Hi-Lite was then removed and newly mixed Hi-Lite was reapplied. After the bleaching was repeated 3 times, the colors of the teeth were measured. This procedure was performed 3 times, once a week for three weeks. After the third bleaching, it was determined that the tooth color of 6 patients was not clinically acceptable; therefore, a fourth bleaching was performed for these 6 patients only. One month after the last bleaching, the patients were recalled and the colors of the teeth were measured. The results are presented by CIEL\*a\*b\* in the table. A t-test analysis revealed significant differences between the values of L\*, a\* and b\* before and after bleaching (p<0.01). However, after 1 month only a slight reversion of the tooth color was recognized, although there was a significant difference in a\* (p<0.05). The results of this study showed quantitatively that Hi-Lite was effective in bleaching of the discolored vital tooth (dE=8.3 just after bleaching and dE=6.9 after 1 month).

	before bleaching		after bleaching		after 1 month
L*	55.8 ± 6.7	dL	5.8 ± 4.9		5.0 ± 2.5
a*	3.5 ± 2.0	da	-1.1 ± 1.5		-0.4 ± 1.8
b*	17.8 ± 2.4	db	-4.3 ± 1.9		-3.8 ± 1.7
		dE	8.3 ± 3.5		6.9 ± 1.9

(mean ± S.D.)

**1280** Detection of Leached Moieties from Dental Composites in Food/Oral Simulating Fluids. S.-Y. LEE\*, C.-T. LIN\*, E.H. GREENER\*, D.L. MENIS\* (School of Dentistry, Taipei Medical College, Taiwan, ROC; \*Northwestern University Dental School, Chicago, IL, USA)

The leaching of components from dental composites has a potential impact on both the structural stability and the biocompatibility of the material. In an attempt to determine the nature of moieties leached from dental composites, Fourier transform infrared (FTIR) spectroscopy was used to analyze two solutions in which three commercially available dental composites (Marathon One - Den-Mat Co.; Z100 - 3M Co.; Herculite XRV - Kerr Co.) were stored. The solutions used were: a food simulating fluid, 75% EtOH, and an artificial saliva, Moi-Stir (Kingwood Labs., Inc.). Specimens (4.3 mm diam. x 2 mm thickness) of the three resin composites were stored at 37°C in 60 mL of either artificial saliva or the 75% ethanol solution for 7, 14, and 30 d. The FTIR spectra were obtained using a liquid sample ATR (attenuated total reflection) cell. No obvious leachable materials were seen from any of the composite specimens stored in artificial saliva up to 30 d of immersion. For the composites stored in ethanol, the observed spectra revealed increases in peak heights over time. In general the spectra of the leachates show the same principle absorption bands for the three composite systems. Methacrylate skeletal vibrations (1015-815 cm<sup>-1</sup>) and -CH<sub>2</sub> alkane, C-H asymmetrical deformation vibrations (1520-1460 cm<sup>-1</sup>) appeared after 14 d of storage. A very strong peak characteristic of the aliphatic C=C moiety (1640cm<sup>-1</sup>) and carbonyl C=O (1730 cm<sup>-1</sup>) occurred after 14 d. The peak heights of these two functional bands increased as a function of time and after 30 d of storage were approximately 5-7 times those produced after 7 d. Irregular O-H bands (3500-3300 cm<sup>-1</sup>) were also observed after 30 d in ethanol. Irreversible processes such as chemical composition changes by leaching may contribute to irreversible material degradation.

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