A Comparative Study of Some Dental Cements Used in Orthodontics

P. L. Sadowsky, B.D.S., M.Dent.
D. H. Retief, M.Sc., B.D.S., Ph.D.

The problem of enamel decalcification occurring beneath orthodontic bands during treatment has always been one of great concern to orthodontists. This decalcification has been attributed to many factors among which are seal breakdown, inadequate structural and bonding strength, and the solubility of currently used dental cements in oral fluids. No doubt poor oral hygiene is also an important factor associated with this decalcification.

The dental literature contains numerous reports of the higher incidence of caries in patients undergoing orthodontic treatment. In 1937 Noyes[1] concluded that increased caries formation in these patients was related to poor oral hygiene. Gibbon[2] pointed out that much unfavourable criticism was directed at orthodontic services by both the laity and the profession and that many parents believed that excessive tooth destruction was the penalty paid for having "teeth straightened." Gibbon, however, concurred with Noyes when he stated that orthodontic treatment could be an important contributory factor in the increased caries incidence depending upon the extent to which it limited the maintenance of hygienic conditions in the mouth.

Noyes also incriminated the failure of the cement to maintain its seal between the enamel and the orthodontic band. Other authors[3,4,5] were in agreement with the earlier observations of Noyes.

From the Department of Orthodontics and Dental Research unit of the University of the Witwatersrand, Johannesburg, South Africa.

Lefkowitz and Bodecker[6] reported that it was the dental cement itself, through its liquid component which contained a high concentration of phosphoric acid, that was primarily responsible for decalcification occurring beneath orthodontic bands. Their comments referred to the phosphate cements, and later workers[7,8,9] have reaffirmed these views. Other researchers[10,11] held contrary views, however, and have reported no ill effects on enamel due to the actions of the cements themselves.

Gursin[12] studied the effect on the solubility of the enamel under the cement by adding stannous fluoride to the dental cement. By including stannous fluoride in the cement liquid a beneficial effect was imparted by reducing acid solubility of the enamel. Wei and Sierck[13] also advocated the addition of stannous fluoride to the cements used for orthodontic band placement in an attempt to impart anticariogenic properties.

Shannon[14] stated that, although there were controversial points of view, it appeared that orthodontic treatment brought about a significant increase in caries risk. He considered it not surprising that the phosphoric acid used in some cement liquids would decalcify the tooth surface. He believed that very thin mixes of cements would allow this demineralisation to take place prior to setting of the cement, but that evidence was lacking that a properly prepared mix of cement was in itself harmful to the tooth surface.

In view of the concern regarding decalcification occurring beneath orthodontic bands, many new dental cements have been developed. These have been made commercially available to the profession to lessen the undesirable decalcification which plagues orthodontic practice. These cements include composite cements which require etching of the enamel surfaces prior to band cementation, carboxylate cements which chelate with calcium of the enamel, cements containing fluoride as one of their components, and "improved" zinc phosphate cements.

The object of this investigation was to compare the tensile bond strengths of four currently used orthodontic cements both to human enamel surfaces and to stainless steel orthodontic band material. The cements selected were representative of each of the four groups mentioned above. The marginal leakage at the enamel-cement interfaces was evaluated. In addition, the scanning electron microscope was used to examine normal human enamel surfaces, teeth exhibiting enamel decalcification, enamel surfaces etched with the "conditioner" provided with one of the cements, and the fitting surface of stainless steel orthodontic band material. Furthermore, since the solubility and disintegration of the cements currently used for orthodontic band cementation may play an important role in the ingress of bacteria, plaque, and debris beneath orthodontic bands and hence influence enamel decalcification, solubility and disintegration tests were undertaken.

Materials and Methods

Four cements of varying composition that are currently available for orthodontic band cementation were selected. These were 1) a composite cement,* 2) a carboxylate cement,† 3) a silicophosphate cement,‡ and 4) a zinc phosphate cement.§

Tensile bond strength to human enamel

The tensile disruptive force required to break an experimental bond was used as a measure of the bond strength of the cement to enamel. This tensile test was selected because a subcommittee of the Dental Materials group on standard test methods for direct filling materials of the International Association for Dental Research recommended in 1967 that a tensile test should be used in preference to shear or punch-out type tests. The reasoning was that a tensile test was less dependent upon the surface morphology and the visco-elastic properties of the materials being tested. It is generally agreed, however, that tensile-loading tests should be used to express the tensile adhesive-enamel bond strength rather than as a test of adhesion.

The preparation of the experimental test specimens and subsequent evaluation of the tensile bond strengths were carried out according to a method developed by Hanke[15] and modified by Phillips *et al.*[16]

Sixty freshly extracted human maxillary central incisor teeth were stored in a refrigerator at —4°C until required. The labial surfaces of these teeth were ground flat on a polishing machine, care being taken not to expose dentine. The roots of the teeth were cut off and the crowns mounted in a mould with self-curing acrylic resin to expose the flattened surfaces. Final polishing of the exposed enamel was done on wet 600 grit silicon carbide paper on the polishing machine immediately prior to preparing the experimental bonds.

---

*"Once," Lee Pharmaceuticals.

†"Durelon," Espe Fabrik Pharmazeutischer Präparate GmbH.

‡"Fluoro-thin," S. S. White Dental Products.

§"Zinc Cement," Lee Smith Company.

The sixty mounted teeth were divided into four groups of fifteen each. The exposed surfaces of the teeth to be used for testing the composite cement were etched for two minutes with the "conditioner" provided with this material. The conditioner consisted of a 50 percent phosphoric acid solution. The etched surfaces were thoroughly washed to remove residual acid and dried. Acid etching of the polished enamel surfaces was not carried out prior to the preparation of the experimental bonds with the other three cements. Split-ring polyether rubber matrices with inside diameter of 6 mm were placed on the exposed enamel surfaces and a close fitting metal band slipped around the matrix to maintain the contour and hence the inside diameter of the ring. Each of the four cements was mixed separately according to the manufacturers' instructions, the space in the matrix filled with a portion of the cement and the remainder placed in an undercut recess in a brass ball bearing. The balls were carefully aligned on the matrix and the cements allowed to set for 15 minutes at room temperature and then stored in water at 37°C for 24 hours prior to testing.

The prepared specimens were tested in an Instron Table Model 1026 tensile testing machine to determine tensile bond strengths. These specimens were mounted in a test jig consisting of a series of joints assembled to produce a universal joint. The test jig was designed to allow proper alignment of specimens in the testing machine and to eliminate as nearly as possible all forces other than tensile during the application of the load. A loading speed of 0.05 cm min^{-1} was applied and the force required to break an experimental bond automatically recorded. The enamel-cement bond strengths were calculated and expressed in megapascals (MPa).

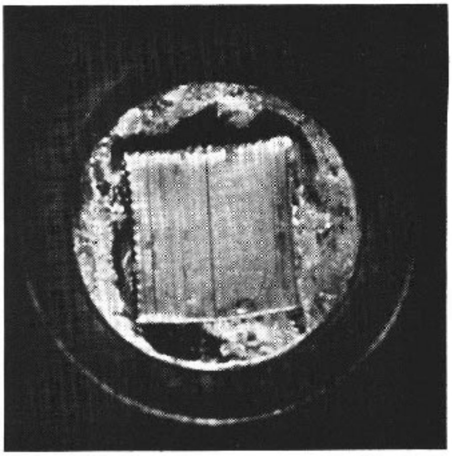

Fig. 1 Specially prepared brass studs with band material soldered in place.

The results were subjected to statistical analysis.

Tensile bond strength to stainless steel orthodontic band material

To assess the tensile bond strengths of the cements to band material, special brass studs were prepared on a lathe and squares of band material soldered to the studs so that the band surface normally apposed to the tooth surface was exposed (Fig. 1). The solder used had a melting point of 220°C. The design of the brass studs enabled suitable mounting in the Instron tensile testing machine.

Preparation of the test specimens with the four cements was done as described for the enamel-cement bond strength tests. No prior etching was carried out for the testing of the composite cement, but all the band metal surfaces were cleaned with ether-alcohol prior to assembly of the experimental specimens. Ten specimens were tested for each cement.

The results were subjected to statistical analysis.

Marginal leakage

Marginal leakage at the enamel-cement interface was studied by means of a fluorescent dye. A small amount of composite cement was placed on etched enamel surfaces of extracted human teeth (preserved as described previously) and allowed to set for one hour at room temperature. The other three cements were similarly placed on unetched enamel surfaces. The entire surface of all the teeth, with the exception of a small area surrounding the cement, was coated with several applications of nail varnish. This procedure excluded the subsequent penetration of the dye at any site of entry other than the enamel-cement interfaces. Each of the four groups of teeth was placed in a solution containing one tablet of Blak-Ray red fluorescent dye in 25 cm^3 water at 37°C for one week. This dye was used with success by Holliger[17] to demonstrate marginal leakage at the interfaces between various restorative materials and tooth structure.

Very thin ground sections of the teeth with the composite cement were prepared through the cement and the underlying tooth structure. In the case of the teeth with the carboxylate and the silicophosphate cements, it was found necessary to first embed these teeth in a high clarity casting resin prior to ground section preparation. Failure to embed these specimens resulted in the cement breaking away from the enamel surfaces when attempting to prepare thin ground sections. No dye penetration studies were possible with the zinc phosphate cement since the cement became dislodged from the enamel surfaces during incubation.

The prepared sections were examined by transmitted ultraviolet light irradiation.

Scanning electron microscope studies

Selected specimens were examined, namely, the enamel surfaces of freshly extracted human teeth, areas of decalcified enamel on extracted human teeth, the enamel surfaces of extracted human teeth which were etched for two minutes with the composite cement "conditioner," and the fitting surfaces of stainless steel orthodontic band material. The specimens were mounted on aluminum stubs and coated with silver in a high vacuum evaporator. They were then viewed in a Cambridge S4 Stereoscan scanning electron microscope operated at 15 kV with the beam-specimen angle varied to obtain the best surface projection.

Solubility and disintegration

Each of the four cements was tested for solubility and disintegration. The test procedure employed was that described by the American Dental Association for testing solubility and disintegration of dental zinc phosphate cement (A.D.A. specification No. 8) and for dental silicate cement (A.D.A. specification No. 9).[18] These two testing procedures are identical and, since there are at the present time no standardised tests for testing solubility and disintegration of composite or carboxylate cements, each of the four cements employed in this study was tested by the same method. The solubility and disintegration tests of the American Dental Association for dental zinc phosphate cement and for dental silicate cement are identical to those laid down by the International Organisation for Standardisation (I.S.O. Recommendations No. 1566 and 1565).[19,20]

Results

Tensile bond strength to human enamel

The tensile bond strengths of the four cements to enamel are detailed in Table I. The tensile bond strength of the composite cement to the etched enamel surface was the highest and a mean tensile bond strength of 11.1 ± 3.5 MPa recorded. The mean tensile bond strength of the carboxylate cement to human enamel was 2.7 ± 1.2 MPa, of the silicophosphate cement

TABLE I
Tensile Bond Strength of the Cements to Enamel

Cement	Number of specimens	Mean Tensile bond strength. MPa	S.D. MPa
Composite	13	11.1	3.5
Carboxylate	7	2.7	1.2
Silicophosphate	15	0.6	0.4
Zinc phosphate	15	0.4	0.4

0.6 ± 0.4 MPa and of the zinc phosphate cement 0.4 ± 0.4 MPa.

Statistical analysis of the differences between the means of the bond strengths of the four cements to enamel was undertaken using the Student's "t" test. It was found that the differences between the tensile bond strengths of the composite and carboxylate cements and the carboxylate and silicophosphate cements were significant at the 5 percent level. There was no significant difference, however, between the silicophosphate and zinc phosphate cements. It must be pointed out, however, that during testing of the composite cement, two specimens did not fail at the interface. In one specimen the embedded tooth fractured and pulled out of the embedding acrylic, while another failed due to fracture within the enamel. These two were excluded from the series. In the testing of the carboxylate cement, eight of the fifteen specimens failed within the material and not at the site of bonding. For obvious reasons the specimens which failed within the material were excluded in the statistical analysis. Only the tensile bond strengths of the specimens which failed at least partially at the interface were evaluated.

Tensile bond strength to orthodontic band material

The tensile bond strengths of the cements to band material are represented in Table II. The tensile bond strength of the composite cement to band material was found to be the highest and a mean tensile bond strength of 2.5 ± 0.5 MPa was recorded. The mean tensile bond strength of the carboxylate cement was 1.1 ± 0.8 MPa and that of the silicophosphate cement 0.9 ± 1.1 MPa. No recordings from the zinc phosphate cement on band material were possible since, on removal from the incubating bath, all the prepared cement specimens were found to be detached from the band material.

TABLE II
Tensile Bond Strength of Cements to Stainless Steel Orthodontic Band Material

Cement	Number of specimens	Mean tensile bond strength. MPa	S.D. MPa
Composite	10	2.5	0.5
Carboxylate	9	1.1	0.8
Silicophosphate	10	0.9	1.1
Zinc phosphate	10	—	—

Statistical analysis of the differences between the bond strengths of the three cements to band material was undertaken using the Student's "t" test. There was a significant difference between the composite and carboxylate cement at the 5 percent level, but none between the carboxylate and silicophosphate cements. During testing of the carboxylate cement, one specimen failed in the material and was excluded from the statistical analysis.

Marginal leakage

There was no evidence of dye penetration at the enamel-composite cement interface nor at the enamel-carboxylate cement interface. However, at the enamel-silicophosphate cement interface, dye seepage was evident and also the dye penetrated the cement itself. Marginal leakage with the zinc

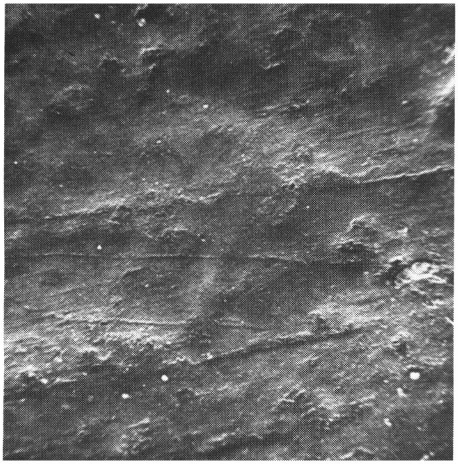

Fig. 2 Featureless appearance of sound human enamel (SEM x 1000).

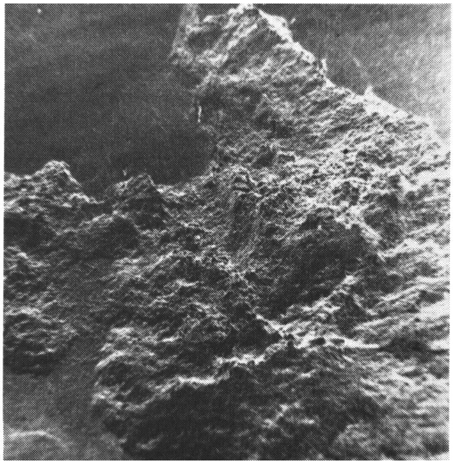

Fig. 3 Scanning electron micrograph of a tooth which displays areas of decalcification of enamel (SEM x 50).

phosphate cement could not be studied due to detachment of the cement from the enamel surfaces in the incubating bath.

Scanning electron microscope studies

The scanning electron micrograph of sound human enamel is shown in Figure 2. It displays a featureless surface topography. A scanning electron micrograph of an extracted tooth with areas of decalcified enamel is seen in Figure 3 and a higher magnification of this decalcified area in Figure 4. An enamel surface which had been etched for two minutes with the "conditioner" supplied with the composite cement demonstrated the characteristic prism-end structure produced by the acid etching (Fig. 5). A higher magnification indicated the preferential etching action of the conditioner and revealed the characteristic honeycomb appearance (Fig. 6). The scanning electron micrograph of the fitting surface of the stainless steel orthodontic band material revealed a grooved pattern (Fig. 7). The scanning electron micrographs show clearly the differences between pathologic enamel decalcification and acid etching with the "conditioner" which is a phosphoric acid solution.

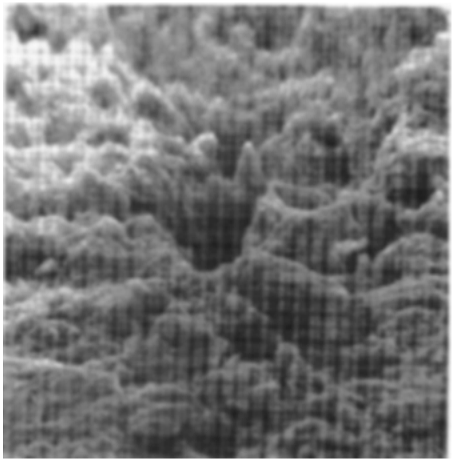

Fig. 4 A higher magnification of the area of decalcification shown in Figure 3 (SEM x 1000).

Solubility and disintegration

Neither the composite nor the carboxylate cements showed any evidence of solubility or disintegration over the 24 hour test period when reported to the nearest 0.1 percent. The silicophosphate cement displayed solubility and disintegration to the extent of 0.8 percent by weight and the zinc phosphate cement to the extent of 0.1 percent at 24 hours. These are within the maximum limits of 1.0 and 0.2 percent

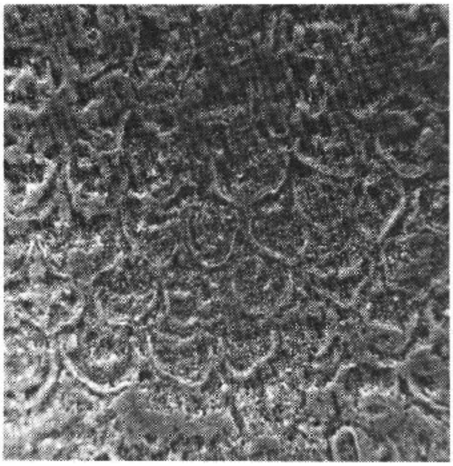

Fig. 5 The characteristic prism-end structure produced by the acid etching of enamel with the "conditioner" supplied with the composite cement (SEM x 1000).

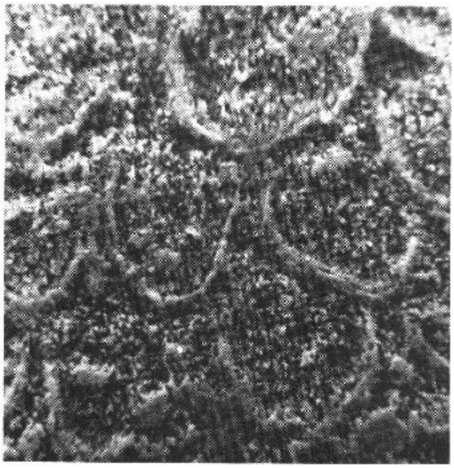

Fig. 6 A higher magnification of an enamel area etched with the "conditioner" shows its preferential etching action producing the characteristic honeycomb appearance (SEM x 2500).

by weight, respectively, as laid down by the specifications used in this study.

Discussion

The cementation of an orthodontic band results in the formation of two interfaces, namely, the enamel-cement interface and the band-cement interface. This is diagrammatically pre-

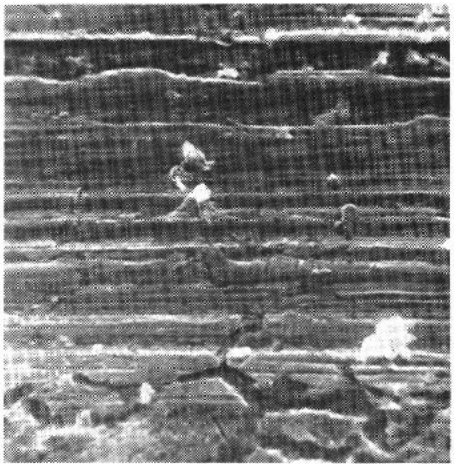

Fig. 7 A scanning electron micrograph of the unpolished fitting surface of a piece of band material (SEM x 500).

sented in Figure 8. The most important interface in so far as the prevention of decalcification and caries formation is concerned is the enamel-cement interface. Failure at this interface could be an important cause of enamel decalcification occurring beneath orthodontic bands.

Gwinnett and Matsui[4] stated that none of the materials available to the dental profession at that time consistently maintained adhesion to tooth structure in the oral environment. In this context adhesion is defined as the molecular attraction between the surfaces of bodies in contact or the attraction between molecules at an interface. This force is called adhesion when unlike molecules are attracted and cohesion when molecules of the same kind are attracted. The material added to produce the adhesion is known as the adhesive and that to which it is applied the adherend. The interface is the zone between the interacting substances.[21]

The success of the retention of orthodontic bands depends, to a large degree, upon the properties of the cement used in the cementation of these bands. One of the desirable properties

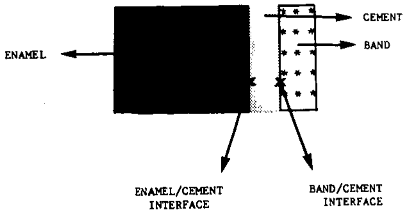

Fig. 8 A diagrammatic representation of the two interfaces produced by cementation of an orthodontic band.

of an orthodontic cement is thus its tensile bond strength to tooth enamel. The results of the tests carried out under the experimental conditions described indicated that the composite cement displayed the highest tensile bond strength to the enamel surfaces.

The composite cement tested in this series required the prior etching of the enamel surface with a "conditioner" supplied with the cement. The effect of etching is to remove the organic cuticle from the enamel surface and thus produce a high energy surface over which the adhesive will readily spread. In addition, the preferential etching action of the acid produces micropores in the enamel surface into which the setting cement will flow and results in mechanical retention of the cement. This ensures the formation of stronger bonding forces between the cement and etched enamel.[4,22,23] The scanning electron microscope studies clearly illustrated the effect of etching of the enamel surfaces.

A question may be posed as to the possible permanent damage done to enamel surfaces that have been etched prior to orthodontic band placement and also performed prior to direct bonding of orthodontic attachments to tooth enamel. Etching produces a loss of surface hydroxyapatite as is evidenced by a white opaque appearance of the etched enamel which is visible macroscopically. Newman and Facq[24] reported that scanning electron microscope photographs revealed that removal of the adhesive and repumicing of the bonded surface restored the tooth surface to its original pumiced appearance. This finding, they stated, corroborated clinical observations and *in vivo* surface-replica interferograms. Lenz and Mühlemann[25] studied the effects of saliva *in vivo* and *in vitro* on etched enamel surfaces and noted that the characteristic prism-end pattern of etched enamel disappeared when exposed to the oral environment for 2 to 48 days. The surfaces became smooth resembling nonetched enamel. They concluded that the disappearance of the etched pattern in teeth exposed to the oral environment was due to abrasion or remineralisation. Lee et al.[26] have shown by means of electron microprobe X-ray spectrophotometry that complete remineralisation of enamel surfaces etched with 50 percent citric acid occurred 45 days after etching and subsequent exposure to the oral environment. Retief[27] has proposed a model to explain interfacial failure. He concluded that no interfacial fracture between an adhesive system and etched enamel should be classified as an interfacial failure, but rather as failure occurring both within the material and the enamel. He substantiated his proposed model by means of surface roughness measurements and atomic absorption spectrophotometry. This proposed model corroborates the observation of Gwinnett and Matsui[4] that, if a dental material was lost from the etched enamel surface, the material which had penetrated into the enamel provided continued protection of the enamel crystals to acid attack by encapsulation of these crystals.

It is noteworthy to mention that carboxylate cements have been reported to chelate to calcium and thereby provide a chemical bond to the enamel.[28] The spectroscopic studies of Beech[29] have confirmed the presence of this chemical interaction between tooth enamel and the polycarboxylate cements.

Smith[30] reported that clinical experience has demonstrated that the stainless steel interface was the most variable part of the bonding system when carboxylate cements were used for direct bonding of orthodontic attachments to teeth. Retief et al.[31] were the first to gain additional mechanical retention of their adhesive formulation to band material for direct bonding techniques by welding stainless steel gauze to the band material.

During the testing of the tensile bond strengths of the four cements to enamel, eight of the fifteen carboxylate cement specimens failed within the material (Table II). Phillips[32] reported a similar finding and stated that most carboxylate specimens failed in cohesion rather than in adhesion at the enamel-cement interface. He concluded that the strength of the bond to enamel exceeded the linear tensile strength of the cement.

It is considered that the significance of the cement at the band-cement interface is important only in so far as its luting and "gap-filling" properties are concerned. This will have the action of mechanically holding the band in place against the tooth. The absence of marginal leakage at both the composite and carboxylate enamel-cement interfaces is a highly desirable property in the prevention of enamel decalcification.

The results of the solubility and disintegration tests carried out under the conditions described indicated that neither the composite cement nor the carboxylate cement displayed any solubility or disintegration when reported to the nearest 0.1 percent by weight. The solubility and disintegration of the silicophosphate cement was 0.8 percent by weight and that of the zinc phosphate cement 0.1 percent. Both these levels of solubility and disintegration are within the limits permitted by the standards laid down by the American Dental Association and the International Organisation for Standardisation. It must, however, be pointed out that the solubility and disintegration of the cements used for orthodontic band cementation are important factors in the prevention of decalcification occurring beneath orthodontic bands since cements that are leached out will allow the ingress of food particles and bacteria between the tooth enamel and the band material and predispose to enamel decalcification. An ideal cement for band cementation would be one which is completely insoluble and does not disintegrate in the oral cavity over the entire period during which the band remains in place.

Cornell[33] found that, although a number of materials gave excellent adhesion to tooth structure initially, all failed to continue to do so when tested under physiological conditions over extended periods of time. It is not uncommon for orthodontic bands to remain cemented for a period of up to 24 months during the course of treatment. In addition, Beebe[34] reported that a patient's tooth cannot be thoroughly dried at room temperature even if a vacuum pump was applied to his mouth. Furthermore, even if it were possible to dry a tooth surface completely, the dryness could not be maintained because of the fluid flow which occurs from the pulp of the tooth to the enamel surface.[35,36] As water is ever present on the tooth surface and as it actually functions as a liquid adhesive, a dental adhesive must compete with it for the binding sites on the tooth surface. Retief,[37] in referring to the presence of water in the oral cavity, stated that it may be the precursor of chemical activity at the adhesive-adherend interface which will eventually dislodge the bond. In addition, many adhesive materials may absorb water leading to swelling and dimensional changes in their bulk which will lead to stress concentrations at the interface and this, in turn, will have an adverse effect on the bond strength.

We wish to stress that the results obtained in this study should not be directly extrapolated to the situation obtaining in the oral cavity since laboratory evaluations are carried out under simulated intraoral conditions. In the oral cavity, however, additional conditions exist which tend to counteract the mechanisms of bonding and adhesion. These factors were fully discussed by Retief.[37]

Conclusions

It is suggested that decalcification occurring beneath orthodontic bands is, among other factors, related to the tensile bond strength of the cement to the enamel surface, its marginal leakage at the enamel-cement interface, and its solubility and disintegration.

Under the laboratory conditions described in this study the composite cement exhibited the greatest tensile bond strength to both enamel and band material. The composite cement also displayed no marginal leakage at the enamel-cement interface and showed no solubility or disintegration under the experimental conditions. It could therefore be anticipated that, of the four cements evaluated in this study, the composite cement would have the greatest potential for eliminating pathologic enamel decalcification occurring beneath orthodontic bands.

School of Dentistry
University of the Witwatersrand
Johannesburg, South Africa

Acknowledgments

The authors wish to acknowledge the technical assistance of the staff of the Dental Research Unit. Thanks are also due to Mr. Hall of the Computer Division of the National Institute for Personnel Research for his advice on the statistical evaluation of the results, to the staff of the Electron Microscope Unit of the University of the Witwatersrand and to Miss B. Slack for typing the manuscript.

This research project was partly financed by Elida Gibbs (Pty) Ltd.

References

1. Noyes, H. J.: Dental caries and the orthodontic patient. *J. Am. Dent. Assn.*, 24:1243-1254, 1937.
2. Gibbin, F. E.: Control of caries during orthodontic treatment. *Int. J. Orthodont. and Oral Surg.*, 23:1205-1211, 1937.
3. Docking, A. R., Donnison, J. A., Newbury, C. R. and Storey, E.: The effect of orthodontic cement on human enamel. *Austr. J. Dent.*, 57: 139-149, 1953.
4. Gwinnett, A. J. and Matsui, A.: A study of enamel adhesives. The physical relationship between enamel and adhesive. *Arch. Oral Biol.*, 12: 1615-1620, 1967.
5. Ballenseifen, J. W. and Madonia, J. V.: A study of dental plaque in orthodontic patients. *J. Dent. Res.*, 49:320-324, 1970.
6. Lefkowitz, W. and Bodecker, C. F.: Concerning the vitality of the calcified dental tissues II. *J. Dent. Res.*, 17:453-463, 1938.
7. Castello, I., Massler, M., Monteleone, V. L. and Suher, T.: The effect of zinc oxyphosphate cement on enamel. *Am. J. Orthodont.*, 34:271-277, 1948.
8. Dolce, J. J.: Caries incidence in relation to orthodontic therapy. *Am. J. Orthodont.*, 36:534-545, 1950.
9. Meyers, M. J.: Protection of enamel under orthodontic bands. *Am. J. Orthodont.*, 38:866-873, 1952.
10. Bach, E. N.: Incidence of caries during orthodontic treatment. *Am. J. Orthodont.*, 39:756-778, 1953.
11. Wisth, P. J.: The role of zinc phosphate cement in enamel surface changes on banded teeth. *Angle Orthodont.*, 40:329-333, 1970.
12. Gursin, A. V.: A study of the effect of stannous fluoride incorporated in dental cement. *J. Oral Therap. and Pharm.*, 1:630-636, 1965.
13. Wei, S. H. Y. and Sierck, D.: Fluoride uptake by enamel from zinc phosphate cement containing stannous fluoride. *J. Am. Dent. Assn.*, 83: 621-624, 1971.
14. Shannon, I. L.: Caries risk in teeth with orthodontic bands. *J. Acad. Gen. Dent.*, 20:24-28, 1972.

15. Hanke, G.: *Screening Test Methods in Measuring Adhesion of Selected Dental Resins.* M.S. Thesis, Indiana University School of Dentistry, 1968.
16. Phillips, R. W., Swartz, M. L. and Rhodes, B.: An evaluation of a carboxylate adhesive cement. *J. Am. Dent. Assn.,* 81:1353-1359, 1970.
17. Holliger, H. H.: Penetration of restoration margins. A fluorescent dye study. *Dent. Rad. and Phot.,* 40:9-14, 1967.
18. American Dental Association: *Guide to Dental Materials and Devices,* 5th ed., pp. 151-160. Chicago, 1970-1971.
19. International Organisation for Standardisation: *ISO Recommendation R1565.* Dental Silicate Cement, 1st ed. ISO: Geneva, 1970.
20. International Organisation for Standardisation: *ISO Recommendation R1565.* Dental Zinc Phosphate Cement, 1st ed. ISO: Geneva, 1970.
21. Retief, D. H.: Adhesion in dentistry. *J. Dent. Assn. S. Afr.,* 28:11-24, 1973.
22. Lee, B. D., Phillips, R. W. and Swartz, M. L.: The influence of phosphoric acid etching on retention of acrylic resin to bovine enamel. *J. Am. Dent. Assn.,* 82:1381-1386, 1971.
23. Retief, D. H.: Effect of conditioning the enamel surface with phosphoric acid. *J. Dent. Res.,* 52:333-341, 1973.
24. Newman, G. V. and Facq, J. M.: The effects of adhesive systems on tooth surfaces. *Am. J. Orthodont.,* 59:67-75, 1971.
25. Lenz, H. and Mühlemann, H. R.: *In vivo* and *in vitro* effects of saliva on etched or mechanically marked enamel after certain periods of time. *Helv. Odont. Acta,* 7:30-33, 1963.
26. Lee, H., Ocumpaugh, D. E., Shaffer, J. and Sheble, A. M.: Sealing of developmental pits and fissures: IV Measurement of *in vivo* fluoride pickup by electron microprobe spectrophotometry. *J. Dent. Res.,* 51: 634-639, 1972.
27. Retief, D. H.: Failure at the dental adhesive-etched enamel interface. *J. Oral Rehab.,* 1:265-284, 1974.
28. Smith, D. C.: A new dental cement. *Brit. Dent. J.,* 125:381-384, 1968.
29. Beech, D. R.: A spectroscopic study of the interaction between human tooth enamel and polyacrylic acid (polycarboxylate cement). *Arch. Oral Biol.,* 17:907-911, 1972.
30. Smith, D. C.: Lutes, glues, cements and adhesives in medicine and dentistry. *Biomed. Eng.,* 8:108-115, 1973.
31. Retief, D. H. and Dreyer, C. J.: Epoxy resins for bonding orthodontic attachments to teeth. *J. Dent. Assn. S. Afr.,* 22:338-346, 1967.
32. Phillips, R. W.: *Skinner's Science of Dental Materials,* 7th ed., p. 490. Philadelphia: Saunders, 1973.
33. Cornell, J.: Adhesion to natural teeth. In: *Workshop Proceedings, Adhesive Restorative Dental Materials,* ed. R. W. Phillips, p. 159, Owen Litho Service, Indiana, 1961.
34. Beebe, R. A.: Gas absorption studies on bone material. In: *Workshop Proceedings, Adhesive Restorative Dental Materials,* ed. R. W. Phillips, pp. 75-84, Owen Litho Service, Indiana, 1961.
35. Bergman, G.: Techniques for microscopic study of the enamel fluoride *in vitro. Odont. Revy,* 14:1-7, 1963.
36. Linden, L.: Microscopic observations of fluid flow through enamel *in vitro. Odont. Revy,* 19:349-365, 1968.
37. Retief, D. H.: The intra-oral factors affecting adhesion. *J. Dent. Assn. S. Afr.,* 25:392-399, 1970.