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Guide to the use of dental materials

Guide pour l'application des produits dentaires

Anleitung zur Anwendung von zahnärztlichen Werkstoffen

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Foreword

This Published Document has been prepared under the direction of the Dental Standards Committee. BSI Committee DNC/—, whose cooperating organizations are listed on the back cover of this Published Document, takes collective responsibility for its preparation under the authority of the Board of BSI. The Committee wishes to acknowledge the personal contributions of the following members of the Dental Materials Panel, to whom the preparation was entrusted: Dr J W McLean, OBE; Prof M Braden; Dr D Brown; Dr B E Causton; Dr E C Combe; Mr M W Miller; the late Dr T C Tranter; Prof N E Waters; Dr A D Wilson; Mr N J Bunton.

This guide has been published to assist dental practitioners in the choice and use of materials. That such a guide is necessary is evidenced by the fact that significant developments in materials are occurring continually. This guide is designed to show what properties are required of materials, for example, as laid down by British Standards, and also to give an indication of manipulative techniques to obtain optimum properties. Properties and applications of new materials, for which specifications do not yet exist, are also emphasized.

Compliance with a British Standard does not of itself confer immunity from legal obligations.

Guide to the use of dental materials

Section one. General

1. Scope

This Published Document is a guide to the choice and use of dental materials. A list of bibliographic references is given in appendix A.

2. References

The titles of the publications referred to in this Published Document are listed on page 40.

Section two. Cements

3. Introduction

3.1 Definition of cement. A cement is the product of an acid-base reaction formed by the interaction between a base (in powder form) and an acidic liquid. A salt hydrogel is formed that causes the cement to set and harden.

Cements include filling materials and luting agents, but exclude resin-based luting agents. A cement is supplied as two components that when mixed together form a hardenable paste.

3.2 Requirements and applications. A cement is required to have adequate working time, after preparation, and to set and harden rapidly so that it may be finished within a few minutes of preparation. It should be hydrolytically stable and preferably be resistant to acid attack. It should have sufficient strength for the application, i.e. for filling supported cavities in low stress situations, or for luting. An aesthetic filling cement has to be sufficiently translucent to match enamel in appearance. A luting cement has to have a low film thickness. Cements are used for a wide variety of applications: aesthetic filling (class III and class V including erosion lesions), temporary filling, cavity lining(s), luting, pulp capping and root canal filling(s).

3.3 Classification. Cements may be classified either on the basis of the powder or on the basis of the liquid. The classification given in table 1 is based on the powder, there being two basic types: zinc oxide cements and ion-leachable glass cements.

4. Zinc oxide based cements

4.1 Description. A zinc oxide based cement has deactivated zinc oxide as the principal constituent of the powder.

The liquid may be orthophosphoric acid or a poly (carboxylic acid), either in aqueous solution or in a non-aqueous organic liquid such as eugenol.

4.2 Zinc phosphate cement*

4.2.1 Composition. Zinc phosphate cement is supplied as a powder and a liquid. A range of composition is given in table 2.

Table 1. Classification of cements

Liquid	Powder containing zinc oxide	Ion-leachable glass powder
Orthophosphoric acid, aqueous solution	Zinc phosphate cement	Silicate cement
	Silico-phosphate cement	
Polymeric carboxylic acid, aqueous solution	Zinc polycarb-oxylate cement	Glass ionomer cement
Eugenol	Zinc oxide/eugenol (ZOE) cement	-
2-Ethoxybenzoic acid/eugenol	EBA cement	-
Other organic liquid	Non-eugenol cement	-

Table 2. Composition range of a typical zinc phosphate cement

Powder (% by mass)		Liquid (% by mass)	
Zinc oxide (ZnO)	89 % to 92 %	Total phosphate (as H ₃ PO ₄)	45 % to 63 %
Magnesium oxide (MgO)	3 % to 10 %	Aluminium	1 % to 3 %
Alumina (Al ₂ O ₃)	0 % to 7 %	Zinc	0 % to 10 %
Silica (SiO ₂)	0 % to 2 %	Water	to 100 %

*See also BS 3364.

The powder is formed by sintering the components at a temperature above 1100 °C to reduce reactivity, and milling. The liquid always contains aluminium to reduce reactivity and prevent excessive crystallinity developing in the matrix.

4.2.2 Manipulative technique. Zinc phosphate cement is characterized by being excessively exothermic, thus the mixing technique is designed to dissipate heat. Place the powder and liquid on a glass slab, preferably in known amounts. Avoid excessive exposure of the liquid (either in the bottle or on the slab) to the atmosphere, as the consequent gain or loss of water will adversely affect the cement's properties. Divide the powder into the following proportions: 1/16, 1/16, 1/8, 1/4, 1/4, 1/4. Incorporate these portions successively, in the order given, into the liquid allowing 10 s to 15 s for mixing in each portion (using an acid-resistant spatula) and mix the paste thoroughly. The total mixing time should be 90 s. If the components cannot be accurately proportioned, the paste, when used for filling, should be mixed as thickly as possible. For luting purposes, the paste has to be mixed to a fluid consistency.

4.2.3 Setting reaction. Zinc phosphate cement sets by the gelation of an amorphous zinc orthophosphate matrix formed as the pH of the paste increases. The set cement consists of zinc oxide particles embedded in this matrix. Subsequently the surface of the cement, on contact with moisture, levels off to form zinc phosphate ($Zn_3(PO_4)_2 \cdot 4H_2O$) crystallites. Setting is accelerated by increases in the powder/liquid ratio and temperature. Setting is also affected by the phosphoric acid concentration.

4.2.4 Properties. The required properties for zinc phosphate cement are given in table 3.

4.2.5 Application. The principal application of zinc phosphate cement is as a luting agent for bridges, crowns

and inlays. It is also sometimes used as a cavity lining and for the restoration of deciduous teeth.

4.3 Zinc polycarboxylate cement

4.3.1 Composition. Zinc polycarboxylate cement is supplied as a powder and a liquid which may be encapsulated for mechanical mixing. A range of composition is given in table 4.

The zinc oxide is similar to that used in zinc phosphate cement and is deactivated by sintering at a temperature above 1100 °C. The poly(acrylic acid) is prepared by aqueous polymerization.

4.3.2 Manipulative technique. Very little heat is generated when zinc polycarboxylate cement is mixed, hence the mixing technique differs from that for zinc phosphate cement. Place the powder and liquid on a glass slab, preferably in known amounts. Incorporate the powder into the liquid as quickly as possible and mix thoroughly. This technique gives maximum working time. Although the properties of this cement are less affected by gain or loss of water from the liquid than those of zinc phosphate cement, it is advisable to avoid excessive exposure of the liquid to the atmosphere. It is an adhesive cement and care has to be taken in cleaning substrates to which it is to be bonded.

4.3.3 Setting reaction. Setting results from gelation of a salt, as the pH of the paste increases, formed by the ion-binding of zinc ions to polycarboxylate chains following the extraction of zinc ions from the zinc oxide powder by the acidic liquid. This is an ionic cross-linking reaction, so that a transient elastomeric behaviour ('cobwebbing') is observed between the initial plastic state and the final rigid condition.

4.3.4 Properties. The properties given in table 5 have been proposed by ISO† as requirements for zinc polycarboxylate cement.

Table 3. Requirements for zinc phosphate cement

Class	Net setting time* at 37 °C		Compressive strength (24 h)	Film thickness		Water-leachable material content (24 h)	Arsenic content	Lead content
				μm				
	min.	max.	MPa	Type I	Type II	mg/kg	mg/kg	
	min.	max.	min.	max.	max.	max.	max.	max.
I (fast setting)	2.5	5.5	70	25	40	2.0†	2.0	100
II (normal setting)	4.5	8.5	70	25	40			

Table 4. Composition range of a typical zinc polycarboxylate cement

Powder (% by mass)	Liquid (% by mass)
Zinc oxide (ZnO) 88 % to 97 %	Poly (acrylic acid), molecular mass 23 000 to 49 000 32 % to 42 %
Magnesium oxide (MgO) 5 % to 10 %	Water to 100 %

*Measured from the completion of mixing.

†Milligrams of phosphorus pentoxide (P_2O_5) per gram of cement.

‡International Organization for Standardization.

Table 5. Proposed requirements for zinc polycarboxylate cement

Type	Manipulation time at 23 °C min	Net setting time* at 37 °C min	Compressive strength (24 h) MPa	Tensile strength (24 h) MPa	Film thickness μm	Water-leachable material content (24 h) $\mu\text{mho cm}^{-1} \text{ g}^{-1}$	Arsenic content mg/kg
	min.	max.	min.	min.	max.	max.	max.
1 (cementing material)	2.0	9.0	50	6	25	400	2.0
2 (filling material)	1.5	5.0	50	6		400	2.0

*Measured from the completion of mixing.

4.3.5 Applications. Zinc polycarboxylate cement is used principally as a luting agent, but also as a cavity lining, and for the direct cementation of orthodontic appliances.

4.4 Zinc oxide/eugenol cement†

4.4.1 General. Zinc oxide/eugenol (ZOE) cement differs from other zinc oxide cement in two respects: the type of zinc oxide used is quite different and the cement is non-aqueous.

4.4.2 Composition. The composition of zinc oxide/eugenol cement can be extremely complex and variable. The basic formulation is an 'active' zinc oxide powder (formed by the oxidation of zinc in oxygen or the low temperature (400 °C to 600 °C) thermal decomposition of salts) and eugenol liquid. However, additives, both accelerators and fillers, play an important role in the formulation of this cement. Common accelerators are zinc acetate, added (0.2 % *m/m*) to the powder, and acetic acid, added to the liquid. Polymer reinforcement may be added either to the powder (poly(methyl methacrylate) or hydrogenated rosin (abietic acid)) or to the liquid (polystyrene). Sometimes these materials are supplied in a two-paste form. One paste is prepared by adding a

plasticizer to the powder and the other by adding a neutral filler to the liquid. An important subclass is the so-called EBA cement, where the liquid is a 5:3 mixture by volume of 2-ethoxybenzoic acid and eugenol. The principal constituent of the powder is zinc oxide with a reinforcing filler of quartz or alumina, and some hydrogenated rosin (added to reduce sensitivity to moisture).

4.4.3 Manipulative technique. Zinc oxide/eugenol cement is extremely easy to mix, convenient to use and is characterized by a long working time. It is prepared by placing the powder and liquid on a glass slab and incorporating the powder in the liquid and mixing thoroughly.

4.4.4 Setting reaction. Setting occurs as the zinc oxide powder interacts with the eugenol to form a zinc eugenolate matrix containing unreacted zinc oxide as filler. The reaction rate is affected by the nature of the zinc oxide and the presence of acid, acid salt accelerator and moisture.

4.4.5 Properties. The required properties for the filling material (according to ISO 3106) and the luting agent (according to ISO 3107) are listed in tables 6 and 7 respectively.

Table 6. Requirements for zinc oxide/eugenol filling material

Consistency: disc diameter at 23 °C	Setting time at 37 °C		Compressive strength (24 h), min.	Water-leachable material content (24 h), min.
	Type 1 (fast setting)	Type 2 (normal setting)		
25 ± 1 mm	3 min to 5.5 min	4.5 min to 7 min	25 MPa	0.5 % (<i>m/m</i>)

Table 7. Requirements for zinc oxide/eugenol cementing material

Consistency: disc diameter at 23 °C	Setting time at 37 °C		Compressive strength (24 h), min.	Water-leachable material content (24 h), min.	Film thickness, max.
	Type 1 (fast setting)	Type 2 (normal setting)			
25 ± 1 mm	4 min to 7.5 min	6.5 min to 10 min	35 MPa	0.2 % (<i>m/m</i>)	25 μm

*See also ISO 3106 and ISO 3107.

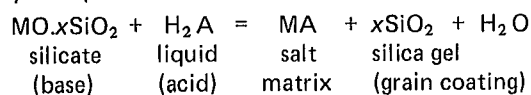
4.4.6 Applications. Zinc oxide/eugenol cement is principally used as a temporary filling and luting agent. Some experts advocate the use of reinforced ZOE and EBA cements for permanent luting. The cement is also used for root canal fillings and for pulp capping.

5. Ion-leachable glass based cements

5.1 Introduction

An ion-leachable glass based cement has an ion-leachable (aluminosilicate) glass as the principal constituent. The liquid may be orthophosphoric acid (aqueous) or an aqueous solution of a polymeric carboxylic acid.

The essential setting reaction of ion-leachable glass cement may be represented thus:



where H_2A represents the acid liquid and A the acid anion. M represents all the cations, M and A are taken to be notionally divalent for simplicity of representation although in fact they will be mono-, di-, tri- or polyvalent.

5.2 Silicate cement*

5.2.1 Composition. Silicate cement is supplied as a powder and a liquid, which may be encapsulated for mechanical mixing. The powder is a complex ion-leachable glass and may be regarded as a fluorine-containing aluminosilicate.

The powder is formed by fusing a mixture of silica and alumina with fluorides and phosphates. The melt is shock-cooled and ground to a fine powder. An excessive concentration of phosphoric acid in the liquid is detrimental to cement formation. The composition range of a typical silicate cement is given in table 8.

5.2.2 Manipulative technique. Place the powder and liquid on a glass slab, preferably in known amounts. Avoid excessive exposure of the liquid to the atmosphere as it will lose or gain water and so affect cement's properties. Divide the powder into the following proportions: 1/2, 1/4, 1/4. Incorporate these portions successively, in the order given, into the liquid allowing 15 s for mixing in each portion. Using an acid-resistant spatula, mix the whole paste for a further 15 s. The total mixing time is 1 min. If the materials cannot be accurately proportioned, the cement paste should be mixed as thickly as possible. Some materials are supplied in capsules and mixing is achieved by mechanical means. After insertion in the cavity, the cement should be allowed to set under a matrix strip. After removal of the matrix strip, the restoration should be treated with petroleum jelly before finishing, and with varnish after finishing.

5.2.3 Setting reaction. Silicate cement sets and hardens as the result of the precipitation of an amorphous aluminium orthophosphate gel as the pH of the system increases. Ions are extracted from the ion-leachable glass by the acidic liquid where they combine with anions to form salts. As the pH rises the aluminium phosphate gel is formed and the cement sets. Insoluble calcium fluoride and soluble sodium dihydrogen phosphate are formed in side reactions. The set cement consists of a gel-matrix in which are embedded particles of complex morphology, consisting of an unattached glass case surrounded by an ion-depleted outer layer that is essentially a silica gel.

5.2.4 Properties. The required properties for silicate cement are listed in table 9.

5.2.5 Applications. Silicate cement is used almost solely for the aesthetic restoration of anterior teeth.

Table 8. Composition range of a typical silicate cement

Powder (% by mass)		Liquid (% by mass)	
Silica (SiO_2)	31 % to 42 %	Total phosphate (as H_3PO_4)	46 % to 56 %
Alumina (Al_2O_3)	27 % to 29 %	Aluminium	0 % to 2 %
Calcium oxide (CaO)	7 % to 9 %	Zinc	4 % to 9 %
Sodium oxide (Na_2O)	7 % to 11 %	Water	to 100 %
Fluorine (F)	13 % to 22 %		
Phosphorus pentoxide (P_2O_5)	3 % to 5 %		
Zinc oxide (ZnO)	0 % to 3 %		

Table 9. Requirements for silicate cement

Net setting time† at 37 °C		Strength in compression (24 h)	Opacity $C_{0.70}$ value		Water-leachable material content (24 h)	Arsenic content	Lead content
min.	max.	min.	min.	max.	max.	max.	max.
2 min	5 min	170 MPa	0.35	0.55	9.0†	2 mg/kg	40 mg/kg

*See also BS 3365 : Part 1.

†Measured from the completion of mixing.

‡Milligrams of phosphorus pentoxide (P_2O_5) per gram of cement.

5.3 Silico-phosphate cement*

5.3.1 Composition. Silico-phosphate cement is a hybrid of the silicate and zinc phosphate cements. The powder is a silicate glass to which minor amounts of deactivated zinc oxide powder have been added. The liquid is the same as that used for silicate cement. There are two types, a filling material and a fine-grain version used for luting.

5.3.2 Manipulative technique. Manipulation is similar to that for silicate cement (see 5.2.2).

5.3.3 Setting reaction. The setting reaction may be regarded as a combination of that of the silicate and zinc phosphate cements.

5.3.4 Properties. The properties listed in table 10 have been proposed as requirements for silico-phosphate cement.

5.3.5 Applications. Silico-phosphate cement is used as a semi-translucent luting agent for porcelain crowns and inlays, for filling deciduous teeth and as a temporary filling material.

5.4 Glass ionomer cement†

5.4.1 Composition. Glass ionomer cement is supplied as a powder and liquid, which may be encapsulated for mechanical mixing. The powder is a complex ion-leachable

glass and is a type of fluorine-containing aluminosilicate glass. It is more basic than that used in silicate cement.

The glass is prepared by fusing the constituents at a temperature between 1150 °C and 1300 °C and shock cooling the melt. The powder is ground to a maximum particle size of 50 µm for the filling material and to a maximum particle size of 20 µm for the luting agent. The polymer is prepared by aqueous polymerization. The composition of a typical glass ionomer cement is given in table 11.

5.4.2 Manipulative technique. Glass ionomer cement is the least exothermic of all the aqueous based dental cements and rapid mixing is necessary. Place the powder and liquid, preferably in known amounts, on a chilled glass slab. Incorporate two-thirds of the powder into the liquid and mix within 10 s. Sweep in the remainder of the powder and mix thoroughly. The total mixing time should be between 45 s and 1 min. If the components cannot be accurately proportioned, filling material should be mixed as thickly as possible. Luting versions should be mixed to a fluid state. After placement, the cement should be allowed to set under a matrix strip and then coated with petroleum

Table 10. Proposed requirements for silico-phosphate cement*

Type	Consistency: disc diameter at 23 °C mm	Net setting time† at 37 °C min	Compressive strength (24 h)	Film thickness	Water-leachable material content (24 h)	Opacity		Arsenic content	Lead content
			MPa	µm		C _{0.70} value		mg/kg	mg/kg
			min.	max.		max.	min.	max.	max.
I (cementing medium)	28 ± 1	4.0 to 8.0	140	25	9.0†	0.35	0.90	2.0	100
II (restorative material)	23 ± 1	2.0 to 5.0	170		6.0†			2.0	100

*The values given in the table are taken from the draft revision of ISO 3824 'Dental silicophosphate cement (hand-mixed)', which will be implemented in the second revision of BS 3365 : Part 2. ISO 3824 also includes type III, dual-purpose cement.

†Measured from completion of mixing.

‡Milligrams of phosphorus pentoxide (P₂O₅) per gram of cement.

Table 11. Composition of a typical glass ionomer cement

Powder (% by mass)		Liquid (% by mass)	
Silica (SiO ₂)	30.1 %	Copolymer of acrylic and itaconic acids (molecular mass 10 000)	47.5 %
Alumina (Al ₂ O ₃)	5.5 %		
Calcium oxide (CaO)	24.8 %		
Sodium oxide (Na ₂ O)	2.7 %		
Phosphorus pentoxide (P ₂ O ₅)	5.8 %	Tartaric acid	5.0 %
Fluorine (F)	20.3 %	Water	to 100.0 %

*See also BS 3365 : Part 2, which has been proposed for revision.

†See also BS 6039.

jelly or wax. The restoration should be preferably finished immediately after set using carving techniques. The restoration should then be protected by varnish or wax.

NOTE. It is essential that early moisture contamination of this cement be avoided.

5.4.3 Setting reaction. Glass ionomer cement sets in two stages. Initial gelation results from the formation of an insoluble calcium polysalt and the cement can be carved at this stage. Later the aluminium polysalt is formed, which contributes to the final hardening and rigidity of the cement. Essentially the process is one in which polyanion chains are cross-linked by metal ions, extracted from the surface of the glass particles, possibly as fluoride complexes, by the acidic liquid. Setting and hardening is accompanied by an increase in pH. The reaction is a complex one and is not fully understood. It is accelerated by increases in the powder/liquid ratio and temperature, and by the presence of tartaric acid. The set cement consists of an aluminium and calcium polysalt matrix in which are embedded particles of complex morphology having a case of unattached glass sheathed by an outer layer of silica gel.

5.4.4 Properties. The required properties for glass ionomer cement are listed in table 12.

5.4.5 Applications. Glass ionomer cement is supplied in two versions: a filling material and, in fine-grain form, a luting agent. The filling material is used for the aesthetic restoration of anterior teeth (class III and class V including erosion lesions) and for the restoration of deciduous teeth. The luting agent is used as a cavity liner. Both filling and luting versions may be used as a pit-and-fissure sealant.

Section three. Resins

6. Fissure sealants

6.1 Introduction

6.1.1 General. Fissures form ideal sites for the accumulation of bacterial plaque, which are not accessible to cleaning. In attempts to prevent this and thereby reduce the incidence the fissural caries, a wide range of materials have been tried for occluding the fissures, with varying degrees of success. Present day materials include diacrylates (in conjunction with acid etch procedures) and adhesive dental cement.

6.1.2 Diacrylates. Current fissure sealant materials in this group are based on the reaction product of bisphenol A and glycidyl methacrylate (bis-GMA resin). The parent resin is too viscous for direct use and the viscosity is normally reduced by addition of other materials such as methyl methacrylate monomer.

Two methods of polymerization are available:

(a) *chemical*: using conventional benzoyl peroxide/*NN*-dimethyl-*p*-toluidine system;

(b) *photochemical*: to activate the base resin, one drop of the initiator (2 % benzoin methyl ether) is added to the base resin (1 ml) before use. After activation, the viscosity increases slowly, so a fresh mix should be prepared each day. On irradiation with ultraviolet (u.v.) light (365 nm), free radicals are produced which effect polymerization. This system entails the disadvantage of the cost of a suitable u.v. source, but the operator has control of the setting time.

Whilst the precise physical parameters that determine the effectiveness of a resin as a fissure sealant have not yet been characterized with any precision, there is no doubt that the bis-GMA formulations have so far proved the most satisfactory sealants, although this should not be taken to imply that this will necessarily be so in the future.

6.1.3 Filled resins. It was considered initially that a low viscosity monomer was essential to achieve satisfactory adhesion, and that difficulties might be expected to arise with highly-filled resin. This has not, however, proved to be the case in practice, the adhesion achieved being just as good as with unfilled resin. Filled resins have higher abrasion resistance and exhibit decreased creep. Abrasion resistance makes diagnosis of the state of the sealant easier.

6.1.4 Adhesive dental cements. The recently introduced zinc polycarboxylate and glass ionomer cements have both been tried as fissure sealants.

Polycarboxylate cement is adhesive to enamel and to a lesser extent to dentine. This adhesion is not improved by etching. Retention seems to be poor.

Clinical studies suggest that glass ionomer cement presents a viable alternative to resin systems for fissure sealing.

Its retention is variable but this does not detract from their cariostatic effect which is achieved not only by a physical barrier as with resin, but also by a continuing fluoride leach.

Table 12. Requirements for glass ionomer cement

Type	Consistency: disc diameter at 23 °C mm	Setting time at 37 °C min	Working time min	Compressive strength (24 h) MPa	Diametral tensile strength MPa	Film thick- ness µm	Water-leachable material content (24 h) % by mass	Opacity		Arsenic content mg/kg (p.p.m.)	Lead content mg/kg (p.p.m.)
		max.	max.	min.	min.	max.	max.	min.	max.	max.	max.
1 (luting agent)	30 ± 2	7.5	3.0	65	6.0	25	1.0	-	-	2.0	40
2 (restorative material)	30 ± 2	5.0	2.75	125	10.0	-	0.7	0.35	0.90	2.0	40

A technique of slightly widening the fissures has been suggested to aid retention of glass ionomer cement because it will not flow into very small crevices as well as resin sealant. Glass ionomer cement has the advantage of leachable fluoride ions and good biocompatibility, but it is adversely affected by water contamination during setting, and for a short period after, and protection with varnish is necessary.

6.2 Acid-etch procedures

6.2.1 Theory

6.2.1.1 Surface porosity. The etchant effects differential dissolution of the prism structure. Generally, mineral acids attack the prism cores preferentially, whilst in the case of chelating agents it is the margins of the prisms that dissolve most rapidly. The line of demarcation between these two modes of attack is not, however, as sharp as was originally supposed. Orthophosphoric acid is the most commonly used etchant, and the depth of etch decreases continuously with increasing concentration, from 14 μm at 20 % (V/V) to 2 μm at 70 % (V/V) for a treatment of 1 min. Concentrations of 30 % (V/V) to 50 % (V/V) are, however, most frequently used in practice. This acid is often buffered with 7 % zinc oxide by mass but the precise merit of this addition is in some doubt. The overall effect of the etching process is surface porosity, with the total depth of enamel affected not exceeding 30 μm .

6.2.1.2 Tag formation. The liquid monomer penetrates the surface porosity so that after polymerization, 'tags' of solid resin are formed penetrating the enamel surface. The length of these tags is normally up to 50 μm , but in some cases lengths of 100 μm have been reported. It is not easy, however, to reconcile these high values with the figure of 30 μm for the maximum depth of enamel affected. Further work is required to resolve this apparent anomaly.

6.2.1.3 Enamel surface with high free energy. The other effect of the etchant is to remove the surface organic layer, or pellicle, together with the outer layer of enamel that has become saturated with ions taken up from the saliva and therefore of low reactivity. The net result is the production of a very reactive surface, i.e. one with a very high free energy. This condition is essential if the liquid monomer is to wet the enamel surface properly.

6.2.1.4 Increase in surface area. The etching action also produces a large increase in the surface area of the enamel, which improves the prospects of good bonding between the resin and enamel.

6.3 Manipulative technique

6.3.1 Isolation of teeth. Ideally a rubber dam should be used, but this is not always practicable. Isolate the teeth to be sealed in one quadrant of the mouth with cotton wool rolls. If mandibular teeth are involved, suitably position a flanged ejector to prevent interference from the tongue.

6.3.2 Etching. Wash the tooth surface to remove loose surface debris, dry and apply the etchant for the time recommended by the manufacturer. A sable-hair brush, gauge 00, is ideal for this purpose and preferable to a cotton wool pledget, which increases the likelihood of the acid solution making contact with the gingival margin. Keep the etchant continuously agitated during treatment.

6.3.3 Rinse and dry etched surface. A washing time of 15 s using a stream of water alone is recommended. This is then followed by drying with oil-free air. Position the exit tube of a high capacity aspirator close to the occlusal surface of the tooth so that the washing water is removed immediately

and not allowed to accumulate in the oral cavity. Dry the surface carefully with compressed air. It should exhibit a 'frosted white' appearance, but if not, repeat the etching procedure.

6.3.4 Application of sealant. With external energy polymerized sealant, after adding initiator allow to stand for 5 min to allow air bubbles to escape. Apply the sealant monomer to the dry etched enamel surface with the minimum of delay, using another sable-hair brush. Special precautions should be taken to eliminate any possibility of confusing this brush with the one used for applying the etching solution. It is not sufficient to place the monomer on the enamel surface and rely on penetration of the pores occurring spontaneously; brush the sealant well into the surface. With a chemically-polymerized resin, the use of a brush may be complicated by the resin setting in the bristles, thereby rendering the brush useless. It will therefore need to be placed immediately after use in a suitable solvent (e.g. trichloroethane or suitable chlorinated solvent).

6.3.5 Hardening of sealant. If external energy polymerization is being used, place the tip of the light guide about 1 mm above the occlusal surface and irradiate for the time recommended by the manufacturer. Traverse the light guide across the occlusal surface to compensate for the non-uniform intensity distribution in the light source. Most chemically-polymerized sealants require approximately 60 s to 90 s to harden.

6.3.6 Checking the 'hardened' sealant. Finally, check the hardness of the sealant using a blunt explorer. Avoid excessive pressure as used when attempting to diagnose caries, as this can seriously damage films that have not hardened completely and impair their anti-cariogenic effect. The surface of the sealant should be completely smooth and the tip of the explorer should slide easily over it. Repair any detected defects in the surface by the addition of more sealant. If the hardness is not satisfactory, establish the reason. If an adequate degree of polymerization has not been achieved for some reason, the resultant polymer is unlikely to be effective as a sealant. In the diagnosis of the degree of hardness, make due allowance for any 'air-inhibited' sticky layer on the surface of the sealant. This layer can be easily removed with a damp cotton wool pledget.

6.4 Aspects of technique requiring special attention.

Meticulous attention to detail is essential if optimum results are to be obtained.

6.4.1 Differences in etching rates of teeth. Since some teeth show a greater resistance to etching than others, it is in all cases important that the effectiveness of the etching procedure be monitored visually; a 'frosted white' appearance of the enamel should be evident.

6.4.2 Contamination of the etched surface. Contamination of the dry freshly etched and highly reactive enamel surface has to be carefully avoided. Contamination can occur by saliva, humidity and water and/or oil in the compressed air supply.

6.4.3 Agitation of etching solution. Efficient agitation of the etching solution is essential if adequate and uniform etching of the enamel is to be achieved.

6.4.4 Changes in intensity of external energy source. If external energy polymerization is to be carried out, the efficiency of the light source should be regularly checked by polymerising a thin film of sealant on a glass microscope slide and checking the hardness of the resultant

polymer. The end of the light guide should never be contaminated with sealant as this reduces the intensity of u.v. radiation.

6.4.5 *Is an initial prophylaxis necessary?* It is generally considered that if the etchant is correctly agitated then the surface pellicle is removed and a perfectly satisfactory etch is obtained without any prophylaxis.

6.5 Possible problems associated with the use of sealant

6.5.1 *Microleakage.* It is likely that marginal leakage of sealant will be on an extremely small scale.

6.5.2 *Progression of caries under sealant.* There is as yet no evidence to suggest that caries progression can occur under a sealant that has been correctly applied, and so has been in position for a few years.

6.5.3 *Increased caries susceptibility after premature sealant loss.* There is no evidence to suggest that any increase in caries susceptibility occurs as a result of the etching process.

6.5.4 *Dangers associated with use of u.v. radiation.* There are special risks associated with certain medical conditions that are hypersensitive to u.v. radiation. Also, some patients may be using photosensitizing drugs.

6.6 Effectiveness of sealant technique. From the considerable amount of data in the literature, both on sealant retention and on percentage reduction in caries, it can be concluded that it is important for sealant placement to be carried out as soon as possible after eruption of teeth (see 6.7.1). In the case of first and second molars, it is often difficult to avoid water contamination. In clinical trials, lower success rates may be achieved with younger (six- to eight-year-old) patients, and considerable variation between operators has been recorded.

6.7 General

6.7.1 *Choice of patients.* Some care is required, since if all molars and premolars are treated, considerable clinical time will be wasted in sealing fissures that would not have decayed. Dietary habits do not appear to have any effect on the life of sealant.

In selecting teeth for sealing, the form of the fissures should receive due consideration. Teeth with well-coalesced fissures or shallow occlusal anatomy are probably non-susceptible and not worth sealing. Sealing should be effected as soon as possible after eruption since the teeth are then in their most vulnerable state owing to the presence of a hypocalcified layer at the enamel surface.

Sealing in of very early caries is admissible. If this were not so, the technique would not be viable, since it is not possible to clinically detect the earliest stages of carious attack. If there is any suspicion of cavitation, however, minimal restorations are called for.

6.7.2 *Complementary role of fluoride and fissure sealants.* It is frequently stated that the fissure sealant technique is of little value because of the problem of smooth surface caries that arises at a slightly greater age. This is an unrealistic approach since the same criticism could be levelled against the fluoridation approach. Any treatment that even delays the need for operative procedures is worthy of careful consideration. It also has to be recognized that fluoride is of limited efficacy where fissure caries is concerned, as the caries reduction is probably only about 15%. In view of the high incidence of fissure caries, the use of sealants to prevent decay of the occlusal surfaces would seem to be fully justified. Subsequent interproximal caries,

if it should develop, can in any case be dealt with by the modified class II approach. A combination of fluoride with fissure sealants is therefore essential if the maximum preventive effect is to be achieved. The two techniques should be regarded as essentially complementary in character.

6.7.3 *Regular servicing of sealants.* Regular servicing of sealants is essential, as it is for restorations in general. If sealants are incorrectly applied they are lost very rapidly. Those surviving the first six months can be expected to have a long life.

Loss of sealant due to attrition is not, by itself, a reason for applying more sealant. Such loss is inevitable. The crucial question is whether any parts of the fissure network are not properly occluded. Any such deficiencies should be repaired. If any doubt exists whether the fissures are completely occluded, it is probably wise to add a little more sealant.

6.7.4 *Extension for prevention.* The advent of adhesive resin necessitates a drastic re-appraisal of the technique of fissure sealing. Sound enamel and dentine should no longer be removed unnecessarily.

7. Varnishes and liners

7.1 Varnishes

7.1.1 *Composition.* Varnishes contain polystyrene or a resin, such as copal, and a volatile solvent such as chloroform, ether or acetone. On application of this solution, the solvent evaporates leaving a thin film of resin.

7.1.2 *Applications.* Varnishes are used:

- (a) for the protection of silicate and glass ionomer cement restorations;
- (b) to seal exposed dentinal tubules to protect the pulp from the effect of irritant components of filling material and bacteria occupying the crevice between the material and the cavity wall;
- (c) to reduce marginal leakage, particularly around amalgam restorations. The importance of cavity sealants is emphasized with non-gamma-2 amalgam restorations, as micro-leakage is thought to increase owing to the absence of corrosion products.

7.2 Liners

7.2.1 *Composition.* The types of liners available are a suspension of calcium hydroxide in a resin solution; and two-paste systems containing calcium hydroxide.

7.2.2 *Applications.* Liners are used to neutralize the effects of acidic components of cement; and as pulp capping material, where they can stimulate the formation of secondary dentine.

8. Resin based filling materials*

8.1 Definition and classification. A resin based filling material may or may not contain fillers. BS 5199 defines a composite filling material as one containing 50 % or more by mass of inorganic filler. This definition excludes material with organic fillers even though in the general sense they have a composite structure. A filling material with organic filler particles may contain a micronized filler within the organic filler particles; should the mass of such an inorganic filler exceed 50 % of the total mass of the set filling, this material would be considered as a composite according to BS 5199.

8.2 Manipulative technique. See table 13.

*See also BS 5199.

Table 13. Manipulative technique for composite filling materials

Technique	Comments
Avoid contamination of the preparation by saliva or blood.	The incorporation of saliva or blood results in a weakening of the filling and possibly a poor colour match.
Use a polyester matrix band or crown form wherever possible.	The matrix band reduces oxygen diffusion into the cured resin, and a higher degree of polymerization is therefore achieved. Conventional composites are difficult to polish using stones or other abrasives. However, casting the composite against a polyester strip produces a glass-smooth surface. However, this surface consists mostly of unfilled resin and is rapidly abraded to leave the characteristic rough composite surface.
Avoid incorporating air during mixing.	Oxygen inhibits the curing of the resin. Trapped air produces voids in the cured resin and reduces its strength.
The composite should be introduced into the cavity without delay after the recommended mixing time.	Once mixed, the viscosity of the composite rises rapidly, and delay in packing increases the risk of marginal leakage.
Apply pressure to the composite during curing.	The pressure compensates for most of the early setting shrinkage. This aids marginal adaption and reduces marginal leakage.
Do not overfill the cavity; keep finishing procedures to a minimum.	Because composites are difficult to polish, the area to be finished has to be minimal if discolouration of the surface is to be avoided.
Carry out delayed finishing (24 h to 28 h) to allow absorption of water by the restoration.	This relieves the tension on the enamel when an acid etch has been used, thereby reducing the possibility of fracture of the enamel margins during finishing procedures.
Avoid the use of metal burs and instruments.	The filler abrades the metal causing a grey discolouration.
u.v. and visible light cured composites Do not try to cure too thick a layer at any one time.	The light will fail to cure the lower part of the layer. This will result in a band of low molecular mass resin and a weakening of the restoration at that point.
Give the resin at least the recommended period of exposure to the light.	Failure to do so will result in uncured layers deep in the restoration.

8.3 Composition and method of presentation. The composition of typical types of resin-based filling materials is given in table 14.

8.4 Setting reaction

8.4.1 Unfilled or filled poly(methyl methacrylate) based filling materials. An unfilled or filled poly(methyl methacrylate) based filling material sets by an addition polymerization of methyl methacrylate. Initiation is by radicals produced when benzoyl peroxide reacts with an activator. The activator can be either an aromatic tertiary amine or sulphonic acid. The reaction progresses by addition of monomer and radical, resulting in a chain-like molecule. The chain only ceases to grow when either there is no more monomer or the radical is lost from the chain (see 9.3.3).

8.4.2 Composite filling materials. A composite filling material also sets by a radical-addition reaction. However, composites based on bis-GMA or urethane dimethacrylates form a cross-linked matrix rather than a chain-like molecule. The reaction can be initiated in the same way as described in 8.4.1, or stable radicals can be produced using u.v. light on benzoin methyl ether or by visible light on α -diketones. The propagation and termination of these setting reactions are as described in 8.4.1.
NOTE. Heat and sunlight can initiate addition reactions. Hence, store composite in cool places and replace lids immediately after use. Oxygen is a very good terminator of radical addition reactions. Hence, avoid thin, air-exposed layers.

Table 14. Composition of typical types of resin based filling materials

Composition	Comments
Direct filling acrylic Powder Poly(methyl methacrylate) (molecular mass 10^6) Benzoyl peroxide ($\leq 1\%$) Pigment (oxides) Liquid Methyl methacrylate (100 g) Hydroquinone (0.0025 g) Dimethyl- <i>p</i> -toluidine (2.5 g)	Poly(methyl methacrylate) beads, particle size 5 μm to 70 μm . Benzoyl peroxide is catalyst for 1 % polymerization of methyl methacrylate. Hydroquinone stabilizes methyl methacrylate monomer. Dimethyl- <i>p</i> -toluidine activates benzoyl peroxide at room temperature.
Composite filling material, two-paste Catalyst paste bis-GMA (100 g) Benzoyl peroxide (1 g) Ground quartz or aluminosilicate glass (surface treated with a vinyl silane) (75 g) Ethylene dimethacrylate (or other dimethacrylate) (25 g) Activator or 'universal' paste Composition as for catalyst paste except that the benzoyl peroxide is replaced by less than 2.5 g of dimethyl- <i>p</i> -toluidine, or now more commonly a substituted equivalent aromatic amine.	bis-GMA is an aromatic dimethacrylate resin. Benzoyl peroxide is a catalyst for the curing reaction. Surface treatment of ground quartz filler improves strength and durability of composite. The dimethacrylate is a diluent for bis-GMA to reduce the viscosity of the paste and ease mixing. Some manufacturers replace all or some of the bis-GMA with a urethane dimethacrylate. Activator for benzoyl peroxide. The substituted tertiary amines have better colour stability.
Composite filling material, paste/liquid Composition as for two-paste material except that all the filler is in the 'universal' paste.	Composite restorative material is also supplied in encapsulated form (composition as for two-paste material).
Composite filling material, light-cured (u.v. or visible) u.v. cured paste bis-GMA (100 g) Quartz filler (75 g) Benzoin methyl ether (1 g) Ethylene dimethacrylate Light-cured paste Aluminosilicate glass Urethane dimethacrylate α -diketone	The paste is cured in situ using a u.v. gun. The benzoin methyl ether is a u.v. polymerization activator. The α -diketone is a light-activated polymerization initiator.

8.5 Physical properties

8.5.1 Unfilled poly(methyl methacrylate) based filling materials. Unfilled poly(methyl methacrylate) based filling materials are inferior in many respects to conventional composite filling materials, particularly in respect of their:

- (a) higher exotherm on setting;
- (b) higher coefficient of thermal expansion;
- (c) higher water absorption;
- (d) greater setting shrinkage;
- (e) lower abrasion resistance;
- (f) poorer colour stability;
- (g) lower hardness;
- (h) lower compressive strength;

- (i) greater susceptibility to marginal leakage;
- (j) greater irritation to the pulp;
- (k) greater susceptibility to creep.

One advantage of these materials is that they can be polished.

8.5.2 Composite filling materials. A composite filling material complying with BS 5199 has the following properties:

- (a) a minimum working time of 1 min;
- (b) a setting time of not more than 8 min;
- (c) a transverse strength greater than 50 MN/m²;
- (d) colour stability;
- (e) non-toxic to pulpal or oral tissues.