Materials, Chemicals and Methods for Dental Applications

Johannes Karl Fink

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Contents

Preface

This book focuses on the materials used for dental applications. The text focuses on the basic issues and also the literature of the past decade. The book provides a broad overview of dental materials.

Chemicals that are used for the preparation and fabrication of dental materials are explained. Also, the desired properties of these materials are discussed. The relevance of the chemical, physical, and mechanical properties is elucidated.

Methods for the characterization and classification, as well as clinical studies are reviewed here. In particular, materials for dental crowns, implants, toothpaste compositions, mouth rinses, and also materials for toothbrushes and dental floss are discussed.

For example, in toothpaste compositions several classes of materials are incorporated, such as abrasives, detergents, humectants, thickeners, sweeteners, coloring agents, bad breath reduction agents, flavoring agents, tartar control agents, and others. These chemicals are detailed in the text and the chemical structures for a lot of these chemicals are also shown in the figures.

This text may be of importance for students of dental hygiene, therapy and nursing. Anincrease of knowledge regarding the chemical issues in this field is expected. Also, chemists and other scientists that are interested in these topics could be interested in reading this text to expand their basic knowledge.

How to Use This Book

Utmost care has been taken to present reliable data. Because of the vast variety of material presented here, however, the text cannot be complete in all aspects, and it is recommended that the reader study the original literature for more complete information.

Index

There are three indices: an index of acronyms, an index of chemicals, and a general index. In the index of chemicals, compounds that occur extensively, are not included at every occurrence, but rather when they appear in an important context. When a compound is found in a figure, the entry is marked in boldface letters in the chemical index.

Acknowledgements

I am indebted to our university librarians, Dr. Christian Hasen-hüttl, Dr. Johann Delanoy, Franz Jurek, Margit Keshmiri, Dolores Knabl Steinhäufl, Friedrich Scheer, Christian Slamenik, Renate Tschabuschnig, and Elisabeth Groß for their support in literature acquisition. Many thanks to Dr. Gerhard Kindermann, and coworkers, who inspired me to write this book in the course of my dental treatment there, and in particular thanks to Carmen Piribauer, who checked the manuscript. In addition, many thanks to the head of my department, Professor Wolfgang Kern, for his interest and permission to prepare this text.

I also want to express my gratitude to all the scientists who have carefully published their results concerning the topics dealt with herein. This book could not have been otherwise compiled.

Last, but not least, I want to thank the publisher, Martin Scrivener, for his abiding interest and help in the preparation of the text. In addition, my thanks go to Jean Markovic, who made the final copyedit with utmost care.

Johannes Fink
Leoben, 6th February 2018

1

Dental Materials

The basic materials used in dentistry are metals, ceramics and polymers. There are monographs concerning the materials used in dental applications (1,2).

1.1 History

The early polymeric materials for dental usage were chemically cured, two-component systems (3). These tooth-colored materials provided a better esthetics than amalgam.

However, initially there were many remaining issues left concerning the chemical and physical properties required to withstand the aggressive oral environment. In particular, high shrinkage, high wear, color changes and lack of bonding to tooth surfaces were the issues associated with these early materials.

The history of dental polymeric materials is shown in Table 1.1.

Adhesive systems have been developed that adhere well not only to enamel, but even to moist dentin. In addition, composites have been made stronger, with a higher wear resistance and color stability. Furthermore, both composites and adhesives have been modified to be curable on demand by exposure to light (3).

Improvements on the composite side were achieved, to a great extent, by optimizing the fillers, while the chemistry behind the organic resin matrix remained essentially the same since the pioneering work of R. L. Bowen in the 1960s. Practically all composites employ dimethacrylates such as TEGDMA, Bis-GMA or UDMA, which are radically polymerized as the primary resin.

Table 1.1 History of dental polymeric materials (3).

Event	Date
Synthesis and polymerization of methyl methacrylate	1901
Use of poly(methyl methacrylate) (PMMA) as denture base resin (Germany)	1930
First acrylic filling material	1944
Addition of inorganic fillers (non-bonded) to direct filling materials	1951
Investigation of epoxy resins as direct filling materials	1955
Acid-etch technique introduced (Buonocore)	1955
Dimethacrylates (Bis-GMA) and silanized inorganic filler investigated as direct filling material (Bowen)	1958
Bis-GMA composites marketed	1964
Development of polymeric coatings on fillers (Dental Fillings Ltd)	1968
UV-cured dimethacrylate composite resins	1973
Visible-light-cured dimethacrylate composite resins	1977
Introduction of Filtek™ Silorane System to the market	2007

1.2 Properties

1.2.1 Acronyms for Compounds in Dental Compounds

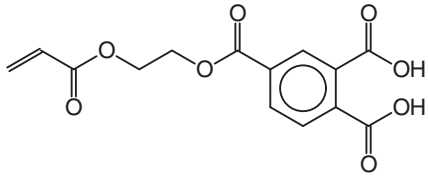
Common acronyms for compounds used in dental formulations are collected in Tables 1.2 and 1.3. Some of these compounds are shown in Figure 1.1. Initiators and inhibitors are shown in Figure 1.2.

1.2.2 Standards in Dentistry

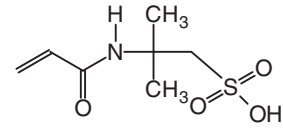
Standards that are used in dentistry are summarized in Table 1.4.

Color stability is an important characteristic of dental materials and it is expected that the test methods in this International Standard ISO 7491:2000 will be referred to in the International Standards specifying such materials (5).

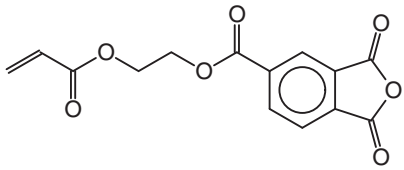
ISO 10477:2004 classifies polymer-based dental crown and bridge materials and also specifies their requirements. In addition, this standard specifies the test methods to be used to determine the compliance with these requirements (6). It is applicable to polymer-based dental crown and bridge materials for laboratory-fabricated



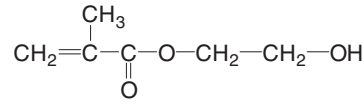
4-Acryloylethyl trimellitic acid



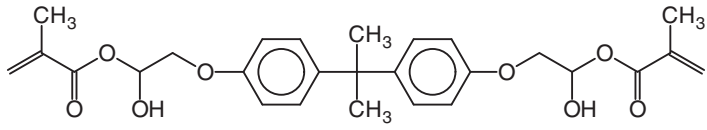
2-Acrylamido-2-methyl-
1-propanesulfonic acid



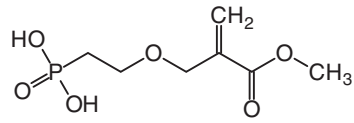
4-Acryloyloxyethyl trimellitate anhydride



2-Hydroxyethyl methacrylate



Bisphenol A diglycidyl methacrylate

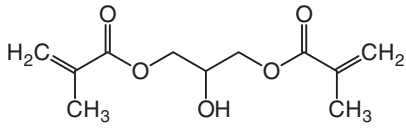


Ethyl 2-[4-(dihydroxyphosphoryl)-2-oxabutyl]acrylate

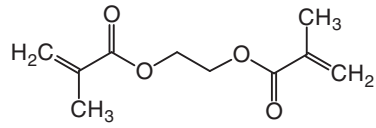
Figure 1.1 Monomers used in dental formulations.

Table 1.2 Acronyms for monomers (4).

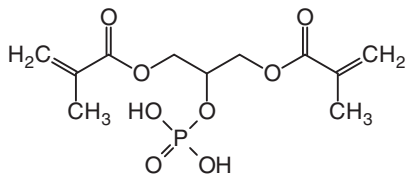
Acronym	Chemical Name
4-AETA	4-Acryloyloxyethyl trimellitate anhydride
4-AET	4-Acryloylethyl trimellitic acid
AMPS	2-Acrylamido-2-methyl-1-propanesulfonic acid
Bis-MEP	Bis[2-(methacryloyloxy)ethyl] phosphate
Bis-EMA	Ethoxylated bisphenol A glycol dimethacrylate
Bis-GMA	Bisphenol A diglycidyl methacrylate
BPDM	Biphenyl dimethacrylate or 4,40-dimethacryloyloxy-ethyloxycarbonylbiphenyl-3,30-dicarboxylic acid
DMAEMA	Dimethylaminoethyl methacrylate
EAEPA	Ethyl 2-[4-(dihydroxyphosphoryl)-2-oxabutyl]acrylate
EGDMA	Ethylene glycol dimethacrylate
GDMA	Glycerol dimethacrylate
GPDM	Glycerol phosphate dimethacrylate
HDDMA	1,6-Hexanediol dimethacrylate
HEMA	2-Hydroxyethyl methacrylate



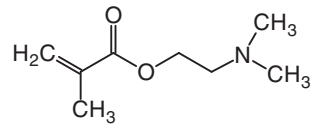
Glycerol dimethacrylate



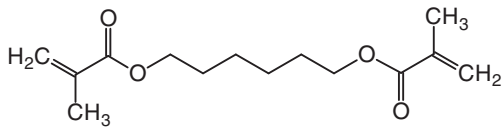
Ethyleneglycol dimethacrylate



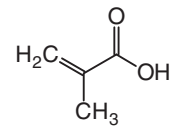
Glycerol phosphate dimethacrylate



Dimethylaminoethyl methacrylate



1,6-Hexanediol dimethacrylate

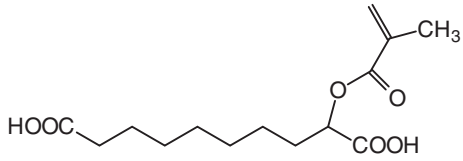


Methacrylic acid

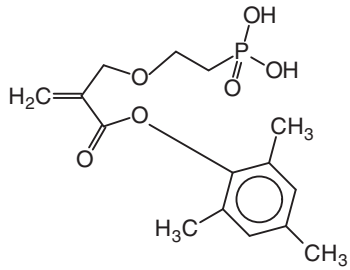
Figure 1.1 (cont.) Monomers used in dental formulations

Table 1.2 (cont.) Acronyms for momomers (4)

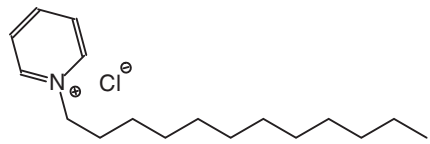
Acronym	Chemical Name
HFGA-GMA	Hexafluoroglutaric anhydride-glycerodimethacrylate adduct
HPMA	2-Hydroxypropyl methacrylate
MA	Methacrylic acid
MAEPA	2,4,6-Trimethylphenyl-2-[4-(dihydroxyphosphoryl)-2-oxabutyl]acrylate
MAC-10	11-Methacryloyloxy-1,10-undecanedicarboxylic acid
10-MDP	10-Methacryloyloxydecyl dihydrogen phosphate
MDPB	Methacryloyloxydodecylpyridinium bromide
4-META	4-Methacryloyloxyethyl trimellitate anhydride
4-MET	4-Methacryloyloxyethyl trimellitic acid
MMA	Methyl methacrylate
MMEP	Mono-2-methacryloyloxyethyl phthalate (sometimes also called PAMA Phthalic acid monomethacrylate)
5-NMSA	*N*-Methacryloyl-5-aminosalicylic acid
NPG-GMA	*N*-Phenylglycine glycidyl methacrylate
NTG-GMA	*N*-Tolyl glycine glycidyl methacrylate or *N*-(2-hydroxy-3-((2-methyl-1-oxo-2-propenyl)oxy)propyl)-*N*-tolyl glycine
PEGDMA	Poly(ethylene glycol) dimethacrylate
PEM-F	Pentamethacryloyloxyethylcyclohexaphosphazene monofluoride
PENTA	Dipentaerythritol pentaacrylate monophosphate
Phenyl-P	2-(methacryloyloxyethyl)phenyl hydrogenphosphate
PMDM	Pyromellitic diethyl methacrylate or 2,5-dimethacryloyloxyethyloxycarbonyl-1,4-benzenedicarboxylic acid
PMGDM	Pyromellitic glycerol dimethacrylate or 2,5-bis(1,3-dimethacryloyloxyprop-2-yloxycarbonyl)benzene-1,4-dicarboxylic acid
Pyro-EMA	Tetramethacryloyloxyethyl pyrophosphate
TCB	Butane-1,2,3,4-tetracarboxylic acid di-2-hydroxyethyl methacrylate ester
TEGDMA	Triethylene glycol dimethacrylate
TMPTMA	Trimethylolpropane trimethacrylate
UDMA	Urethane dimethacrylate or 1,6-di(methacryloyloxyethylcarbamoyl)-3,30,5-trimethylhexane



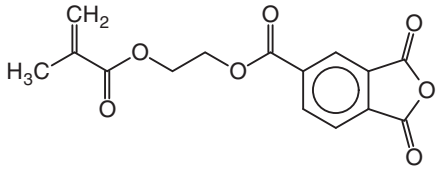
11-Methacryloyloxy-1,10-undecanedicarboxylic acid



2,4,6-Trimethylphenyl-2-[4-(dihydroxyphosphoryl)-2-oxabutyl]acrylate

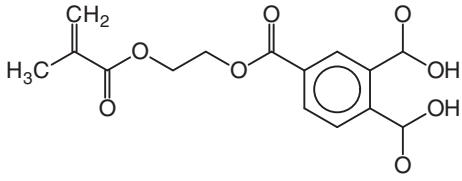


Methacryloyloxydodecylpyridinium bromide

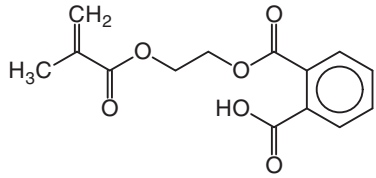


4-Methacryloyloxyethyl trimellitate anhydride

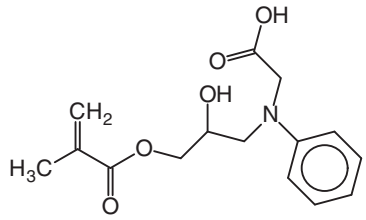
Figure 1.1 (cont.) Monomers used in dental formulations



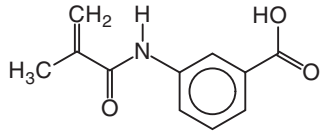
4-Methacryloyloxyethyl trimellitic acid



Mono-2-methacryloyloxyethyl phthalate



N-Phenylglycine glycidyl methacrylate



N-Methacryloyl-5-aminosalicylic acid

Figure 1.1 (cont.) Monomers used in dental formulations

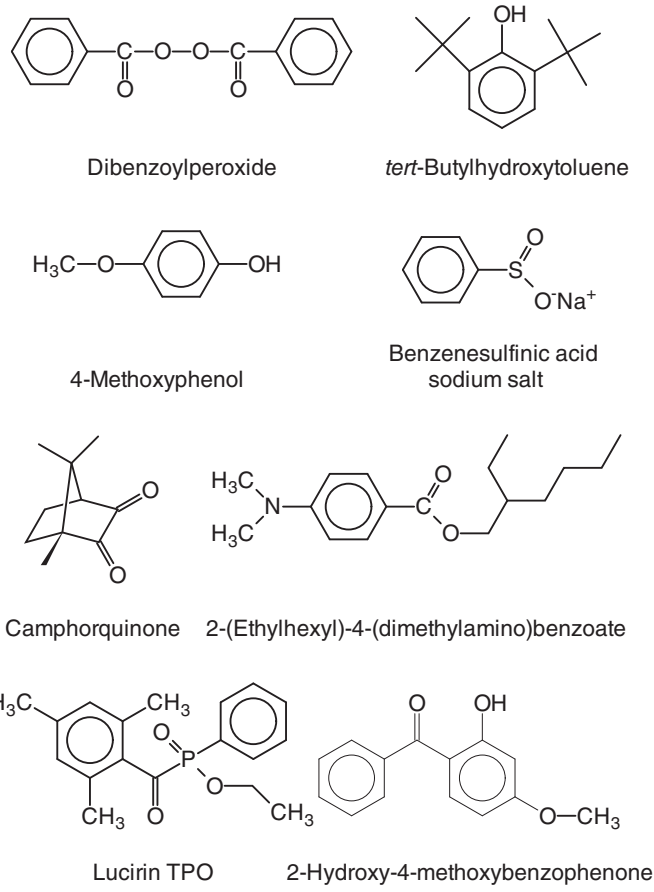


Figure 1.2 Initiators and inhibitors.

Table 1.3 Acronyms for initiators and inhibitors (4).

Acronym	Chemical Name
BHT	Butylhydroxytoluene or butylated hydroxytoluene or 2,6-di-(*tert*-butyl)-4-methylphenol (inhibitor)
BPO	Dibenzoyl peroxide (redox initiator)
BS acid	Benzenesulfinic acid sodium salt (redox initiator)
CQ	Camphorquinone or 1,7,7-trimethylbicyclo-[2,2,1]-hepta-2,3-dione (photoinitiator)
DHEPT	*N*,*N*-Di-(2-hydroxyethyl)-4-toluidine (co-initiator)
MEHQ	4-Methoxyphenol or monoethyl ether hydroquinone (inhibitor)
ODMAB	2-(Ethylhexyl)-4-(dimethylamino)benzoate (co-initiator)
TPO	Lucirin TPO, BASF (photoinitiator) also ethyl(2,4,6-trimethylbenzoyl)phenylphosphinate
UV-9	2-Hydroxy-4-methoxybenzophenone (photoinitiator)

permanent facings or anterior crowns that may or may not be attached to a metal substructure. It also applies to polymer-based dental crown and bridge materials for which the manufacturer claims adhesion to the metal substructure without macromechanical retention such as beads or wires.

ISO 4049:2009 specifies the requirements for dental polymer-based restorative materials supplied in a form suitable for mechanical mixing, hand-mixing, or intraoral and extraoral external energy activation, and intended for use primarily for the direct or indirect restoration of cavities in the teeth and for luting (9).

The change in color of the facial surface gingiva can be used to determine and optimize the efficacy of antigingivitis treatments (11). Chronic inflammatory disease of the gingiva and periodontium results in destruction of gingival connective tissue, periodontal ligament, and alveolar bone. Clinically, inflammation is seen as redness, swelling, and bleeding observed upon probing. The procedure according to ASTM E2545-07 is suitable for use in diagnosis and monitoring, research and development, epidemiological or other surveys, marketing studies, comparative product analysis, and clinical trials.

ISO 4823:2015 specifies the requirements and tests that the state-of-the-art body of knowledge suggests for helping determ-

Table 1.4 Standards in dentistry.

Standard	Name	Reference
ISO 7491:2000	Dental Materials – Determination of colour stability	(5)
ISO 10477:2004	Dentistry – Polymer-based crown and bridge materials	(6)
ISO 10139-1:2005	Dentistry – Soft lining materials for removable dentures – Part 1: Materials for short-term use	(7)
ISO 9333:2006	Dentistry – Brazing materials	(8)
ISO 4049:2009	Dentistry – Polymer-based restorative materials	(9)
ISO 9693-1:2012	Dentistry – Compatibility testing – Part 1: Metal-ceramic systems	(10)
ASTM E2545-07	Standard Test Method for Objective Measurement of Gingival Color Using Digital Still Cameras	(11)
ISO 4823:2015	Dentistry – Elastomeric impression materials	(12)
ISO 6872:2015	Dentistry – Ceramic materials	(13)
ASTM E3014-15	Standard Practice for Managing Sustainability in Dentistry	(14)
ISO 9693-2:2016	Dentistry – Compatibility testing – Part 2: Ceramic-ceramic systems	(15)
ISO 10139-2:2016	Dentistry – Part 2: Materials for long-term use	(16)
ISO/TS 16506:2017	Dentistry – Polymer-based luting materials containing adhesive components	(17)

ine whether the elastomeric impression materials, as prepared for retail marketing, are of the quality needed for their intended purposes (12).

ISO 6872:2015 specifies the requirements and the corresponding test methods for dental ceramic materials for fixed all-ceramic and metal-ceramic restorations and prostheses (13).

The standard ASTM E3014-15 (14) presents a set of generally recognized activities governing the management of sustainability in dentistry and related dental service practice and a management system framework that assists dentists and dental service organizations to enhance their respective organizational performance and effectiveness.

ISO/TS 16506:2017 specifies test methods and information of bond strength to dentine and physical and chemical performances of dental polymer-based luting materials containing adhesive components (17). The materials are supplied in a form suitable for mechanical mixing or hand-mixing, including using auto-mixing tips, for self-curing and/or external energy activation, or non-mixing for external energy activation.

Additional ISO standards with respect to dental materials can be found online in the ISO standards catalogue (18).

1.2.3 Adhesion in Restorative Dentistry

The issues of adhesion in pharmaceutical, biomedical, and dental fields and the theories and mechanisms of adhesion have been described in a monograph (19).

Bonding agents play a crucial role in the effective sealing and retention of resin-based composite restorations, which have been increasingly placed and replaced by dentists (20). Actually, direct adhesive restoration with composite resins has become the procedure of choice for the treatment of anterior and posterior teeth.

However, the long-term durability of those restorations may be compromised due to progressive loss of the integrity of adhesive interfaces. This means that no adhesive strategy is free from technique sensitivity. The specificity and proportion of different constitutive molecules, the interaction between them and substrates can differ greatly from one class of adhesive system to another, which can affect bond quality. An overview of the most important issues in

dental adhesion and adhesive systems has been presented, also their composition and clinical use have been discussed (20).

1.2.4 Fracture Toughness

IPS e.max CAD and IPS e.max Press from Ivoclar Vivadent AG are lithium disilicate glass ceramics marketed as interchangeable materials indicated for the same clinical uses (21).

However, different crystal sizes of lithium disilicate are formed during the processing of each of these materials, a factor that could lead to significantly different mechanical properties.

As mechanical failure is always associated with a crack-initiation/crack-propagation process, fracture toughness values could be useful in comparing the different ceramics and possibly predicting clinical performance (21).

The notchless triangular prism specimen K_{ic} test was used to determine and compare the fracture toughness value K_{ic} of the two glass ceramics. A significantly higher K_{ic} value was determined for IPS e.max Press than for IPS e.max CAD. Fractured surfaces, characterized by SEM, showed a marked difference between the two materials, suggesting a more complete crystallization in IPS e.max Press, which was most likely responsible for the higher K_{ic} determined (21).

1.2.5 Biocompatibility of Dental Adhesives

The accomplishment of developing a truly adhesive bond between a restorative material and the natural tooth structures is the goal of adhesive dentistry (22). Dentine adhesive systems come into close contact with dental and oral tissue, especially the pulp and gingival cells.

Due to this close and long-term contact, adhesives should exhibit a high degree of biocompatibility. Biocompatibility is one of the most important properties of dental materials. It has been demonstrated that various components of adhesives can be released (22).

Numerous *in-vitro* investigations have shown that released monomers and other components can cause damage to cultured cells. In addition, many *in-vivo* studies have shown that uncured components which reach the pulpal space cause inflammatory response

and tissue disorganization. Only a combination of various *in-vitro* and *in-vivo* tests can provide an overview of the interaction of biomaterials with the host. Therefore, it is necessary on a regular basis to carry out and reverify the biological compatibility of the increasing number of new dental materials. Adhesives should be biofunctional, protective, and preventive, with health-promoting effects that contribute to a better prognosis for restorative treatments and their biocompatibility (22).

1.2.6 Testing the Cytotoxicity

The leukocyte viability can be tested using the trypan blue exclusion technique (23). Trypan blue is an azo dye (24). In biosciences, it is used as a vital stain to selectively color dead tissues or cells blue. Trypan blue is shown in Figure 1.3.

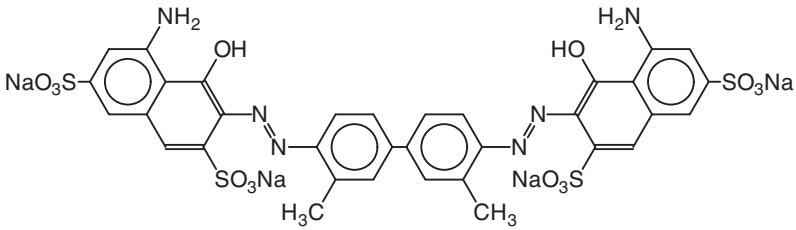


Figure 1.3 Trypan blue.

Since cells are very selective in the compounds that can pass through the membrane, trypan blue is not absorbed in a viable cell. However, trypan blue traverses the membrane in a dead cell. Hence, dead cells are shown as a distinctive blue color under a microscope. Since live cells are excluded from staining, this staining method is also described as a dye exclusion method (24).

In the trypan blue exclusion technique, a cell suspension is mixed with 0.4% trypan blue (Sigma) and analyzed with an Olympus light microscope under 100-fold magnification. For each test group, 1000 leukocytes are analyzed by counting the unstained, i.e., viable cells. Blue-colored cells are considered to be nonviable (25,26).

1.2.7 Degradation of Dental Polymers

The degradation of dental polymers has been described in a monograph (27). Here also the development of appropriate measuring devices has been reviewed.

The principal modes of dental composite material degradation have been reviewed (28). Particular emphasis has been placed on the selection of the monomer resins, the filler content, and the degree of monomer conversion after the clinical materials are cured.

Also, the loss of the mechanical properties and the leaching of components from the composites have been described. Studies dealing with the chemical breakdown of the materials by agents that are present in the oral cavity have been also reviewed. It could be shown that there are many reasons for the biochemical stability of composite resins in the oral cavity (28).

The potential degradation of monomer systems is dependent on the inherent chemical stability of the atomic groups making up the monomer (28). In the case of dental resins, a resistance to oxidative and hydrolytic mechanisms is important, since there is the potential for both to occur as a result of the exposure of components to salivary fluids.

Basically all dental restorative resin monomers in commercial products are based on the coupling of chemical moieties via ester linkages. However, esters are highly prone to hydrolysis. The predominant resin monomers consist of complexed methacrylate resins. These were introduced as early as 1956 (29). A hybrid molecule was described that was polymerized through methacrylate groups coupled with bisphenol A derivatives via ester groups.

An experimental dimethacrylate monomer was synthesized by the reaction of glycidyl methacrylate (29) and was later produced by the reaction of methacrylic acid and the diglycidyl ether of bisphenol A (30).

The synthesis and the characterization of a series of multifunctional methacrylate-based dental monomers have been described (31). Triethanolamine was reacted with glycidyl methacrylate and methacryloyl chloride to produce a series of multifunctional methacrylate-end-capped compounds for conversion to quaternary ammonium fluoride monomers with decyl-substituted side chain to afford antibacterial dental monomers.

The chemical structure of all samples was characterized by Fourier transform infrared and proton nuclear magnetic resonance spectroscopy. The thus obtained monomers have the potential to replace 2,2-bis[4-(2-hydroxy-3-methacryloyloxypropyl)phenyl]-propane as base monomer of universal resin-based dental composites in the presence of a diluting monomer, e.g., triethylene glycol dimethacrylate, due to their multifunctionality as well as their possible antibacterial activity (31).

1.2.8 Effect of Modulated Photoactivation on Polymerization Shrinkage

The influence of modulated photoactivation on axial polymerization shrinkage, shrinkage force, and hardening of light-curing and dual-curing resin-based composites was investigated.

Three light-curing resin composites (SDR bulk-fill, Esthet X flow, and Esthet X HD) and one dual-curing material (Rebilda DC) were subjected to different irradiation protocols with an identical energy density of 27 $J\,cm^{-2}$ (32):

1. High-intensity continuous light (HIC),
2. Low-intensity continuous light (LIC),
3. Soft-start (SS), and
4. Pulse-delay curing (PD).

Axial shrinkage and shrinkage force of specimens with 1.5 *mm* thickness were recorded in real-time for 15 *min*. The Knoop hardness was determined at the end of the experimental observation (32).

The Knoop hardness test is a microhardness test used particularly for very brittle materials or thin sheets, where only a small indentation may be made for testing purposes. A pyramidal diamond point is pressed into the polished surface of the test material with a known load for a specified dwell time, and the resulting indentation is measured using a microscope (33).

Statistical analysis revealed no significant differences among the curing protocols for both Knoop hardness and axial shrinkage, irrespective of the composite material (32).

Pulse-delay curing generated the significantly lowest shrinkage forces within the three light-curing materials: SDR bulk-fill, Esthet X flow, and Esthet X HD. High-intensity continuous light created

the significantly highest shrinkage forces within Esthet X HD and Rebilda DC, and caused significantly higher forces than LIC within Esthet X flow. Pulse-delay curing decreases the shrinkage forces compared with high-intensity continuous irradiation without affecting hardening and the axial polymerization shrinkage (32).

1.2.9 Ceramics Versus Resin Composites

For posterior partial restorations an overlap of indication exists where either ceramic or resin-based composite materials can be successfully applied. The fatigue resistance of modern dental ceramic materials versus dental resin composites has been compared (34).

Bar specimens of five ceramic materials and resin composites were produced according to ISO 4049 (9) and stored for 14 *d* in distilled water at 37°C. The following ceramic materials were selected for testing: A high strength zirconium dioxide (e.max ZirCAD, Ivoclar), a machinable lithium disilicate (e.max CAD, Ivoclar), a pressable lithium disilicate ceramic (e.max Press, Ivoclar), a fluorapatite-based glass-ceramic (e.max Ceram, Ivoclar), and a machinable color-graded feldspathic porcelain (TriLuxe Forte, VITA). The composite materials selected were: an indirect machinable composite (Lava Ultimate, 3M ESPE) and four direct composites with varying filler nature (Clearfil Majesty Posterior, Kuraray; GrandioSO, VOCO, Tetric EvoCeram, Ivoclar Vivadent; and CeramX Duo, Dentsply).

Fifteen specimens were tested in water for initial strength in 4-point bending. Using the same test setup, the residual flexural fatigue strength was determined using the staircase approach after 104 cycles at 0.5 *Hz* (n = 25) (34).

The zirconium oxide ceramic showed the highest initial strength of 768 *MPa* and flexural fatigue strength of 440 *MPa*. Both lithium disilicate ceramics were similar in the static test. However, the pressable version showed a significantly higher fatigue resistance after cyclic loading. Both the fluorapatite-based and the feldspathic porcelain showed equivalent initial and cyclic fatigue properties. From the composites, the highest filled direct material, Clearfil Majesty Posterior, showed a superior fatigue performance. From all materials, e.max Press and Clearfil Majesty Posterior showed the lowest strength loss of 29.6% and 32%, respectively, whereas the