Composition of dental resin and composite materials

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The purpose of this investigation was to develop routine methods for qualitative and quantitative analyses of the monomers in dental resin and composite materials. The content of inorganic fillers was determined by combustion analysis. Total filler content of the so-called microfill materials was determined after removing the monomers and additives by means of a solvent.

The monomers were determined qualitatively and quantitatively by means of high performance liquid chromatography. The components in the liquid resin systems were separated by adsorption, partition and gel permeation chromatography. Semi-preparative columns were used for separation of unknown monomers. The fractions were analyzed by means of infrared spectroscopy, mass spectrometry, proton and carbon thirteen magnetic resonance spectroscopy.

This investigation demonstrates the variety in composition of dental resin and composite materials.

Key-words: Chemical analysis; HPLC; GPC; monomers

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The various commercial resin-based filling materials have different physical, chemical, biological and clinical properties. Laboratory studies indicate differences with respect to hardness (8, 25), compressive strength and tensile strength (7, 8, 14), water sorption (4), conversion of the methacrylate groups (20), color stability (17), abrasion (8, 10), in vitro cytotoxicity (9, 24), and antibacterial activity (27).

Clinical investigations indicate that dental composite materials are susceptible to wear and loss of «anatomic form» (11, 13, 15, 16) as well as to discoloration (6, 11, 13, 15). Plaque accumulation has also been shown to be high in these materials (22) which may explain the incidence of secondary caries noted in clinical studies (13).

Both mechanical wear and chemical degradation may cause loss of substance of the polymer matrix of dental composites (20). Chemical degradation may also lead to discoloration (20). The molecular structure of the monomers and the quantity of the different monomers used in dental composite materials will affect all properties of the polymerized materials.

The monomer composition influences the conversion in dental sealants (19). The various unfilled resin systems also have different clinical properties

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Table 1. *Investigated resin coating materials*

Name	Code	Batch No	Manufacturer
Nuva Seal	NS	Base: 74127	The L.D. Caulk Co., DE, USA
Adaptic Bonding Agent	AB	8K 303	Johnson & Johnson Dental Products Co., NJ, USA
Adaptic Glaze	AG	0206D14	Johnson & Johnson Dental Products Co., NJ, USA
Delton Pit & Fissure Sealant	DS	2147D401	Johnson & Johnson Dental Products Co., NJ, USA
Kerr Pit & Fissure Sealant	KS	0106761329	Kerr Manufacturing Co., MI, USA
Estilux Glaze	EG	601229	Kulzer & Co. GmbH, W. Germany
Epoxylite 9075	ES	0355E-1	Lee Pharmaceuticals, CA, USA
Concise Enamel Bond	CE A	6153 2	3M Company, MN, USA
Concise Enamel Bond	CE B	18 JUL 78	3M Company, MN, USA
Concise White	CW	Resin B: 6233F1 Resin A: 71401	3M Company, MN, USA
Saga Sealant	SS	not indicated	Saga Orthodontics, Norway
Compact Enamel Bond	CS	751114	Svedia Dental-Industri AB, Sweden
Profile Bonding	PB	37903	S.S. White Dental Products International, PA, USA

(12, 18, 26). Viscosity, surface tension and contact angle on acid etched human dental enamel are also properties which are dependent on resin compositon (2).

Analyses of many proprietary restorative resin materials have been performed by nuclear magnetic resonance (NMR) spectroscopy (1). The present work was designed to develop routine methods for qualitative and quantitative analysis of monomers in dental resin materials. Many monomers used in the available types of resin-based filling materials and sealant systems are of low volatility and unstable at high temperatures. Various modes of high performance liquid chromatography (HPLC) (23) were investigated for their suitability in routine analyses of such materials.

MATERIALS AND METHODS

The proprietary products listed in Tables 1 and 2 were analyzed for their content of monomers, both qualitatively and quantitatively.

Determination of filler content

The content of inorganic fillers in the conventional dental composite restorative materials and the so-called microfilled materials was determined by burning at 575°C as described in the international standard for resin–based filling materials, ISO 4049. The products containing the so-called microfillers in particles of prepolymerized material were suspended in a solvent. The soluble components were removed by centrifugation and decantation (RC-5 Automatic Superspeed Refrigerated Centrifuge, Du Pont/Sorvall, CT, USA). The filler particles were washed several times with the solvent and the particles were dried at 120°C. The dried particles were weighed and the inorganic content in the prepolymerized particles was determined by combustion.

Table 2. *Investigated dental resin filling materials*

Name	Code	Batch No	Manufacturer
COMPOSITE MATERIALS:			
Nuva-Fil P.A.	NF	012278	The L.D. Caulk Co., DE, USA
Nimetic Composite Dental Restorative	NM	Catalyst: D 327 Base paste: D 320	ESPE GmbH, W. Germany
Epolite 100 Dental Restorative	EP	J M 3	G-C Dental Industrial Corp., Japan
Adaptic Dental Restorative	AD	6C009	Johnson & Johnson Dental Products Co., NJ, USA
Miradapt Dental Restorative	MA	9F902	Johnson & Johnson Dental Products Co., NJ, USA
Fotofil Dental Restorative	FF	3537E836	Johnson & Johnson Dental Products Co., NJ, USA
Protosit Composite	PR	Catalyst: 202–12 Universal 202–13	A. Kettenbach GmbH & Co. KG, W. Germany
Durafill Light-curing Composite	DF	0939	Kulzer & Co. GmbH, W. Germany
Estic Composite	E	Catalyst paste: 71233 Basic paste: 80746	Kulzer & Co. GmbH, W. Germany
Estic microfill Composite	EM A	Catalyst paste 0331277 Basic paste: 0231277	Kulzer & Co., GmbH, W. Germany
Estic microfill Composite	EM B	Catalyst paste: 25.9.79 Basic paste: 25.9. 79	Kulzer & Co. GmbH, W. Germany
Estilux	EL	Dez. 77/73	Kulzer & Co. GmbH, W. Germany
Estilux microfill	ELM	023 1. 78	Kulzer & Co. GmbH, W. Germany
Lee-Fill	LF	060177 FL-1I	Lee Pharmaceuticals, CA, USA
Prestige Dental Restorative	PS A	083176 HP-1I	Lee Pharmaceuticals, CA, USA
Prestige Dental Restorative	PS B	102076 HP-1I	Lee Pharmaceuticals, CA, USA
Prestige Dental Restorative	PS C	111676 HP-1I	Lee Pharmaceuticals, CA, USA
Restodent	RD	121377 I-1I	Lee Pharmaceuticals, CA, USA
Concise Composite	CC	6271S14	3M Company., MN, USA
Silar Composite	SC	9E 1 110579	3M Company, MN, USA
Compact Composite Restorative	CP	760226	Svedia Dental-Industri AB, Sweden
Profile Composite Restorative	PC	042079	S.S. White Dental Products International, PA, USA
NONCOMPOSITE MATERIALS:			
Isopast	IP	Catalyst: 230877 Base: 080777	Vivadent, Liechtenstein

Monomer analyses by HPLC

Solutions containing the monomers were prepared by treatment of the filled and unfilled resins with argon saturated solvents. The solvents used were acetonitrile, methanol, dicloromethane and tetrahydrofuran depending on the chromatographic method. The particles in the filled materials were separated from the solution by centrifugation at 3 000 *g* for 20 min. at 20°C.

The monomer-containing solutions were analyzed by means of HPLC (Liquid Chromatograph, Model 3 500 B, Spectra-Physics, CA, USA with SF 770 Spectroflow, Schoeffel Instrument Corp., NJ, USA; Multiref 901 Differential Refractometer, Optilab, Sweden, and Model CV-6-UHPa-N60 Sample Injection Valve, Valco Instruments Co., TX, USA). All the solvents applied in this investigation were of ultraviolet grade and HPLC-grade (E. Merck, W. Germany, and Rathburn Chemicals Ltd., Scotland). The water was bi-distilled (Fi-streem, Fisons Scientific Apparatus, England). All mobile phase solvents were degassed and saturated with helium.

Qualitative analysis

The monomers were determined qualitatively by means of adsorption chromatography and reversed phase chromatography under the conditions specified in Tables 3 and 4. The components were separated by gradient elution which enabled complete separation of the components in the mixtures and also elution of strongly retained compounds.

A UV-visible spectrophotometer or a refractometer was used for detection of the components. The refractometer was sensitive to changes in condition and composition of the mobile phase. In order to obtain isocratic conditions, a mobile phase of two or more solvents had to be continuously stirred in front of the pump.

Table 3. *Reversed phase chromatography, chromatographic conditions*

Column*:	250mm x 4,6 mm, Spherisorb ODS 5 µm, Phase Separations Ltd., England	
Mobile Phase:	Pump A	H_2O
	Pump B	CH_3OH
	1. Isocratic elution	75 % B
	2. Gradient elution	69 % B, 5 min, isocratic; 69% to 99% B, 5 min, linear gradient
Flow Rate:	0.8 ml/min	
Detector:	UV 254 nm or 210 nm RI 546 nm	

*Two columns connected in series

Table 4. *Adsorption chromatography, chromatographic conditions*

Column*:	250 mm x 4,6 mm. Spherisorb Silica 5 µm, Phase Separations Ltd, England	
Mobile Phase**:	Pump A	CH_2Cl_2; 0.2 % H_2O
	Pump B	CH_2Cl_2/CH_3OH 94/6; 0.2 % H_2O
	Gradient elution	25 % B, 7 min, isocratic; 25% to 99% B, 5 min, linear gradient
Flow rate:	1.2 ml/min	
Detector:	UV 254 nm, RI 546 nm	

* Two columns connected in series.

**Water was added to prevent the columns from drying out.

To separate mono-, di-, tri- and tetra-ethylene glycol dimethacrylate adsorption chromatography was ap-

Table 5. *Semi-preparative chromatography, chromatographic conditions*

Column:	500 mm x 8 mm, Micro-Pak Si-5 Varian Instrument, Division, CA, USA	
Mobile Phase*:	Pump A	CH_2Cl_2; 0.2 % H_2O
	Pump B	CH_2Cl_2/ CH_3OH 94/6; 0.2 % H_2O
	Isocratic elution	25 % B
Flow rate:	1.6 ml/min	
Detector:	UV 254 nm	

*Water was added to prevent the column from drying out.

Table 6. *Gel permeation chromatography, chromatographic conditions*

Column:	300 mm x 3.9 mm, µPorasil GPC 60 Å, Waters Associates Inc., MA, USA
Mobile Phase*:	THF; 0.5 % CH_3OH
Flow rate:	0.2 ml/min
Temperature:	22.5°C
Detector:	UV 265 nm

*CH_3OH was added to prevent adsorption on column material.

plied (Fig. 1). A semi-preparative method was used to identify unknown components (Table 5). Fractions were collected and the solvents of the mobile phase evaporated under vacuum. The collected low-volatile components were identified by means of infrared spectroscopy (Grating Infrared Spectrophotometer, Modell 577, Perkin-Elmer Corp., CT, USA), mass spectrometry (Hitachi Perkin-Elmer RMU-6L Mass Spectrometer, Hitachi Ltd., Japan), proton magnetic resonance (^1H NMR) (Varian EM360A NMR Spectrometer and Varian HA 100–15D NMR Spectrometer, operating at 98 MHz, Varian Associates, CA, USA) and carbon thirteen magnetic resonance (^{13}C NMR) spectroscopy (Fourier Transform NMR-Spectrometer, JNM-FX 60, Jeol Ltd., Japan). In addition, volatile components were identified by means of combined gas chromatography/mass spectrometry (990 Gas Chromatograph, Perkin-Elmer Corp., CT, USA; Hitachi Perkin-Elmer, RMU-6L Mass Spectrometer, Hitachi Ltd., Japan).

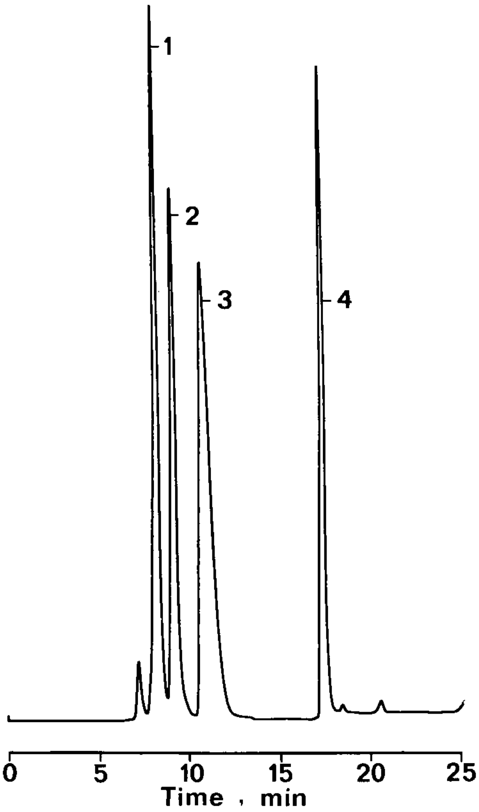


Fig. 1. Separation of mono-(1), di-(2), tri-(3) and tetraethyleneglycol dimethacrylate (4) by adsorption chromatography with a linear gradient from 70 % A + 30 % B to 33 % A + 67 % B in 3 min (Table 5).

Reference substances were supplied from various sources (Table 7) and the purity of the compounds was of different grade. Monomers which were not commercially available were synthesized.

Table 7. *Chemicals used in the investigation*

Ethyleneglycol dimethacrylate	EGDMA	Koch-Light Laboratories, Ltd., England
Diethyleneglycol dimethacrylate	DEGDMA	Ancomer Ltd., England
Triethyleneglycol dimethacrylate	TEGDMA	Koch-Light Laboratories Ltd., England
Polyethyleneglycol dimethacrylate	Poly-EGDMA	Ancomer Ltd., England
Methyl methacrylate	MMA	Koch-Light Laboratories Ltd., England
2,2-Bis [4–methacroyloxy-phenyl] propane	Bis-MA	Aldrich-Europe, Belgium
2,2-Bis[4-(2-hydroxy-3-methacroyloxypropoxy)phenyl] propane	Bis-GMA	Glaxo Laboratories, England
2,2-Bis [4-(2-methacroyloxyethoxy) phenyl] propane	Bis-EMA	Ancomer Ltd., England
2,2-Bis [4-(3-methacroyloxypropoxy) phenyl] propane	Bis-PMA	ESPE GmbH, W. Germany
«Urethane dimethacrylate»	UEDMA	Ivoclar AG, Liechtenstein
2-Hydroxyethyl methacrylate	HEMA	Fluka AG, Switzerland
2,3-Epoxypropyl methacrylate		Aldrich Europe, Belgium
Dibutyl phtalate		Fluka AG, Switzerland
Dicyclohexyl phtalate		Fluka AG, Switzerland
Butyl-2-butoxyethyl phtalate		ICN-K & K Laboratories Inc., CA., USA
4-cyclohexene-cis-1,2-dicarboxylic anhydride		Koch-Light Laboratories Ltd., England
Cyclohexane-1, 2-dicarboxylic anhydride		Merck-Schuchardt, W. Germany
Pyridine		Merck – Schuchardt, W. Germany

Syntheses of methacrylate derivatives of cyclohexane and cyclohexene

A mixture of 17 mmol 2-hydroxyethyl methacrylate, 17 mmol cis-1,2-cyclohexane-dicarboxylic anhydride, 1 mg 1-hydroxy-4-methoxybenzene in 10 ml pyridine was heated for 2 hours at 100°C. The reaction mixture was poured on ice and acidified with concentrated hydrochloric acid. The monoester was extracted with chloroform. Using a saturated aqueous potassium carbonate solution the monoester was brought into the aqueous phase. When this phase was acidified with concentrated hydrochloric acid the 1,2-cyclohexanedicarboxylic monomethacroyloxyethyl ester precipitated.

A mixture of 10 mmol of the synthesized monoester, 10.3 mmol 2.3-epoxypropyl methacrylate, 0.8 mmol pyridine and 25 mg potassium nitrite was stirred and gently heated to 70°C. This temperature was maintained for 40 hours. The reaction micture was poured on ice and the dimethacrylate monomer c-HaDMA (Fig. 4) was extracted with dichloromethane. The solvent was removed under vacuum. The dimethacrylate monomer was purified by semi-preparative HPLC using isocratic conditions with 75 % A and 25 % B (Table 5). The purified product was used as reference material.

The same procedure was used for synthesizing the corresponding monomethacrylate and dimethacrylate derivatives of cis-4-cyclohexene-1,2-dicar-

Table 8. *Filler content in the materials investigated*

Material	Total filler content wt.–%	Inorganic filler content wt.-%
COMPOSITE MATERIALS:		
NF		80.4
NM catalyst		76.8
NM base		78.5
EP catalyst		30.0
EP base		79.0
AD catalyst		77.3
AD universal		77.4
MA catalyst		82.0
MA universal		82.9
FF		79.4
PR catalyst		71.9
PR universal		76.8
DF	75.0	51.9
E catalyst		9.44
E base		75.7
EM A catalyst	75.1	50.6
EM A base	76.9	50.7
EM B catalyst	78.2	52.6
EM B base	74.7	50.4
EL		76.9
ELM	75.7	51.2
LF		81.2
PS A catalyst		78.0
PS A universal		78.4
PS B catalyst		78.9
PS B universal		78.5
PS C catalyst		77.6
PS C universal		78.4
RD part A		0
RD part B		97.1
CC paste B		77.1
CC paste A		77.2
SC paste B	75.6	50.5
SC paste A	75.5	50.1
CP catalyst		74.6
CP universal		79.8
PF catalyst		80.2
PF base		80.2
NONCOMPOSITE MATERIALS:		
IP catalyst	61.0	31.5
IP universal	64.0	23.3
SEALANTS:		
KS catalyst		39.2
KS base		38.0
CW paste A		7.7

Coefficient of variation of five values; v = 0.001 for inorganic filler content in restorative materials, and v = 0.005 for total filler content and inorganic fillers in filled sealant.

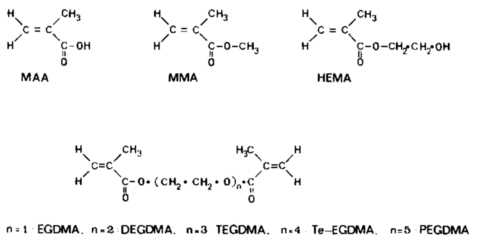


Fig. 2. Monomers of low viscosity identified in dental resin materials.

boxylic acid (c-HeDMA) with the corresponding anhydride as starting material.

Quantitative HPLC-analysis

For the quantitative determination of the monomers in the various commercial products, standard calibration curves were obtained by plotting the peak areas and peak heights of known concentrations of the respective monomers dissolved in methanol or acetonitrile. The peak areas were measured electronically with a computing integrator (Autolab Minigrator, Spectra-Physics, CA, USA). Referring to the standard calibration curves, the peak areas and peak heights from the HPLC-analyses of the different materials, based on three to five samples were transformed into weight-% monomer in the soluble organic part of the analyzed resin and composite materials.

Reversed phase partition chromatography was used for the quantitative analyses (Table 3).

Gel permeation chromatography

It was not possible to characterize the restorative composite NF by means of HPLC. In order to analyze the composition of the organic phase of NF gel permeation chromatography (GPC) was applied. The chromatographic conditions are outlined in Table 6. A calibration curve for molecular weight determinations was established.

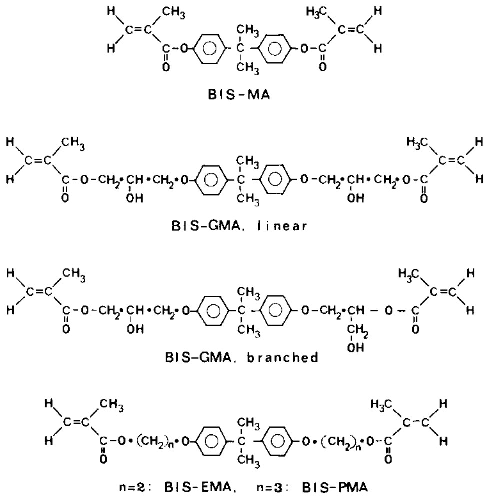


Fig. 3. Dimethacrylate derivatives of bisphenol-A.

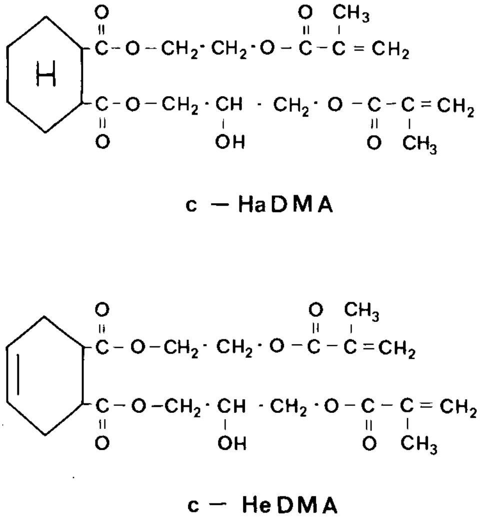
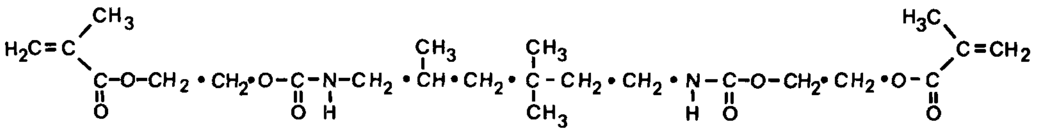


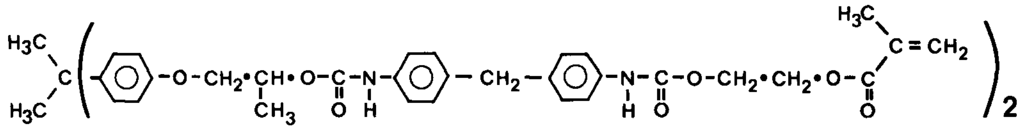
Fig. 4. Dimethacrylate derivatives of 1,2-cyclohexanedicarboxylic acid and 4-cyclohexene-1,2-dicarboxylic acid.

RESULTS

The quantity and type of filler particles in the materials investigated are presented in Table 8. The different identified monomers are shown in Figs. 2–5.



UEDMA



TUDMA, simplified structure

Fig. 5. Dimethacrylate monomers containing urethane groups.

Qualitative analysis

A typical HPLC-chromatogram of a dental resin material is shown in Fig. 6. A commercially used monomer BIS-GMA can be resolved into different components by adsorption chromatography (Fig. 7). A semi-preparative silica column was used for fractionating and collecting the peaks 1, 2 and 3 (Fig. 7). According to mass spectrometric analyses peak 1 and 2 had a molecular weight of 512. By means of ^1H and ^{13}C NMR peak 1 was identified as the linear BIS-GMA and peak 2 as the branched BIS-GMA (Fig. 3). Mass spectrometry indicated that peak 3 represented the compound BIS-GMMA with only one methacrylate group, i.e. the other side of the molecule has two unsubstituted hydroxyl groups.

NMR-analyses showed that BIS-PMA had a linear structure, i.e. the 1,3-propane dioxy derivative.

The tetraurethane dimethacrylate monomer of FF was characterized by adsorption HPLC using both UV-detector and RI-detector (Fig. 8).

By means of reversed phase semi-preparative chromatography the peak 2 of PS C and peak 4 of PS A were collected (Fig. 9). Analyses of fractions by means of mass spectrometry and ^1H and ^{13}C NMR indicated the monomers c-HeDMA and c-HaDMA shown in Fig. 4. The monomers were synthesized and used as reference materials and the presence of c-HeDMA in PS A and c-HaDMA in PS C was confirmed. The ^1H NMR-spectrum also showed that c-HaDMA was a cis-1,2-cyclohexane-dicarboxylic acide derivative.

Some materials (AD and MA) contained small amounts (less than 2 wt.-%) of methacrylic acid.

Some of the catalyst pastes did not contain any methacrylate monomers. By means of combined gas chromatography/mass spectrometry the liquid part of E catalyst and IP catalyst was identified as phtalate esters (Tables 7 and 10). Mass spectrometric analysis of the separated liquid part of EP catalyst showed that the main component was a silicone oil, a poly(dimethylsiloxane).

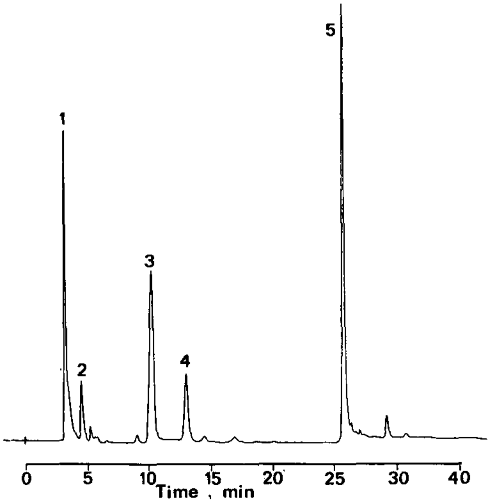


Fig. 6. Separation of the main components in the resin material AG universal by adsorption chromatography: 1 = BIS-MA, 2 = TEGDMA, 3 = BIS-GMA, linear, 4 = BIS-GMA, branched, and 5 = 3,4-xylyldiethanolamine.

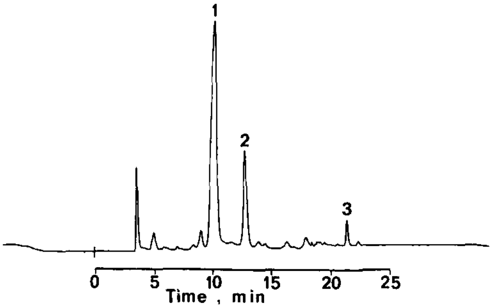


Fig. 7. Separation of the components in BIS-GMA on a silica column with the identified constituents: 1 = BIS-GMA, linear, 2 = BIS-GMA, branched, and 3 = bisphenol-A-diglycidylmonomethacrylate, BIS-GMMA.

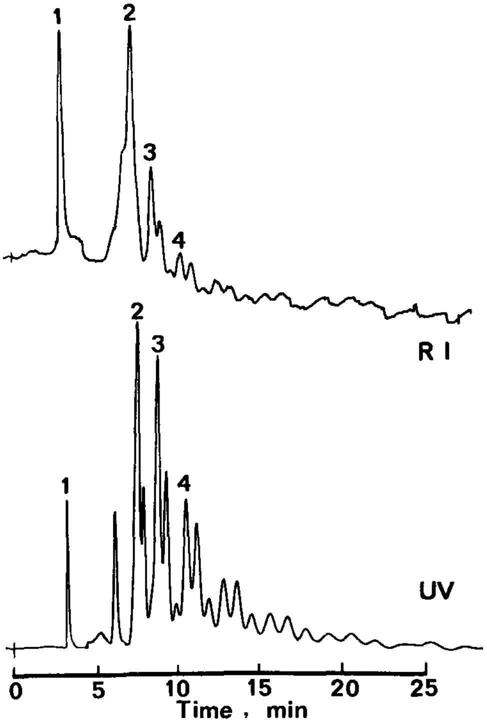


Fig. 8. Adsorption chromatogram of the organic compounds in the composite material FF using a differential refractometer (RI) and an ultra violet spectrophotometer (UV) as detectors. Peak 1 represents EGDMA and the peaks 2 – 4 represent TUDMA and its isomers.

Quantitative HPLC-analysis

A reversed phase chromatogram used for quantitative determinations of the monomers is shown in Fig. 10. The composition of the materials investigated is presented in Tables 9 and 10. The mean coefficient of variation of three to five values was 0.03. This presentation demonstrates that there were large differences with respect to composition in the various commercial materials. Most of the materials contained relatively well defined dimethacrylate monomers. However, BIS-GMA was represented by isomers with a proportion of the linear to the branched BIS-GMA of approximately 3:1 in most of the materials investigated.

GPC-analysis

The liquid part of the restorative composite material NF was characterized by high performance gel permeation chromatography (Fig. 11). NF contained TEGDMA and a polymeric material. GPC-, IR- and NMR-analyses of the polymeric component indicated that it was a linear polyurethane synthesized from BIS-GMA and an alkane-

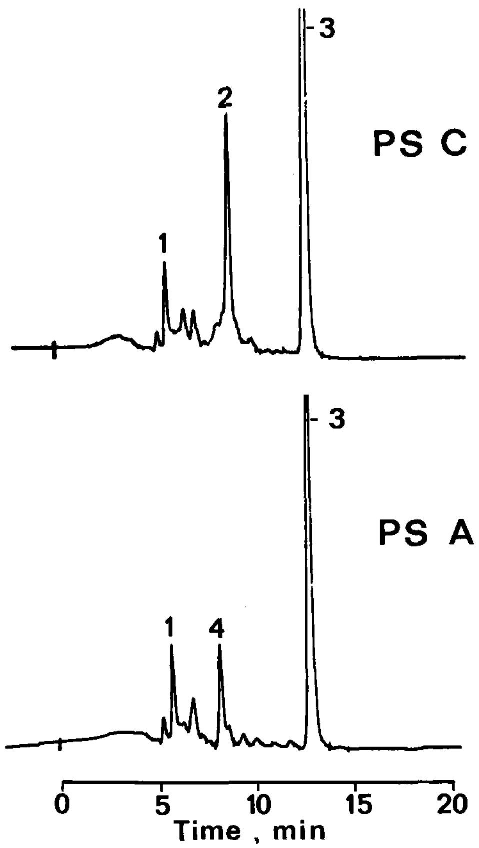


Fig. 9. Reversed phase chromatograms of the soluble organic parts of PS C catalyst and PS A catalyst: 1 = TEGDMA, 2 = dimethacrylate derivative of 1,2-cyclohexanedicarboxylic acid, 3 = benzoylperoxide and 4 = dimethacrylate derivative of 4-cyclohexene-1,2-dicarboxylic acid.

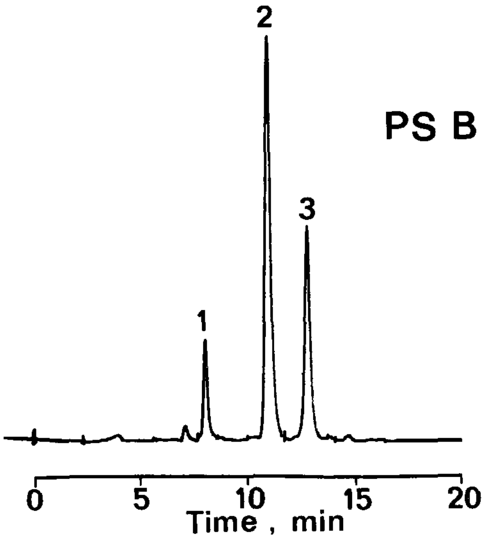


Fig. 10. A representative reversed phase chromatogram; 1 = TEGDMA, 2 = BIS-GMA and 3 = benzoylperoxide.

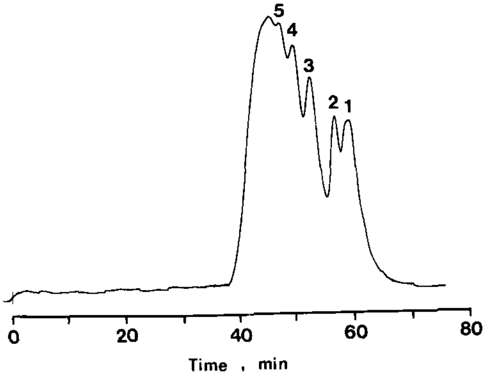


Fig. 11. Gel permeation chromatogram of the organic part of NF soluble in tetrahydrofuran. Peak 1 represents TEGDMA and peaks 2-5 represent the urethane oligomers of BIS-GMA and an alkanediisocyanate.

diisocyanate. The polymer molecules contained the reactive methacrylate groups of the BIS-GMA units. The polymer fractions with lower molecular weights, oligomers, can be distinguished as separate peaks, 2–5. The average molecular weight of the polyurethane was calculated to approximately 5 000.

DISCUSSION

This investigation demonstrates the variety in compositon of the different proprietary dental composite and resin systems. Most of the materials contained the high-viscous monomer BIS-GMA and the relatively low-viscous TEGDMA. Other monomers were, however, found in several of the investigated materials.

When a material is regularly analyzed, variations in monomer com-

Table 9. *Monomers (wt.-%) in dental resin coating materials*

Material	MMA	EGDMA	DEGDMA	TEGDMA	BIS-GMA	BIS-MA
NS base	20				79	
AB catalyst				56	35	5
AB universal				56	36	5
AG Catalyst				33	56	7
AG universal				33	57	7
DS catalyst				53	37	5
DS universal				53	38	5
KS catalyst				31	68	
KS base				31	67	
EG				50	51	
ES part B			98			
ES part A			96			
CE A resin B				47	49	
CE A resin A				50	45	
CE B resin B				49	40	
CE B resin A				51	34	
CW resin B				47	43	
CW resin A				52	42	
SS universal	28				72	
CS catalyst				45	57	
CS base				45	56	
PB catalyst		36			61	
PB base		40			57	

position and degree of purity are noticed. The quality of BIS-GMA is conveniently observed by the quantity of peak 3, i.e. the monomethacrylate derivative BIS-GMMA (Fig. 7). The formation of the linear and branched BIS-GMA isomers with a proportion of approximately 3 to 1 is explained by the syntheses routes using epoxide reactants (3). By the cleavage of an epoxide ring the methacrylate group may add to the terminal or to the internal carbon atom of the epoxide group. A third isomer, the doubled branched BIS-GMA, should also be expected in the mixture in a quantity of about 2 percent. This isomer was, however, not isolated in this investigation.

The many peaks representing the monomer TUDMA (Fig. 8) indicated many isomers. A synthesis route for this monomer points to formation of several isomers (5). There should also be a probability for the formation of byproducts.

For quantitative determination of the monomers reversed phase cromatography was most suitable. By this method isomers do not separate easily, e.g. the isomers of BIS-GMA appear at the given conditions as one peak. In this way the peak areas, and accordingly the total monomer amount, are registered more accurately than when several peaks represent a monomer. Thus the quantities of BIS-GMA given in Table 9 and 10 represent the contents of the linear and branched isomers of BIS-GMA. Increased amounts of other BIS-GMA derivatives and impurities will result in lower monomer contents. The same effect also appears with other monomers than BIS-GMA.

For qualitative analysis adsorption

Table 10. *Main components in the liquid part of composite and non-composite dental filling materials (wt.-%)*

Materials	HEMA	EGDMA	TEGDMA	BIS-GMA	BIS-MA	BIS-EMA	BIS-PMA	UEDMA	c-HeDMA	C-HaDMA	Remarks
COMPOSITE											
NF			49								a
NM catalyst							100				
NM base							100				
EP catalyst											b
EP base			44	54							
AD catalyst			9	75	9						
AD universal			9	75	9						
MA catalyst			30	55	6						
MA universal			30	55	6						
FF		46									c
PR catalyst			15	81							
PR universal			12	84							
DF			33	35				29			
E catalyst											d,e,f
E base			30	68							
EM A catalyst			38	59							
EM A base			29	28				40			
EM B catalyst			34					66			
EM B base			46	53							
EL			30	69							
ELM			35	39				24			
LF			18	57		21					
PS A catalyst	8								26		
PS A universal				58							g
PS B catalyst			39	58							
PS B universal			17	60		22					
PS C catalyst	5									50	
PS C universal			17	60		22					
RD part A			37	60							
RD part B											h
CC paste B			24	73							
CC paste A			22	72							
SC paste B			50	35							
SC paste A			55	35							
CP catalyst			23	76							
CP universal			22	75							
PF catalyst		30		63							
PF base		32		64							
NON-COMPOSITE											
IP catalyst											i, j
IP base								99			

Remarks:

a) Linear polyurethane of BIS-GMA and an alkane diisocyanate
b) Suspension of 45 % dibenzoyl peroxide and 55 % poly(dimethylsiloxane)
c) Probably a mixture of TUDMA isomers and byproducts.
d) 70 % Butyl-2-butoxyethyl phtalate
e) 8 % dibutyl phtalate
f) Approximately 5 % other phtalates
g) 38 % of a mixture of mono-, di-, tri-, tetra-, and pentaethyleneglycol dimethacrylate. The approximate proportions are 0,5 : 1,0 : 5,1 : 4,3 : 2,4
h) Powder
i) 87 % dibutyl phatalate
j) 9 % dicylohexyl phtalate

chromatography was most suitable. By means of semi-preparative chromatography components could be fractionated and collected for identification by other analytical methods. For characterizing a resin system containing polymers, GPC was an applicable method.

The properties of the resin-based materials can be better understood when the composition of the materials is known, e.g. the quantities of unreacted methacrylate groups in polymerized dental filling and sealant materials are determined by type and quantity of the various monomers (19, 20). This investigation has shown that HPLC is a suitable method for routine analyses of dental resin systems.

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