Calcium Aluminate Cement as Dental Restorative. Mechanical Properties and Clinical Durability

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“Life is short, art long, opportunity fleeting, experiment slippery, judgement difficult”

Hippocrates

Till Hans, Noomi, Signe och Noah
Abstract

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In 1995, the Swedish government recommended the discontinuation of amalgam as restorative in paediatric dentistry. Because the mercury content in amalgam constitutes an environmental hazard, its use has declined. The use of resin composites is increasing, but the polymerisation shrinkage of the material is still undesirably high, and the handling of uncured resin can cause contact dermatitis. A new restorative material has recently been developed in Sweden as an alternative to amalgam and resin composite: a calcium aluminate cement (CAC). CAC has been marketed as a ceramic direct restorative for posterior restorations (class I, II) and for class V restorations. This thesis evaluates mechanical properties and clinical durability of the calcium aluminate cement when used for class II restorations. Hardness, \textit{in vitro} wear, flexural strength, flexural modulus, and surface roughness were evaluated. A scanning electron replica method was used for evaluation of the interfacial adaptation to tooth structures \textit{in vivo}. The durability was studied in a 2-year intra-individually clinical follow-up of class II restorations.

Major results and conclusions from the studies are as follows:

- The CAC was a relatively hard material, harder than resin-modified glass ionomer cement but within the range of resin composites. The CAC wore less than resin-modified glass ionomer cement but more than resin composite.
- Flexural strength of CAC was in the same range as that of zinc phosphate cement and far below that of both resin composite and resin-modified glass ionomer cement. Flexural modulus of CAC was higher than both resin composite and resin-modified glass ionomer cement. The low flexural strength of CAC precludes its use in stress-bearing areas.
- Surface roughness of CAC could be decreased by several polishing techniques.
- For CAC restorations, interfacial adaptation was higher to dentin but lower to enamel compared with resin composite restorations. Fractures were found perpendicular to the borders of all CAC restorations and may indicate expansion of the material.
- After 2 years of clinical service, the class II CAC restorations showed an unacceptably high failure rate. Material fractures and tooth fractures were the main reasons for failure.

Key words: mechanical properties, clinical, restorations, ceramic, cement, resin composite, adaptation, SEM
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Preface

This thesis is based on the following papers. The papers are referred to in the text by their Roman numerals:


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Introduction

A historical perspective

The Etruscans came from the Near East and established themselves in Italy between 1000 and 600 B.C. The quality of their work was outstanding, and their skills were put to good use as they fashioned artificial teeth. They devised bands of soft pure gold to surround remaining teeth. A variety of bridgework has been found where they had replaced one or more missing teeth. The artificial replacements were cadaver teeth and in some cases human teeth. In addition, the Mayas in South America (300-900 A.D.) were skilled dentists; they placed beautifully carved stone inlays in carefully prepared cavities in the upper and lower anterior teeth. The inlays were made of minerals such as jadeite, iron pyrites, hematites, turquoise, quartz, serpentine, and cinnabar (from which mercury is extracted). The cavities were prepared in living teeth using a round hard tube similar in shape to a drinking straw made of either jade or copper. The ancient oral surgeons spun a drill between their hands using a rope, and they applied a slurry of powdered quartz in water to cut a perfectly round hole into the enamel and dentin. To fit the cavity exactly, the stone inlays were ground so accurately that many of them have remained in situ for a thousand years. In order to supplement frictional retention, the space between the inlay and cavity walls was sealed with cements. Examination of the remnants shows that they were made of a variety of minerals, principally calcium phosphate (Ring, 1985). There is also evidence that the Mayas performed implantation of non-organic materials in living persons. A mandible fragment dating from about 600 A.D. is the earliest found example of a presumably successful evidence of this. The mandible has been thoroughly studied and three tooth-shaped pieces of shell have been placed in the sockets of three missing incisor teeth.
More than a thousand years before the western world developed an amalgam the Chinese developed a silver amalgam for direct restorations. “Silver paste” is mentioned in Chinese literature as early as 659 A.D. in the “materia medica” of Su Kung. During the Ming period, its formulation was discussed: 100 parts mercury to 45 parts of silver and 900 parts of tin. Trituration of these ingredients produced a paste said to be as solid as silver. Two Frenchmen, the Crawcour brothers, came to America in 1833 with what they claimed to be a new direct restorative—a crude amalgam. Their habit of leaving carious matter in the teeth and their blatant advertising brought down upon them the wrath of many of the most prominent members of the dentist profession, and after a few months they were forced to return to France. The following years led to a debate between organised dentists, who at that time only represented a tiny percentage of practising dentists, and untrained and unprincipled practitioners. The American Society of Dental Surgeons (ASDS) fought against amalgam. This became a very serious matter since every member was required to sign a pledge promising not to use amalgam or risk being expelled. This great controversy was referred to as the Amalgam War. In 1850, the ASDS was forced to rescind the pledge since so many dentists refused to sign it. The Amalgam War ended 1870 through a group of dentists initiating what they called a New Departure: they believed that no single restorative material could serve equally well in every case.

Greene Vardiman Black, born in 1836 in Illinois USA, brought dentistry into the modern era. Black was an indefatigable researcher. He standardised operative procedures more than any dentist before him. He also invented numerous machines for testing alloys. In 1895, he published the first detailed study of the properties of amalgam. His *Dental Anatomy*
appeared in 1890. In 1908, his two-volume *Operative Dentistry* was issued. Apart from several books, he also authored five hundred articles. Two major still famous contributions were his principle “extension for prevention” preparing the margins of a cavity out to point where they can be readily reached by a toothbrush, and his “standardised rules of cavity preparation”. Black made a prophetic statement to some students in 1896: “The day is surely coming, and perhaps within the lifetime of you young men before me, when we will be engaged in practising preventive, rather than reparative, dentistry. When we will so understand aetiology and pathology of dental caries that we will be able to combat its destructive effects by systemic medication” (Ring, 1985).

**Dental restoratives of today**

Amalgam has been an accepted part of dental therapeutics for more than 150 years and is still widely used for posterior restorations. The reasons for its popularity lie in its ease of manipulation, relatively low cost, and long clinical lifetime. Concern has arisen with reference to mercury from both a biological and an environmental point of view. However, it is presently believed that dental amalgam presents an acceptable risk-to-benefit ratio when properly used. In Sweden, the Board of Social Welfare recommends that dentists not use amalgam in children and pregnant women. Until the 1960s, the chemical composition of available amalgam alloys was essentially the same as those of the most successful systems investigated by GV Black. Attempts were made to increase the strength of dental amalgams by increasing the copper content of the traditional amalgam alloy. This development led to the high-copper amalgams of today that became standard in 1986. The typical composition of high-copper amalgam alloy is ∼ 41 – 70% Ag, ∼ 15 – 30% Sn, and ∼ 12 – 28% Cu (O’Brien, 2002).
The amalgamation reaction of the alloy with mercury (known as trituration) for traditional amalgam can be described as follows (van Noort, 1994):

\[
\begin{align*}
\text{Ag}_3\text{Sn} & \quad + \quad \text{Hg} \\
\gamma & \quad \text{mercury powder} \\
\rightarrow & \quad \text{Ag}_3\text{Sn} \quad + \quad \text{Ag}_3\text{Hg}_3 \quad + \quad \text{Sn}_7\text{Hg}_2
\end{align*}
\]

Being the weakest part, the phase \(\gamma_2\), can be minimised in high-copper amalgams where the reaction occurs (van Noort, 1994):

Excess mercury is removed by condensation of the amalgam when placed in the cavity. The total remaining amount mercury in the final restoration is between 45 – 50% (van Noort, 1994). The cavity preparation for amalgam restorations requires undercuts to ensure retention of the amalgam restoration (macromechanical retention), which means that often more tooth substance than the actual caries lesion has to be removed.

Attempts to substitute mercury with gallium have been made and so-called gallium alloys have been introduced for class II restorations. Compared with amalgam these formulas have exhibited a decrease in marginal adaptation, increased number of tooth fractures, increased tooth cracks, and increased tarnish and corrosion (Fidela et al. 1996).

The first material developed as an aesthetic direct restorative was silicate cement. Introduced in the late 1800s, the cement was prepared from alumina-silica glass and a phosphoric acid liquid. The popularity of silicate cement was related to the aesthetic property, the slow release of fluoride,
and the high compressive strength, which placed silicate cement among the strongest inorganic cements known at that time. The fluoride content helped to protect adjacent tooth enamel from caries and was of therapeutic value. Many of the silicate restorations were successful and clinical life times of up to 20 years are reported (Robinson 1971, Bowen et al. 1968). However, silicate cement was highly soluble in oral fluids and lacked adequate mechanical properties. The search for replacement materials led to the development of polymeric anterior restorative resins. Early attempts were made to use resins as a restorative material by employing methyl methacrylate, intended for heat-curing crown and bridge applications. Cold cured resins were widely used at the beginning of the 1950s in class III and V restorations. Later supplemental retention was accomplished by using pins and the acid-etch technique. Soon the dental market became overloaded with resin materials, and a number of problems manifested clinically. The major problem was their shrinkage of up to 10% in volume during polymerisation of the materials. This resulted in marginal leakage and allowed the ingress of bacteria leading to discoloration and recurrent caries and pulpal inflammation (Brännström 1984, Browne and Tobias 1986). The shortcomings of the unfilled resins led to the consistent use of silicate cements. The idea of improving the cold curing resins by mixing them with inorganic filler particles to form the first resin composites and thereby reducing polymerisation shrinkage and thermal expansion dates to the 1950s (Roulet, 2000). In 1956, Bowen performed pioneering work at the National Bureau of Standards in Washington. In the search for an improved resin, a modification of the epoxy resins, known in dentistry as “Bowen’s resin” or bisphenol A-glycidyl methacrylate (Bis-GMA), was introduced in 1962. Bowen combined silica powder with the Bis-GMA monomer and achieved a highly loaded (around 70% by weight) restorative. The addition of filler particles improved the resins, and the so-called resin
composites exhibited increased strength, increased hardness, and decreased polymerisation shrinkage. The superior qualities of the resin composites in comparison with the unfilled resins resulted in universal acceptance of the resin composites in the 1970s. They replaced the unfilled resins and the silicate cements in class III-V cavities. However, as clinical use increased, the disadvantages of the resin composites appeared: poor marginal adaptation, difficulties in maintaining a polished surface, lack of adhesion to tooth structure, and unfavourable aesthetic results. In class I and II cavities, amalgam was the direct restorative used during the 1970s. The search for improved resin composites has continued mainly by trying different types and sizes of filler particles and classification can be made into following groups:

**Macrofilled or conventional resin composites** also called the first generation of resin composites. The filler particles are composed of ground quartz, borosilicate glass, or lithium aluminium glass. The particle size is relatively large, 15 - 35 µm. The size of the fillers did not permit adequate polishing, resulting in rough surfaces and high plaque retention.

**Microfilled resin composites** contain micro-fine filler particles of spherical colloidal silica particles 0.01 – 0.12 µm in diameter. The filler load is limited to 20 – 55% by volume or 35 – 60% by weight. They were developed in 1974 and marketed at the end of the 1970s.

**Hybrid (or blend) resin composites** have a combination of colloidal and fine particles (0.5 –3.0 µm) as filler. Colloidal particles fill the space between the fine particles and the filler content is around 60 – 65% by volume. Hybrids currently dominate the market and can be further categorised by their average filler particle size. Recently nano-
fillers have been introduced in certain resin composites and bonding agents.

In currently available resin composites, the organic matrix is most commonly composed of Bis-GMA and/or urethane dimethacrylate (UDMA). These monomers are highly viscous and require the addition of low molecular weight diluents such as triethyleneglycol dimethacrylate (TEGDMA) to achieve a clinically workable consistency and to allow incorporation of filler. Filler particles are of inorganic composition. In addition to quartz, fine sized particles may be composed of barium or lithium aluminium silicate glasses, borosilicate glass, barium, strontium, or zinc glasses. Resin composites are available in either single-paste or in two-paste systems. Two-paste systems are chemically activated and are supplied as two pastes because the initiator and the accelerator must be kept separate until mixing. Single-paste systems are light-activated and therefore delivered in opaque disposable syringes or capsules. Light-activated composites are currently the most widely used systems. When placed in the cavity, the polymerisation of resin composite is initiated by free radicals (easily activated compounds) that start a conversion of monomers to a highly cross-linked, stiff polymer. A major shortcoming of resin composites is the polymerisation shrinkage, which occurs during conversion of monomers to polymer and is about 1.5 – 4% of the total material volume (O’Brien, 2002). The shrinkage during polymerisation creates stresses at the tooth/resin composite interface that may exceed the strength of bond between the resin composite and enamel or dentin. Bond failure at the interface allows an influx of oral fluid and greatly contributes to the risk of marginal staining, secondary caries, and postoperative sensitivity. Gap formation between resin composites and dentin can be counteracted by the use of dentin bonding systems. An intermediary layer between the dentin
and the resin composite, the hybrid layer, is created. This is made possible by the infiltration of hydrophilic resins into dentin. The intimate contact between resin and dentin inhibits gap formation. In addition, a higher degree of retention of the restoration is achieved leading to minimum removal of healthy tooth tissue for retention reasons. This adhesive property makes minimal cavity design possible due to strictly defect-oriented cavity preparation.

Uncured resin has a high allergy inducing potential. Dental personnel are at considerable risk to develop occupational allergy to acrylics (Kanerva et al. 1994). Resin composites are currently used in all types of permanent restorations.

In 1993, a “new” material became available, compomer (polyacid modified resin composite). Replacement of fillers by ion-leachable aluminosilicate glass was thought to provide fluoride release. However, the clinical significance of fluoride release from compomer restorations seems doubtful. The indication area of compomer materials is somewhat unclear. The durability reported for compomer restorations in deciduous teeth shows differences in success and indicates the technique sensitivity of the material (Andersson-Wenckert et al. 1997, Welbury et al. 2000). Huth et al. (2004) have shown acceptable survival rates for permanent class II compomer restorations after 4-year clinical service.

Parallel to the development of resin composites, a totally different tooth coloured restorative was introduced at the beginning of the 1970s, the glass ionomer cement. The material was developed by Wilson and Kent in 1969 and combined the successful properties of silicate cement and polycarboxylate cement (Wilson and McLean, 1988). Polycarboxylate
cement was used as a lining material and for luting of crowns and bridges. Glass ionomer cement had the strength and almost the translucency of the dental silicate cements and showed a greater resistance to acid attack. It also appeared to have the adhesive and biocompatible properties of polycarboxylate cements. The glasses for the glass ionomer cements contain three main components, silica, alumina, and calcium fluoride. The polyacids mostly used are copolymers of acrylic and itaconic acid or acrylic and maleic acid. The setting of the glass ionomer cement takes place via an acid-base reaction that may be said to have three different phases:

**Dissolution:** The acid goes into solution and slowly degrades the outer layer of the glass particles. This layer becomes depleted with a release of calcium and aluminium ions and only silica gel remains. The hydrogen ions that are released from the acid diffuse to the glass to make up for the loss of calcium, aluminium, and fluoride ions.

**Gelation:** The calcium ions react with the negatively charged carboxyl groups of the acid and cross-link the polyacids into an amorphous network holding the glass particles in place. It is essential that contamination by moisture or drying of the restoration be avoided during this critical phase.

**Hardening:** The aluminium ions start to cross-link with the polymer molecules. Due to their trivalent nature, they ensure a higher degree of cross-linking. The final strength of the material is thereby provided. There is a continuation of the formation of aluminium salt bridges, and water becomes bound to the silica gel, which now surrounds the residual core of each of the glass particles.

An important beneficial property of glass ionomer cement, beside its fluoride release, is its adhesive property, which allows minimal removal of healthy tooth tissue. Reinforcement of glass ionomer cement has been sought by incorporation of amalgam alloy, unfortunately with poor clinical
results. Addition of glass-metal powders resulted in cermet ionomer cements, but they have not been more successful in class II cavities than conventional glass ionomer cement. The newest modification of glass ionomer cement consists in reinforcement with light-curable resin (resin-modified glass ionomer cement). Resin-modified glass ionomer cement shows improved mechanical properties as compared with conventional glass ionomer cements (Peutzfeldt 1996, Uno et al. 1996, Xie et al. 2000). More importantly, they are less technique sensitive. Resin-modified glass ionomer cement has almost totally superseded conventional glass ionomer cement and is currently used in permanent class III and V restorations and in paediatric dentistry.

**Adhesive dentistry**

Black’s methods of cavity preparation were material driven and tooth destructive. Based on the characteristics of amalgam, the dentist was forced to cut large cavities to secure retention of the material. Adhesive dentistry rests on possibilities of adhering the restorations to tooth structure. A new cavity preparation philosophy has emerged, namely that of minimising cavity size and preserving healthy tooth tissue by removing only the caries lesion. These possibilities of minimising cavities and preserving healthy tooth tissue have become reality due to the adhesive character of resin composite and glass ionomer cement. In 1955, Buonocore introduced the adhesive capacity of resin composite by describing the enamel etching technique (Buonocore, 1955). In 1982, Nakabayashi and others described the formation of the hybrid layer, monomer penetration into acid-etched dentin and enamel (Nakabayashi et al. 1982). In 1987, Fusayama described the concept of total etching as a successful way of restoring cavities (Fusayama, 1987). In 1991, Kanca successfully promoted the total etching–total bonding concept in the US in opposition of most dental schools who
claimed that etching dentin is harmful to the pulp (Kanca, 1991). Currently the enamel and dentin bonding technique is accepted world-wide and is routinely used in resin composite restorations and makes it possible to bond the material to the tooth structure without using invasive methods. The modern minimally invasive cavity preparation technique together with the general caries decline in the industrialised countries has the potential to change the view of the dental profession from restorative care to prevention.

**Calcium aluminate cement**

The general definition of the word cement is a material with adhesive and cohesive properties, which makes it capable of bonding mineral fragments into a compact whole (Neville, 1995). This definition embraces a large variety of materials. The principal constituents of the cements for building and civil engineering are compounds of lime (CaO), which leads to the name calcareous cement. When making concrete, the cement has the property of setting and hardening under water by virtue of a chemical reaction. These cements are, therefore, called hydraulic cements. Hydraulic cements consist mainly of silicates and aluminates of lime and can be classified broadly as natural cements, Portland cements, and high-alumina cements (Neville, 1995).

*Natural cement* is the name given to cement obtained by grinding and calcining clay infused limestone. Natural cements are rather variable in quality since the adjustment of composition by blending is not possible.

The name *Portland cement* originates from the resemblance of the quality and colour of the hardened cement to Portland stone, a limestone quarried in Dorset (England). The name describes cement obtained by intimately mixing calcareous and argillaceous, or other silica-, alumina-, and iron oxide-bearing materials. The process of manufacturing Portland
cement essentially consists of grinding the raw materials thoroughly, mixing them in certain proportions, and burning them in a large rotary kiln at a temperature of up to about 1450°C. At this temperature, the material sinters and partially fuses into balls known as clinker. The clinker is cooled down and ground to a fine powder, and some gypsum is added. The resulting product is the commercial Portland cement so widely used throughout the world.

At the beginning of the 20th century, Bied developed high-alumina cement as a solution to the problem of decomposition of Portland cement under sulphate attack (Neville, 1975). A feature of high-alumina cement is its high rate of strength development; about 80% of its ultimate strength is achieved within 24 hours. This is due to rapid hydration, which in turn means a high rate of heat development. The rapid hardening is not accompanied by rapid setting, but the final set follows the initial set more rapidly than is the case with Portland cement. To accelerate the setting, lithium salts can be used. Typically, high-alumina cement has its initial set about 2-hours after application, and its final set within 3-hours (Neville, 1995). A pore solution of high-alumina cement has a pH of 11.4 – 12.5. The phenomenon of conversion is well known for high alumina cement concrete and can be referred to as the change from hexagonal to cubic form of the hydrates. At higher temperature, only the cubic form exists; at room temperature, either form may exist, but the hexagonal crystals slowly convert spontaneously to cubic form. Higher temperature speeds up the process, and intermittent periods of elevated temperature have a cumulative effect on the conversion. Conversion can take place only in the presence of water and not in desiccated concrete since re-dissolving and re-precipitation reactions are involved. The conversion leads to a loss of strength since the crystals differ in density. Under a condition that the overall dimensions of
the body are constant, conversion with internal release of water results in an increase of porosity.

Several details in the manufacture of high-alumina cement contribute to the higher price of this material compared to Portland cement. One is the power consumption: the point of fusion in the furnace takes place at 1600°C. Another is the high cost of bauxite, and a third is the hardness of the material, which leads to a considerable wear of the tube-mills. High-alumina cement concrete can withstand very high temperatures and is one of the foremost refractory materials.

The traditional name—high-alumina cement—refers to the large proportion of alumina. Structural failures associated with high-alumina cement occurred in England in the early 1970s and all structural use ceased. Because of the adverse publicity, the name *aluminous cement* came into use. However, since there are other cements also containing significant proportions of alumina this term is not correct (Neville, 1995). A third name, *calcium aluminate cement*, is more appropriate. Similarly, Portland cement should be referred to as a calcium silicate cement. This latter name, however, is never used. The compositions of calcium aluminate cements and Portland cements are shown in Figure 2.

In 1987, Hermansson, being a chemist and driven by a vision of a new dental restorative more biocompatible than amalgam, developed a calcium aluminate cement for dental purposes (Doxa Certex, 2001). The same year he started the company Doxa Certex AB, later Doxa AB, and the development of the calcium aluminate cement continued. The first patent regarding the material as a dental restorative was achieved in 1990 (Doxa Certex, 2001). During the late 1990s, Doxa AB finalised the recipe and the commercial version of the calcium aluminate cement called Doxadent received CE marking 2000. The product was introduced on the dental market in October the same year (Figure 3).
Figure 2. Compositions of Portland cements and calcium aluminate cements (Büchel et al. 2000).

Figure 3. Tooth 25 restored with the experimental version of Doxadent. Teeth 24, 26 restored with resin composite and 27 with amalgam.
Doxadent was claimed to be an alternative to amalgam in class I, II, and V cavities. Mechanical properties such as hardness, compressive, and flexural strength of the new dental restorative were claimed to be sufficient for its use. The expansion of the calcium aluminate cement was stated to be 0.05-0.1%, which would lead to an elimination of possible gaps between the Doxadent restoration and the tooth. Extensive marketing of the new “bioceramic” restorative followed, and Doxadent was available on the Swedish dental market for about 2 years after its introduction. A successor of the calcium aluminate cement Doxadent is presently under development. The new material is claimed to display increased translucency, biocompatibility, and \textit{in situ} hydroxyapatite forming ability (Lööf \textit{et al.} 2003). To my knowledge, no clinical studies are on their way.

The manufacturer of Doxadent recommended the cavities to have rather parallel axial cavity walls to ensure retention of the restoration. No lining or bonding system was said to be necessary, and Doxadent should be inserted into a moist cavity. Doxadent was supplied as small tablets and activated when dipped into a liquid for about 10 seconds (Figure 4). The tablets were then to be placed in the cavity and crushed with a special instrument rather similar to an ordinary amalgam stopper. The restoration was compacted by hand pressure only. The cavity should be filled with excess, and after initial hardening for about 4-5 minutes, carvers were to be used to create the restoration anatomy. The final finishing was to take place at a later occasion since the material was too soft to tolerate polishing devices at this early stage.
The chemical composition of the hydrated calcium aluminate cement in weight percent is as follows: $\text{Al}_2\text{O}_3$ 43%; CaO 19%; $\text{H}_2\text{O}$ 15%; $\text{ZrO}_2$ 19%; and Si-, Fe-, Mg-, Ti-, and alkali oxides less than 4% (Kraft, 2002). The solution into which the tablet is dipped is ordinary water with a lithium content of 30-90 ppm, which aims to accelerate the hardening process. When the highly compacted calcium aluminate tablet is dipped into the solution, the tablet becomes saturated with water. An acid-base reaction then takes place where the powder acts as the base and water acts as a weak acid. The chemical reaction at 37°C can be summarised as follows (Kraft, 2002):
3 CaO•Al₂O₃ + 12 H₂O → Ca₃[Al(OH)₄]₂(OH)₄ + 4Al(OH)₃
Calcium Aluminate   Water                     Katoite                   Gibbsite

Water dissolves the calcium aluminate and a formation of Ca²⁺, Al(OH)₄⁻, and OH⁻ ions takes place, which is followed almost immediately by precipitation of new solid phases due to saturation of the solution. These precipitates grow until they meet each other. A connected cluster of hydroxide particles is built up continually. Crystallisation takes place and the hydrates grow to sizes of nm to µm (Doxa Certex, 2001).

Clinical durability of direct restoratives

Despite an abundance of dental research, it is not easy to give a clear answer about the actual life span of a certain restorative. This is because of the many variables involved in research and especially in clinical trials. The investigated material and the methods, operators, and the patients influence the results (Letzel 1989, Jokstad and Mjör 1991, Rasmusson and Lundin 1995, Kreulen et al. 1998, Jacobsson et al. 2003).

The durability of amalgam restorations is often used as the gold standard in comparison with new direct restorative materials. Longevity studies of amalgam restorations made under optimal circumstances and a retrospective study show that the survival rate of a class II restoration is in the range of 48-83% at 15 years (Gruythuysen et al. 1996, Letzel et al. 1997, Smales and Hawthorne 1997, Kreulen et al. 1998). The major causes of failure are secondary caries and material or tooth fracture.

In general, it is assumed that longevity studies conducted at dental schools do not reflect clinical reality since the restorations are made under optimal conditions. Multi-centre and cross-sectional studies are thought to
reflect every-day operative dentistry to a higher degree. In cross-sectional studies or replacement studies, the restorations do not have the same baseline. Instead, only restorations needing replacement are counted. This study design does not give the whole answer about the performance of a specific material. In a replacement study conducted in Denmark with 265 participating dentists, a 50% survival rate was shown for class II amalgam restorations at 7.5 years (Qvist J et al. 1990). Major reasons for replacement were secondary caries, material fracture, and unacceptable marginal adaptation.

The development of resin composite and bonding systems has been very rapid, and many materials are on the dental market for a few years before they are replaced. Therefore, no long-term studies of current materials are available. The studies reported below tested materials that are no longer available, and dentin bonding was not routinely used. Longevity studies of these resin composites in class II restorations showed survival rates in the range of 40-80% at 10 years (Lundin and Koch 1999, Raskin et al. 1999, Pallesen and Qvist 2003). 5-year survival rates were in the range of 83-99% (Sturdevant et al. 1988, Wilson and Norman 1991, Wilson et al. 1991, Mjör and Jokstad 1993, El-Mowafy et al. 1994, Rasmusson and Lundin 1995, Kreulen et al. 1999). The main reasons for failure were secondary caries and material fracture. A replacement study showed 50% survival rate at 3-years; the main reasons for failure were secondary caries and material fracture (Qvist V et al. 1990).

The oral environment

For a long-time survival as dental restorative high demands are required for a material. The oral cavity constitutes a tough environment with its combination of moisture, variations in temperature, acid-conditions, micro-flora, and stress loading. In addition, food composition influences the wear
of teeth. It is, for example, known that eating flour processed by a millstone produces heavy tooth abrasion. People who eat mainly vegetables and roots show considerable tooth abrasion. Today, soft drinks with low pH can have an erosive effect.

The tooth should be seen as a unit designed to absorb static as well as dynamic (impact) forces. It is composed of enamel, dentin, root cementum, and pulp. Enamel is the most heavily mineralised and hardest cellular product in the human body. In general, enamel minerals are present as hydroxyapatite (Ca$_{10}$[PO$_4$]$_6$[OH]$_2$). The manner in which enamel is mineralised determines its physical properties. The enamel mantel covering the anatomical crown is of varying thickness from a few microns to a maximum of 2.5 mm. Dentin, a mineralised hard substance, forms the bulk of the tooth and gives it its characteristic shape. The composition of dentin is similar to bone and root cementum but quite different from enamel. Enamel has a higher mineral content. Dentin is softer than enamel but harder than bone and cement. It is also porous and permeable. The most distinctive characteristic of dentin is the presence of cell processes. Throughout life these cell processes can build peritubular dentin and thereby significantly reduce the porosity and permeability of dentin (Schroeder, 1991).

The maximum biting force that can be applied to teeth varies between individuals, but it is generally found greater for men than for women. A maximum biting force of 360-450 N for women and for men 540-650 N has been reported (Okeson, 2003). It has also been noted that the maximum amount of force applied to a molar is usually several times that which can be applied to an incisor. It is assumed that if a force of 756 N were applied to a cusp tip over an area equivalent to 0.039 cm$^2$, the compressive stress would be 193 MPa (Anusavice, 1996). As a curiosity, it
can be mentioned that the highest biting force reported is 4337 N sustained for 2 seconds (Gibbs et al. 1986).

In search of an ideal dental restorative, properties described as desirable include biocompatibility, strength, dimensional stability, adapted thermal properties, tooth binding capacity, sealing ability, excellent aesthetics, and good handling characteristics. However, also the price of a material is of major importance.

Regulations

In the European Community regulation system, dental materials are termed *medical devices used in dentistry*. The CE (Communaute Européenne) marking on product labels denotes the European mark of conformity with the Essential Requirements in the Medical Device Directive. All medical devices marketed in the European Union must have the CE mark of conformity. There are 20 different Directives using the same CE symbol, which allows trade within the European Union. In 1998, the national and regional European approval regulation for dental materials was based on conformity of product standards, such as NIOM, DIN or NF-Dentaire ceased. They were replaced by the European regulatory system, which is based on the Medical Device Directive 93/42/EEC (European Economic Community) and implemented in national laws. In Sweden, it is the law of medical devices SFS (Svensk Författningssamling) 1993:584 and Läkemedelsverkets direction LVFS (Läkemedelsverkets Författningssamling) 2001:6, but the actual use of medical devices in a certain profession is regulated by the National Board of Health and Welfare statute book SOSFS (Socialstyrelsens Författningssamling) 2001:12. The Directive of Medical Devices concerns safety and performance in a general manner. This means that the CE marking is not a quality mark but rather a way of informing the authority that the Directives are accomplished.
Before a new product can enter the European market, technical documentation, risk-assessment, and classification must be available together with assurance of conformity of the Directive. Medical devices can be classified into four groups (I, IIa, IIb, III) depending on the seriousness a possible shortcoming of the product could cause patients or personnel. Direct dental restoratives are classified into group IIa and need evaluation by a notified body before approval (CE certificate). Respective countries approve the notified body, but the producer is free to choose among the notified bodies. The notified body shall render an account to Läkemedelsverket for CE marking. The information should be saved in the European database EUDAMED and is available only to the involved authorities. The CE marking does not require complete declaration of substances; only information about product safety is needed. Products that do not have clinical documentation need to be clinically evaluated, and Läkemedelsverket shall administrate the application for a clinical trial. The producer is responsible for continually supervising the CE marked product and for assuring that the demands stated are fulfilled and that claimed performance is achieved. The producer’s responsibility is only valid if the product has been used according to the instructions.
Aims

This thesis determines the suitability of calcium aluminate cement as a dental restorative in general, and as a class II restorative in particular. The specific purposes of the present thesis were as follows:

- To compare Doxadent with other direct restorative materials with respect to hardness and *in vitro* wear (Paper I).
- To compare Doxadent with other direct restorative materials with respect to flexural strength and flexural modulus (Paper II).
- To determine surface roughness of the experimental version of Doxadent after treatment with different dental polishing techniques *in vitro* (Paper III).
- To compare Doxadent with a resin composite with respect to *in vivo* interfacial adaptation of class II restorations (Paper IV).
- To compare the experimental version of Doxadent with a resin composite with respect to clinical performance of class II restorations in a 2-year follow-up (Paper V).
Material and Method

The materials and methods used in the five studies are described in detail in the respective papers. A brief summary of each is given below.

Hardness and *in vitro* wear (Paper I)

**Hardness**

The investigated materials were calcium aluminate cement (both an experimental and the commercial version of Doxadent), zinc-phosphate cement, glass ionomer cement, resin-modified glass ionomer cement, polyacid-modified resin composite, and resin composite (Table 1). Each material was applied and/or condensed into a cylindrical brass mould (diameter = 10 mm, height = 2 mm). All materials were treated in accordance with the instructions of the manufacturer. One hour after curing, the specimens were transferred to a water bath at 37°C where they were kept for 2 weeks before testing. Five specimens of each material were prepared. The test was performed with a Wallace indentation hardness tester at five points on one side of each sample. A load of 0.01 N was applied for 15 seconds and was followed directly by a load of 1 N for 1 minute. The depth of indentation was then recorded. It follows that Wallace hardness is really a measure of "softness".

**In vitro wear**

The investigated materials are presented in Table 1. Each material was applied and/or condensed into well-defined cavities on a specimen wheel (length = 13 mm, width = 10 mm, height = 2.5 mm). After being stored for 2 weeks, the specimen wheel was mounted in the so-called ACTA wear machine together with an “antagonist” wheel. The sample wheel is one of two motor-driven cylindrical wheels rotating against each other in a bowl...
filled with a slurry to simulate food. A spring force of approximately 15 N pressed the sample wheel against the antagonist wheel. To simulate the sliding action of antagonist teeth, the surface speeds of the two wheels differed by 20%. Before recording of baseline values, a wear run of 20 000 revolutions was performed. The wear experiment itself proceeded for 56 hours and involved 200 000 revolutions of the specimen wheel. The wear was expressed in μm material loss recorded with a dial gauge by use of a mounting that ensured measurement at identical points before and after the wear experiment. Three wheels were produced and run in the wear machine (n = 3). Based on the mean value for each of the three specimens, a new mean value and standard deviation of wear was computed for each material.

**Table 1.** The investigated materials.

<table>
<thead>
<tr>
<th>Brand name</th>
<th>Material</th>
</tr>
</thead>
<tbody>
<tr>
<td>Doxadent</td>
<td>Calcium aluminate cement</td>
</tr>
<tr>
<td>Experimental Doxadent</td>
<td>Calcium aluminate cement</td>
</tr>
<tr>
<td>De Trey Zinc</td>
<td>Zinc phosphate cement</td>
</tr>
<tr>
<td>Chem Fil Superior</td>
<td>Glass ionomer cement</td>
</tr>
<tr>
<td>Vitremer</td>
<td>Resin-modified glass ionomer cement</td>
</tr>
<tr>
<td>Dyract AP</td>
<td>Polyacid-modified resin composite</td>
</tr>
<tr>
<td>Definite</td>
<td>Resin composite</td>
</tr>
<tr>
<td>Filtek Z250</td>
<td>Resin composite</td>
</tr>
<tr>
<td>Tetric Ceram</td>
<td>Resin composite</td>
</tr>
<tr>
<td>Solitaire 2</td>
<td>Resin composite</td>
</tr>
</tbody>
</table>
Flexural strength and flexural modulus (Paper II)
The investigated materials are presented in Table 1. Eighteen rectangular specimens (2x2x25 mm) of each material were produced in split brass moulds (n = 6). All materials were handled in accordance with the instructions of the manufacturers. All specimens were transferred to a water bath at 37°C for 1 day, 1 week, or 2 weeks. The specimens were then ground under running water on #1000 silicon carbide paper, and the dimensions were recorded with a micrometer. Flexural strength and flexural modulus were determined according to ISO standard 4049 for polymer-based materials with a three-point bending test at a cross-head speed of 0.75 mm/min and with 20 mm between the two supports. The test was performed using an Instron Universal Testing Machine, and the specimens were submerged in a water bath maintained at 37°C. A mean value and standard deviation were calculated for each material and storage time.

Surface roughness (Paper III)
The experimental version of Doxadent was studied and a commercial amalgam (ANA 2000) was used as reference material. The specimens were fabricated according to the instructions of the manufacturer and after curing transferred to a water bath at 37°C for 2 weeks. They were then ground on wet #1000 silicon carbide paper to produce a uniform surface, which was used as baseline. The specimens were polished according to eight different treatment modalities (Table 2). The speed produced by the hand-piece was kept constant with a voltmeter for every polishing device used, simulating the clinical situation. The pressure on the specimen was estimated before onset with a scale, and the duration was 60 seconds. Each instrument was used on only one specimen. Surface roughness was determined by measuring roughness average (Ra) in µm with a profilometer, which was
calibrated against a standard before each new measuring session. The cut-off value was kept constant at 0.25 mm with a transverse length of 1.75 mm and a measuring range of 0-9.99 µm and the stylus was moved perpendicular to the path of instrument rotation. Measurements were made at baseline and after each polishing step and means and standard deviations were calculated.

Table 2. Experimental groups with the consecutive polishing steps (a, b, c, d).

<table>
<thead>
<tr>
<th>Group</th>
<th>a</th>
<th>b</th>
<th>c</th>
<th>d</th>
</tr>
</thead>
<tbody>
<tr>
<td>1 DEX</td>
<td>Diamond bur* 76µm</td>
<td>Diamond bur* 46µm</td>
<td>Diamond bur*15µm</td>
<td></td>
</tr>
<tr>
<td>2 DEX</td>
<td>Diamond bur*76µm</td>
<td>Diamond bur*46µm</td>
<td>Diamond bur*15µm</td>
<td></td>
</tr>
<tr>
<td>3 DEX</td>
<td>Sof-lex coarse</td>
<td>Sof-lex medium</td>
<td>Sof-lex fine</td>
<td>Sof-lex extra fine</td>
</tr>
<tr>
<td>4 DEX</td>
<td>Jiffy coarse</td>
<td>Jiffy medium</td>
<td>Jiffy extra fine</td>
<td></td>
</tr>
<tr>
<td>5 DEX</td>
<td>Shofu brownie</td>
<td>Shofu greenie</td>
<td>Shofu super greenie</td>
<td></td>
</tr>
<tr>
<td>6 DEX</td>
<td>Diamond bur*, 46µm</td>
<td>Shofu brownie</td>
<td>Shofu greenie</td>
<td>Shofu sup. greenie</td>
</tr>
<tr>
<td>7 DEX</td>
<td>Aaba universal</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>8 DEX</td>
<td>Diamond bur*, 46µm</td>
<td>Aaba universal</td>
<td></td>
<td></td>
</tr>
<tr>
<td>9 ANA</td>
<td>Subging.finishing bur</td>
<td>Shofu brownie</td>
<td>Shofu greenie</td>
<td>Shofu sup. greenie</td>
</tr>
</tbody>
</table>

DEX = Doxadent experimental, calcium aluminate cement.
ANA = ANA 2000, amalgam. # = 15 500 rpm, *=27 000 rpm.

Interfacial adaptation (Paper IV)

Eight sound and caries-free premolars scheduled for extraction were used in the study, which was approved by the Ethics Committee of the University of Umeå. In each tooth, a mesial and distal box-shaped class II cavity was prepared with a cylindrical diamond bur in a high-speed hand-piece using copious water-cooling. No bevels were prepared and all margins were placed in enamel. The prepared cavities of each tooth were arbitrarily assigned to one of the two experimental groups, Doxadent or Tetric Ceram/Syntac Single-Component. All restorations were made by one
operator. After 1 month functioning time, the premolars were extracted, sectioned, and prepared for SEM analysis.

**Scanning electron microscopy (SEM)**

Sectioning of the restorations was performed in buccal-lingual direction starting at the outermost part of the restoration and continuing with five consecutive sections providing six different cross-sectional surfaces per restoration. To remove the smear layer, the tooth sections were etched with 35% phosphoric acid for 5 seconds and thereafter rinsed with water for 20 seconds and gently dried. Immediately after etching, impressions were made of each section with a polyvinylsiloxane impression material. Positive replica models were fabricated for all sectioned restoration surfaces by pouring epoxy resin into the negative impression. The models were prepared for SEM by mounting on metal stubs and coating with gold by a standard metal evaporation technique. The interface of each restoration to enamel and dentin was evaluated at x275 and x1400 and supplemented when necessary with other magnifications. The quality of the interfacial marginal adaptation between the restorative material and enamel or dentin was evaluated according to a five-point rating scale with increasing degree of openings and breakdown. The scores 1–3 represent acceptable adaptation (with an increase of irregularities at the interface) and scores 4 and 5 represent unacceptable adaptation with crack and gap formation (Table 3). Two operators performed the scoring on microphotographs at a magnification of x275. Quantitative data were obtained by measuring the length of each evaluation score expressed as percentage of the total length of the examined interface. Fractures in enamel or dentin were recorded as well.
Table 3. Scores for interfacial adaptation.

<table>
<thead>
<tr>
<th></th>
<th>Description</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>No interfacial opening or deficiencies</td>
</tr>
<tr>
<td>2</td>
<td>Slight interfacial irregularities</td>
</tr>
<tr>
<td>3</td>
<td>Severe interfacial irregularities, no crack visible</td>
</tr>
<tr>
<td>4</td>
<td>Hairline crack, gap with bottom visible</td>
</tr>
<tr>
<td>5</td>
<td>Severe gap, bottom hardly or not visible</td>
</tr>
</tbody>
</table>

**A two-year clinical evaluation (Paper V)**

Patients visiting the clinic for dental hygienists education in Umeå for their yearly examination and which were in the need of at least one pair of class II restorations were asked to participate in the study: 57 patients attended and a total of 122 posterior restorations were placed. High caries activity, periodontal condition, or para-functional habits were not exclusion criteria. All restorations were replacements of class II amalgam restorations because of secondary caries, fracture, or “non-amalgam” reasons. Each patient received at least two class II restorations of the same size, one in the experimental version of calcium aluminate cement and one in the resin composite (Tetric Ceram/Excite). The resin composite was placed if possible in the same type of tooth as the experimental version of Doxadent in order to make an intra-individual comparison possible. One dentist experienced with both materials placed all restorations according to the instructions of the manufacturer. Fifty percent of the proximal cervical margins were situated apical to the cement-enamel junction. No bevelling of the cavity margins was performed. The experimental Doxadent restorations were finished after at least two days in contrast to the resin composite restorations, which were finished immediately.
Evaluation
Each restoration was evaluated after final finishing (baseline) and after 6, 12, and 24 months. A slight modification of the USPHS (United States Public Health Service) criteria was used to evaluate the quality of the restorations by two calibrated observers (Table 4). Disagreement was resolved by consensus. Bite-wing radiographs were taken of all restorations and colour slides were made of selected cases. The evaluated characteristics of the restorations were described by descriptive statistics, using frequency distributions of the scores.

<table>
<thead>
<tr>
<th>Category</th>
<th>Score</th>
<th>Criteria</th>
</tr>
</thead>
<tbody>
<tr>
<td>Anatomical form</td>
<td>0</td>
<td>Restoration contiguous with tooth anatomy</td>
</tr>
<tr>
<td></td>
<td>1</td>
<td>Slightly under- or over-contoured, contact slightly open</td>
</tr>
<tr>
<td></td>
<td>2</td>
<td><em>Under-contoured, dentin or base exposed, contact is faulty, reduced occlusal height, occlusion affected</em></td>
</tr>
<tr>
<td></td>
<td>3</td>
<td><em>Restoration is missing partially or totally, tooth fracture, restoration causes pain</em></td>
</tr>
<tr>
<td>Marginal adaptation</td>
<td>0</td>
<td>Restoration contiguous with existing anatomic form, explorer does not catch</td>
</tr>
<tr>
<td></td>
<td>1</td>
<td>Explorer catches, no crevice is visible into which explorer will penetrate</td>
</tr>
<tr>
<td></td>
<td>2</td>
<td>Crevice at margin, enamel exposed</td>
</tr>
<tr>
<td></td>
<td>3</td>
<td><em>Obvious crevice, dentin or base exposed</em></td>
</tr>
<tr>
<td></td>
<td>4</td>
<td><em>Restoration mobile, fractured or missing</em></td>
</tr>
<tr>
<td>Colour match</td>
<td>0</td>
<td>Very good colour match</td>
</tr>
<tr>
<td></td>
<td>1</td>
<td>Good colour match</td>
</tr>
<tr>
<td></td>
<td>2</td>
<td>Slight mismatch in colour, shade or translucency</td>
</tr>
<tr>
<td></td>
<td>3</td>
<td><em>Obvious mismatch, outside the normal range</em></td>
</tr>
<tr>
<td></td>
<td>4</td>
<td><em>Gross mismatch</em></td>
</tr>
<tr>
<td>Marginal discoloration</td>
<td>0</td>
<td>No discoloration evident</td>
</tr>
<tr>
<td></td>
<td>1</td>
<td>Slight staining can be polished away</td>
</tr>
<tr>
<td></td>
<td>2</td>
<td>Obvious staining can not be polished away</td>
</tr>
<tr>
<td></td>
<td>3</td>
<td><em>Gross staining</em></td>
</tr>
<tr>
<td>Surface roughness</td>
<td>0</td>
<td>Smooth surface</td>
</tr>
<tr>
<td></td>
<td>1</td>
<td>Slightly rough or pitted</td>
</tr>
<tr>
<td></td>
<td>2</td>
<td>Rough, can not be refinished</td>
</tr>
<tr>
<td></td>
<td>3</td>
<td><em>Surface deeply pitted, irregular grooves</em></td>
</tr>
<tr>
<td>Caries</td>
<td>0</td>
<td>No evidence of caries contiguous to the restoration margin</td>
</tr>
<tr>
<td></td>
<td>1</td>
<td><em>Superficial caries, no operative treatment necessary</em></td>
</tr>
<tr>
<td></td>
<td>2</td>
<td><em>Caries adjacent to restoration, operative treatment indicated</em></td>
</tr>
</tbody>
</table>
**Statistical analysis**

**In paper I**, the data were analysed by ANOVA, Newman-Keuls´ multiple range test and regression analysis.

**In paper II**, the data were analysed by Kruskal-Wallis H tests and Mann-Whitney U tests. For each material, the influence of storage time in water was analysed using one-way ANOVA and Newman-Keuls´ multiple-range tests or in the case of lack of homogeneity of the standard deviations, by Kruskal-Wallis H tests.

**In paper III**, the data were analysed by general linear module, multivariate analysis (ANOVA), and exact test (Montecarlo).

**In paper IV**, the data were analysed by Mann-Whitney U-test and exact test (Montecarlo).

**In paper V**, the data were analysed by Friedman´s two-way analysis of variance test.

In all studies, the level of significance was set at $P<0.05$. 
Results

Hardness and in vitro wear (Paper I)

Hardness values varied between 7.1 and 11.7 µm for the investigated materials. The four resin composites, all for posterior use, differed significantly in hardness showing hardness values between 8.2 and 11.7 µm. Doxadent showed a hardness of 9.2 µm and zinc phosphate cement 9.1 µm. The experimental version of Doxadent (7.1 µm) was significantly harder than any of the other materials. The resin composites and the polyacid-modified resin composite wore significantly less than the other materials tested (20.5 – 40.3 µm). The wear values of Doxadent, the experimental version of Doxadent, and the zinc phosphate cement were 52.9 – 59.5 µm. These materials wore significantly less than the glass ionomer cements with values of 73.9 – 108.5 µm. No linear correlation was found between wear and hardness.

Flexural strength and flexural modulus (Paper II)

Mean values and standard deviations of flexural strength and flexural modulus for each material and storage time are presented in Table 5. Irrespective of storage time, the following general ranking was found between the flexural strengths of the different types of materials with the strongest material mentioned first: resin composite = polyacid-modified resin composite > resin-modified glass ionomer cement > conventional glass ionomer cement > Doxadent ≥ experimental Doxadent ≥ zinc phosphate cement. With respect to flexural modulus, almost the opposite ranking was obtained: experimental Doxadent = Doxadent ≥ zinc phosphate cement ≥ conventional glass ionomer cement ≥ resin-modified glass ionomer cement = polyacid-modified resin composite = resin composite.
For Doxadent and the experimental version of Doxadent, flexural strength decreased significantly from 1 week to 2 weeks, whereas flexural modulus remained unchanged throughout the experimental period.

**Table 5.** Flexural strength and flexural modulus of 1-day-old, 1-week-old, and 2-week-old specimens. Mean and standard deviation.

<table>
<thead>
<tr>
<th>Material</th>
<th>Flexural strength (MPa)</th>
<th>Flexural modulus (GPa)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>1 day</td>
<td>1 week</td>
</tr>
<tr>
<td>Doxadent</td>
<td>18 ± 4</td>
<td>22 ± 4</td>
</tr>
<tr>
<td>DEX</td>
<td>20 ± 2</td>
<td>19 ± 8</td>
</tr>
<tr>
<td>De Trey Zinc</td>
<td>13 ± 3</td>
<td>15 ± 3</td>
</tr>
<tr>
<td>Chem Fil S$\S$</td>
<td>32 ± 8</td>
<td>40 ± 10</td>
</tr>
<tr>
<td>Vitremer</td>
<td>78 ± 10</td>
<td>68 ± 13</td>
</tr>
<tr>
<td>Dyract AP</td>
<td>111 ± 16</td>
<td>114 ± 17</td>
</tr>
<tr>
<td>Tetric Ceram</td>
<td>110 ± 4</td>
<td>102 ± 4</td>
</tr>
<tr>
<td>Filtek Z250</td>
<td>136 ± 18</td>
<td>129 ± 15</td>
</tr>
<tr>
<td>Solitaire 2</td>
<td>104 ± 8</td>
<td>88 ± 10</td>
</tr>
<tr>
<td>Definite</td>
<td>107 ± 6</td>
<td>97 ± 8</td>
</tr>
</tbody>
</table>

$\S$ = Doxadent experimental version, $\S$ = Chem Fil Superior

**Surface roughness (Paper III)**

The significantly smoothest surface of the experimental version of Doxadent was obtained with the Sof-Lex system (Table 6, group 3). The fine Sof-Lex disc gave a Ra value of 0.26 µm. Extra fine Sof-Lex disc re-increased surface roughness to a Ra value of 0.44 µm. Diamond burs at
higher speed, points, and polisher (group 1, 4-7) gave statistically similar Ra values between 0.58 - 0.72 µm. The phenomena with re-increased surface roughness was also seen when the fine polisher Super Greenie was used (group 5, 6). The diamond burs at lower speed (group 2) gave an increased surface roughness compared to baseline, which could not be reduced by any of the finer polishing steps.

Table 6. Mean and standard deviation of roughness average Ra (µm) for each polishing step. The lowest Ra-value for each polishing method is given in italics.

<table>
<thead>
<tr>
<th>Group</th>
<th>Baseline</th>
<th>a</th>
<th>b</th>
<th>c</th>
<th>d</th>
</tr>
</thead>
<tbody>
<tr>
<td>1 DEX</td>
<td>1.69 ± 0.41</td>
<td>2.16 ± 0.35</td>
<td>1.27 ± 0.39</td>
<td>0.66 ± 0.16</td>
<td>-</td>
</tr>
<tr>
<td>2 DEX</td>
<td>1.50 ± 0.30</td>
<td>2.61 ± 0.28</td>
<td>2.30 ± 0.48</td>
<td>2.43 ± 0.49</td>
<td>-</td>
</tr>
<tr>
<td>3 DEX</td>
<td>1.59 ± 0.28</td>
<td>0.74 ± 0.09</td>
<td>0.41 ± 0.06</td>
<td>0.26 ± 0.02</td>
<td>0.44 ± 0.05</td>
</tr>
<tr>
<td>4 DEX</td>
<td>1.40 ± 0.27</td>
<td>1.32 ± 0.21</td>
<td>1.00 ± 0.10</td>
<td>0.72 ± 0.09</td>
<td>-</td>
</tr>
<tr>
<td>5 DEX</td>
<td>1.67 ± 0.24</td>
<td>0.78 ± 0.16</td>
<td>0.64 ± 0.10</td>
<td>1.30 ± 0.70</td>
<td>-</td>
</tr>
<tr>
<td>6 DEX</td>
<td>1.45 ± 0.23</td>
<td>2.68 ± 0.78</td>
<td>1.12 ± 0.28</td>
<td>0.58 ± 0.08</td>
<td>1.60 ± 0.50</td>
</tr>
<tr>
<td>7 DEX</td>
<td>1.34 ± 0.19</td>
<td>0.72 ± 0.09</td>
<td>-</td>
<td>-</td>
<td>-</td>
</tr>
<tr>
<td>8 DEX</td>
<td>1.60 ± 0.26</td>
<td>1.72 ± 0.17</td>
<td>1.09 ± 0.29</td>
<td>-</td>
<td>-</td>
</tr>
<tr>
<td>9 ANA 2000</td>
<td>1.10 ± 0.13</td>
<td>0.41 ± 0.08</td>
<td>0.26 ± 0.02</td>
<td>0.21 ± 0.03</td>
<td>0.17 ± 0.01</td>
</tr>
</tbody>
</table>


### Interfacial adaptation (Paper IV)

The interfacial adaptation of Doxadent to dentin and enamel was compared with that of a resin composite (Tetric Ceram/Sytac Single). Doxadent showed significantly higher percentage of acceptable interfacial adaptation (score 1-3) to dentin (72% versus 49%), but significantly lower percentage
of acceptable adaptation to enamel (84% versus 93%) (Figures 5 and 6). Two different types of fractures, parallel and perpendicular, were observed. The mean length of evaluated enamel boarder per restoration measured was 2.90 ± 1.38 cm for the Tetric Ceram/Syntac Single group and 2.98 ± 1.13 cm for the Doxadent group. Parallel fractures were observed for Tetric Ceram/Syntac Single in 0.19 ± 0.14 cm per restoration and for Doxadent in 0.02 ± 0.02 cm per restoration. No parallel fractures were observed in dentin. Perpendicular enamel fractures radiating from the restoration margins were frequently seen in the Doxadent group but never observed in dentin (Figures 7 and 8). The total amount of perpendicular fractures was 80 for the Doxadent restorations and 3 for the Tetric Ceram/Syntac Single restorations.

Figure 5. Acceptable interfacial adaptation to enamel of one-month-old class II Doxadent restoration. Original magnification x275.
Figure 6. Higher magnification of the interfacial adaptation shown in Figure 5. Original magnification x1400.

Figure 7. A perpendicular enamel fracture radiating from a one-month-old class II Doxadent restoration. Original magnification x275.
A two-year clinical evaluation (Paper V)

All, except one patient with one pair of restorations (experimental version of Doxadent and Tetric Ceram/Excite), were evaluated during the 2-year period. At the 6-month recall, five unacceptable experimental Doxadent restorations (8%) were observed. After 12 months, ten experimental Doxadent (17%) and two Tetric Ceram/Excite (3%) unacceptable restorations were seen. At the 24-month recall, eleven unacceptable experimental Doxadent restorations (18%) were found. This resulted in a significantly higher cumulative failure frequency for the experimental Doxadent restorations (43%) than for the Tetric Ceram/Excite restorations (3%). Main reasons for unacceptable restorations were material fracture, tooth fracture, and erosion (Figures 9 and 10). Reasons for failure and number of unacceptable restorations are presented in Table 7. Small, but still acceptable defects, in the form of marginal ridge chip fractures, were observed in another 12 experimental Doxadent restorations.
Table 7. Reasons for failure and number of unacceptable restorations at 6 months, 1 year, and 2 years.

<table>
<thead>
<tr>
<th></th>
<th>6 months</th>
<th></th>
<th>1 year</th>
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<th>2 years</th>
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<tr>
<td></td>
<td>DEX</td>
<td>TCE</td>
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<tr>
<td>Partial fracture</td>
<td>1</td>
<td>-</td>
<td>3</td>
<td>-</td>
<td>3</td>
<td>-</td>
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<tr>
<td>Cusp fracture</td>
<td>1</td>
<td>-</td>
<td>1</td>
<td>1</td>
<td>4</td>
<td>-</td>
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<tr>
<td>Proximal chip fracture</td>
<td>2</td>
<td>-</td>
<td>3</td>
<td>-</td>
<td>1</td>
<td>-</td>
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<tr>
<td>Erosion</td>
<td>1</td>
<td>-</td>
<td>2</td>
<td>-</td>
<td>2</td>
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<tr>
<td>Endodontic treatment</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>1</td>
<td>1</td>
<td>-</td>
</tr>
<tr>
<td>Caries and fracture</td>
<td>-</td>
<td>-</td>
<td>1</td>
<td>-</td>
<td>-</td>
<td>-</td>
</tr>
<tr>
<td>Total number of failures</td>
<td>5</td>
<td>0</td>
<td>10</td>
<td>2</td>
<td>11</td>
<td>0</td>
</tr>
<tr>
<td>% Failures</td>
<td>8</td>
<td>0</td>
<td>17</td>
<td>3</td>
<td>18</td>
<td>0</td>
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</table>

DEX= Doxadent experimental version, calcium aluminate cement. TCE = Tetric Ceram/Excite, resin composite.

Figure 9. Molar restored with the experimental version of Doxadent. Scored as unacceptable due to material fracture.
Discussion

The term “ceramic” is a collective term for products consisting of non-metallic inorganic compounds normally made usable by high temperature treatment (Büchner et al. 1989). There is also a broader definition of “ceramic” that leaves out the heat treatment, and when used encloses not only the traditional ceramics but also cements (van Vlack, 1973). However, in dentistry, the term ceramic is usually applied for heat-treated ceramics used as dental prostheses. This means that the most appropriate term for the dental restorative Doxadent is calcium aluminate cement and that the term “bioceramic” as used by the manufacturer, is in conflict with usual dental terminology.
In paper I and II, comparison not only of calcium aluminate cement with class II restoratives (resin composites) was desired but also with materials not normally used in posterior cavities (resin-modified glass ionomer cement, conventional glass ionomer cement and zinc phosphate cement) of permanent teeth. The wear of resin composites in the ACTA machine has been found to correlate with clinical wear (Pallav et al. 1988). The clinical relevance of the ACTA wear test performed on calcium aluminate cement is unknown but in the clinic wear also may depend on the extent to which this material is affected by solution and disintegration. The wear results of Doxadent did not differ significantly from that of zinc phosphate cement. *In vivo* studies have shown that zinc phosphate cement has greater solubility and disintegrates more readily than glass ionomer cements and resin cements (Phillips et al. 1987, Hersek and Canay 1996, Buchalla et al. 2000,). This was not reflected in the wear results by the ACTA wear machine. A possible explanation could be that the wear recorded with the ACTA wear machine only to a minor degree is influenced by material solubility and disintegration, since the test is carried out in a relatively short period (56 hours), and the wear measured is thus an accelerated wear. This means that the wear of the surface proceeds with greater rate than does a possible destruction by solution and disintegration. In the case that calcium aluminate cement is also affected by solubility and disintegration in the oral cavity, this effect should be added to the relatively high abrasion of the material in the ACTA wear machine. In corroboration with this theory, both versions of calcium aluminate cement were found to wear markedly more, relatively speaking, than the other materials during the initial wear run of 20 000 revolutions. Engqvist et al. (2002) caution that the interpretation of accelerated wear for calcium aluminate cement could be misleading. Since no time for re-hydration is included in the ACTA wear test, these authors anticipated that wear is less pronounced in the clinical situation due to re-
hydration of the calcium aluminate cement in the surface area. This statement illustrates the difficulties of *in vitro* wear tests to accurately simulate the oral conditions and the need to better understand wear mechanisms occurring in the mouth. It has been shown that not all *in vitro* wear tests resulted in the same ranking order of resin composite and amalgam (Theo *et al*. 1998). The *in vivo* wear process is complicated by the fact that wear is the result of several superimposed mechanisms, and to understand the *in vivo* wear situation requires more than just the understanding of masticatory forces. Temperature changes, microbial composition, food intake, and acid-conditions contribute substantially to the complexity of wear in the oral cavity. Whether *in vitro* wear test of a material is valid or not can only be known if correlations can be made with clinical wear data. The clinical wear of experimental and commercial Doxadent was not measured in any of the studies. However, failures due to erosion were seen in the clinical study.

As with *in vitro* wear, Doxadent did not differ significantly from zinc phosphate cement with respect to hardness. Wallace hardness and *in vitro* wear were not well correlated, which was especially evident for the two versions of the calcium aluminate cement. The experimental version of Doxadent was significantly harder than the marketed version, but the two materials did not differ with respect to wear. This implies that wear of the calcium aluminate cements is determined by other factors than hardness. According to the manufacturer, the two versions differed with respect to the process of tablet pressing, the process having been standardised and optimised to provide the marketed version with homogeneous and improved handling characteristics.

Flexural strength and flexural modulus of the calcium aluminate cement were determined according to the ISO 4049, a standard for polymer-based
filling restoratives (paper II). The standard was chosen since flexural strength is a key mechanical property and Doxadent was claimed to be an alternative for resin composites in class II cavities. The mechanical property included in the ISO standard (#9917) for dental water-based cements, which comprises glass ionomer cements and zinc phosphate cements, is that of compressive strength. A number of materials were tested to make a comparison possible. When testing mechanical properties of ceramic materials it is important to produce specimens without porosities and mechanical induced surface flaws. If a flaw is induced for instance during specimen preparation, the flaw may be decisive for the outcome of the test and not the inherent property of the material. The specimen geometry is also of interest. If the intention is to find the inherent strength of a ceramic material, a round and edge free specimen geometry should be chosen since crack initiation often occurs at sharp edges. The rectangular shape (2x2x25 mm) of the specimen used in ISO 4049 is not ideal for cements, but it does reflect the clinical situation to a certain extent. It may be that the determined flexural strength for the calcium aluminate cement rather reflects the size and distribution of porosities or in other words the handling characteristics, than the true flexural strength.

The supposed change in the manufacturing process between the experimental and the commercial version of Doxadent did not lead to any decisive improvements in mechanical properties. The prolonged water storage of two weeks for the calcium aluminate cement weakened the material. This was not expected since the material is claimed to increase in strength for several weeks post hardening. The flexural strength of calcium aluminate cement reached only about 20% of the strength of the polyacid-modified resin composite and the resin composites, materials that are commonly used for class II restorations. Both clinical and \textit{in vitro}
studies have pointed out the relevance of sufficient flexural strength for restoratives used in stress bearing areas (Östlund et al. 1992, Peutzfeldt 1996, Qvist et al. 1997, Ferracane and Condon 1999, Cobb et al. 2000). Obviously, CE marking of Doxadent did not encompass flexural strength probably because of the difficulty to categorise the material into applicable ISO standards.

It has been suggested that surface roughness influences the degree of plaque accumulation to a higher degree than the surface free energy (Quirynen et al. 1990). However, there is no known common threshold value of surface roughness regarding plaque accumulation for different dental materials (Bollen et al. 1997). The range of surface roughness for different intra-oral materials is wide and the impact of dental treatments on surface roughness is material-dependent. No difference in plaque accumulation and gingival inflammation were found clinically between the calcium aluminate cement, resin composite, and enamel (Konradsson and van Dijken, 2002). Surface roughness has been shown to influence the strength of dental ceramics (Chu et al. 2000, de Jager et al. 2000). Polishing the experimental version of Doxadent with diamond burs at lower speed seems contraindicated since surface roughness did not decrease even after use of the finer polishing steps (paper III). Diamond burs at higher speed or different polishing points decreased surface roughness and gave rather similar results, but gross reduction of material when polished at higher speeds could be observed. The smoothest surfaces for the experimental version of Doxadent was found with Sof-Lex discs. However, the use of Sof-Lex discs on posterior restorations is of little clinical significance due to the tooth anatomy and the limited access. The findings of re-increased surface roughness after the final polishing with extra fine Sof-Lex disc and the extra fine silicone polisher
Super-Greenie were unexpected. An explanation for the absence of a further decrease in surface roughness is that the heterogeneous microstructure of the calcium aluminate cement restricts further improvement of surface smoothness. For amalgams, it has been shown that porosity is one of the structural features that severely limits the polishability and that the extent of porosity formation is influenced by operator variables (Letião, 1980 and 1983). Therefore, it is likely that the operator variable limits the decrease of surface roughness also for the calcium aluminate cement. Scanning electron microscopy was used to evaluate the surface texture of the specimens polished with the Sof-Lex discs, and porosity formation could be seen. However, the extent of porosity could not alone explain the relatively high re-increased Ra values. An additional explanation may be found in the fact that mechanical effects can reduce the life of a ceramic by producing surface flaws that are larger than the original flaws. Mechanically induced surface flaws are cracks caused by localised stress concentration. The stress concentration can result from impact or from point or line contact loading at the interface (Richardsson, 1992).

The evaluation of interfacial adaptation was based on a semi-quantitative method (paper IV). The interfacial border is characterised according to a qualitative scale, and the scores were transformed to relative percentage of the total examined interface. Roulet et al. (1989) have described a similar method, and the method has been used in other in vivo investigations (van Dijken and Hörstedt 1986, van Dijken et al. 1998, Andersson-Wenckert et al. 1998 and 2002, Lindberg et al. 2000). When using the replica method, it is assumed that the conformity with the specimen is very accurate. Grundy (1971) showed a high degree of agreement when he compared specimens observed directly with replica models in SEM. The semi-quantitative
scoring system is highly dependent on reproducibility. Consequently, calibration exercises were conducted. The inter-examiner reliability gave a kappa value of 0.77 between the two operators involved in scoring. The investigated restorations were made in vivo since the interest was focused on how Doxadent restorations are influenced by the oral environment. An intra-individual study design was chosen, in which two restorations were placed in the same tooth. This means that the two restorations were exposed to the same clinical environment. After one month functioning time, the teeth were extracted. During extraction the tooth is exposed to a relatively high force, great care was therefore taken not to damage the restorations. Even if its likely that stress concentrations due to extraction can influence the tooth/restoration interface, a possible trauma would be evident for both restorations. The extent of gap free percentage of interfacial adaptation for Doxadent was found to be lower to enamel and higher to dentin compared with that of the resin composite restorations (Tetric Ceram/Syntac Single Component). Other studies using other bonding agents have shown a higher percentage of gap free interfacial adaptation between dentin and resin composite than the present study (van Dijken et al. 1998, Lindberg et al. 2000). The bonding agent used was chosen since it was a common material at the time of the study. However, Vargas et al. (1997) have shown low shear bond strength for the bonding agent. It has also been demonstrated that the bonding agent used does not completely penetrate and saturate the collagen network which causes decreased adaptation and micro-leakage (Perdigão et al. 1997, Manhart et al. 2001). These findings could explain the relatively low interfacial adaptation found for the bonding agent used in the present study.

The difference in acceptable adaptation between enamel and dentin for resin composite can be explained by the problems associated with resin penetrating “wet” dentin. Doxadent showed a relatively high
adaptation to both dentin and enamel, and the cavities were cleaned with water only and not conditioned in any other way according to the manufacturer’s instructions. Interfacial adaptation is dependent on several factors: how well the operator manages to apply the restorative and the wetability and cleanness of the tooth structures. Calcium aluminate cements for dental purposes are described to have a hydroxyapatite forming potential which can create intimate adaptation with tooth structures (Lööf et al. 2003).

Parallel fractures contiguous to resin composite restorations were expected since they have been observed in earlier studies and are believed to be a result of the polymerisation shrinkage (van Dijken et al. 1998, Lindberg et al. 2000, Andersson-Wenckert et al. 1998 and 2002). However, the high frequency of perpendicular enamel fractures contiguous to all calcium aluminate cement restorations was unexpected (Figure 8). Some of these fractures were found in several consecutive sections of the restoration, while other fractures only were present at one specific section level. Perpendicular enamel fractures have not been found in other studies of interfacial adaptation of resin composites in vivo (van Dijken et al. 1998, Lindberg et al. 2000, Andersson-Wenckert et al. 1998 and 2002). An SEM pilot study, using the experimental version of Doxadent, revealed also a high frequency of perpendicular enamel fractures. A possible explanation could be a continuing expansion of the material. It is unclear whether such an expansion of the calcium aluminate cement may be due to an ongoing chemical reaction and/or water absorption. It is also unclear for how long the expansion proceeds and how it will influence the dimensional changes of the cement. A continuing expansion may have clinical consequences such as increased frequency of tooth and/or material fractures. Berglund et al. (2004) have shown that the dimensional change after 1 year in water storage is three times higher for Doxadent as for two resin composites. The
theoretical thought that a slight expansion of a material will compensate a possible gap-formation seems risky; the possibility of inducing stress to the tooth structures is obvious.

Clinical trials are considered the optimal way to validate not only the outcome of medicine but also dental materials. However, there are problems associated with clinical trials such as patient selection, compliance, and operator variability. These problems could adversely influence conclusions. A wide range of laboratory investigations serve as screening models since they are cheaper and less time consuming than clinical investigations and since they do not expose patients or animals to unnecessary risks. In the clinical study (paper V), an intra-individual study design was chosen since randomised control groups require large patient groups and are extremely demanding to conduct. Patients visiting the clinic for the yearly examination and in need of at least two similar sized class II cavities were asked to participate. All restorations were placed by one operator. Two evaluators (the operator and another dentist, both experienced users of the materials tested) performed evaluations (Ryge et al. 1981, van Dijken 1986, Pallesen and van Dijken 2000). Disagreement between the evaluators was resolved by consensus. The recall rate at the second year was 98.2%, and the cumulative failure frequency for the experimental calcium aluminate cement and resin composite was 43% and 3% respectively. The main reasons of failure were material fracture, cusp fracture, and erosion. In an ongoing study with Doxadent class II restorations, the reported cumulative failure frequency at the 2-year recall was higher than for the experimental version of the material (van Dijken, unpublished results). The clinical result reflects the marked difference in flexural strength found for the tested materials. It seems that flexural strength is correlated to clinical
performance whether the strength is mainly an expression of degree of porosity of the two versions of calcium aluminate cement or an expression of the inherent flexural strength in the materials. High annual failure rates of restorations in stress-bearing areas have been reported for other materials with poor mechanical properties such as the glass ionomer cement and glass cermet cement (Hickel et al. 1988, Smales et al. 1990, Krämer et al. 1994). As with calcium aluminate cement, most of these restorations failed due to material fracture. The resin composite chosen, Tetric Ceram, is a well known class II restorative and had been used at the clinic for about 3 years before the study started. The clinical durability of the resin composite found in the study is in agreement with that of another study (Schoch et al. 1999).

Cusp fracture of posterior teeth is a common phenomenon in dental practice (Bader et al. 2001, Fennis et al. 2002). However, there are few reports describing the occurrence of tooth fractures (Cavel et al. 1985, Bader et al. 1995, Heft et al. 2000). Cusp fractures constitute a significant dental health problem. Amalgam cavity preparation with large undercuts is believed to contribute to cusp fracture. Mastication has been reported as a major cause of cusp fracture. In restored teeth, cuspal deflection under a continuous occlusal loading of the weakened cusps will result in horizontal crack formation which may induce cusp fracture (Rees 1998, Panitvisai and Messer 1995). Clinical studies have shown that dentin bonding of resin composite restorations reduce this problem (Lindberg et al. 2003, Andersson-Wenckert et al. 2004). This was confirmed in the present study where one resin composite restoration and six calcium aluminate cement restorations showed cusp fracture.

The two investigated materials differed in the aspect of cavity preparation. Resin composite restorations are used with micro-mechanical retention, whereas the instructions for the calcium aluminate cement
dictate moderate amalgam preparations with rather parallel cavity walls. The adhesive property of resin composite allows a minimal preparation technique, which spares sound tooth substance and reduces the preparation trauma to the tooth. The term biocompatibility is often described as “the ability of a material to perform with an appropriate host response in a specific application”. Even if a material is considered as non-toxic, it cannot be considered as highly biocompatible if the application in itself requires destruction of sound human tissue.

One of the most influencing parameters on the clinical outcome of a dental restorative is its handling characteristics. It is not insignificant that the preparation of specimen is in accordance with the clinical instruction for the respective material and that the test reflects the clinical situation since the desire is to interpret the results from a clinical point of view and not from a strictly physical point of view. Therefore, it is justified to use specimens and methods reflecting the clinical reality. The calcium aluminate cement has been used according to the manufacturer’s instructions. Great effort was put into thoroughly familiarise with the material and making specimens and restorations in the best way. It seems unlikely that the material would be easier to use in a clinical situation than in the laboratory. The material has a crumbly character and reminds one of a somewhat too dry sand cake. To restore a convex tooth surface with Doxadent is very difficult. Despite the measured hardness of the material gross reduction of the calcium aluminate cement is observed during polishing with low pressure. At the time of the study, three shades of Doxadent were available, all very opaque, which is common for cements. This can be considered a dramatic step back from modern adhesive and aesthetic dentistry.
Over the years, several dental materials have been marketed that turned out in clinical follow-ups not to fulfil their marketing claims (Hickel et al. 1988, Smales et al. 1990, Krämer et al. 1994, Pilebro et al. 1999, van Dijken 2002). At the time of CE marking of Doxadent little was known about calcium aluminate cement functioning as a dental restorative. It can be debated whether a clinical trial would have been appropriate or not before CE marking. The Directive for medical devices mainly addresses general requirements such as design, construction, and manufacturing of the device but no direct clinical evaluation requirement is included. However, the Directive does state that the manufacturers have a clinical responsibility after obtaining the CE marking. It is prohibited to place medical devices on the market if the devices are claimed to have a performance they do not have. It is also prohibited to give a deceptive impression that application of the given medical device is certain to be successful and/or that no harmful effects result when the device is used as intended or used for a prolonged period. Failure of dental restoratives will probably seldom influence general health, but degeneration and fracture of material and tooth will compromise dental health. However, dental health is not included in the Directive requirements. The result of the clinical trial clearly indicates the necessity to include clinical evaluation of new restoratives in the CE marking.
Conclusions

The high failure rate of calcium aluminate cement restorations in the clinical trial clearly shows that the material is not suitable for class II restorations. Based also on the results of the three-point bending test, it is concluded that Doxadent does not possess sufficient strength for a class II restorative. The enamel fractures seen around the in vivo calcium aluminate cement restorations and the relatively high frequency of cusp fracture may indicate an undesirably high expansion of the material. It was also observed that the clinical handling of the calcium aluminate cement required a long learning period. The aesthetics of the calcium aluminate cements as a dental restorative is limited.
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