

Bonding agents: adhesive layer thickness and retention to cavity surfaces with time

Keywords: Bonding agent thickness

SUMMARY

This study assessed bonding agent thickness, cover and the influence of long-term storage on bonding agent retention to enamel and dentine surfaces in cut occlusal cavities in 46 human molar teeth. Two specimens were etched and set aside. The remaining specimens were divided into two equal groups and treated with either Optibond or Scotchbond Multipurpose Plus (SMPP) up to the adhesive stage. Thereafter two specimens from each group were stored for 0, 1, 3, 7 and 14 days and 1, 2, 3, 6 and 12 months in 1% NaCl. After storage the cavity surfaces were examined in a scanning electron microscope (SEM) and scored as to bonding agent cover. One specimen from each treatment was then embedded in resin, two sections prepared midway through each cavity, polished and re-examined in the SEM to measure bonding agent thickness at 13 sites along the cavity surface. Both bonding agents showed highly variable and significant ($P < 0.05$) bonding agent cover and layer thickness according to cavity site, SMPP more so than Optibond. Pooling of SMPP adhesive was apparent in cavity angles. Mean film thickness was significantly different between Optibond ($221 \pm 130 \mu\text{m}$) and SMPP ($118 \pm 106 \mu\text{m}$). There was no significant difference in bonding agent thickness between long- and short-term storage.

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Introduction

The mechanism of adhesive bonding is currently based on acid etching of both the enamel and dentine of the tooth cavity surface. Etching selectively removes the smear layer as well as portions of the enamel rod ends up to a depth of $50 \mu\text{m}$ by a process of demineralisation. Subsequent appli-

OPSOMMING

In hierdie studie is die dikte van bindingsagense, bedekking, en die invloed van langtermyn berging op die retensie van die agens aan glasuur en dentien oppervlakte in voorbereide okklusale kaviteite in 46 menslike molare ondersoek. Twee monsters is geëts en opsy gesit. Die oorblywende monsters is verdeel in twee gelyke groepe en behandel met Optibond of Scotchbond Multipurpose Plus (SMPP) tot by die adhesiewe stadium. Hierna is twee monsters van elke groep vir 0, 1, 3, 7 en 14 dae, en vir 1, 2, 3, 6 en 12 maande, in 1% NaCl geberg. Na berging is die kaviteitsoppervlakte deur middel van 'n skandeer elektronmikroskoop (SEM) ondersoek en punte is toegeken na gelang van die bedekking van die bindingsagens. Een monster van elke behandeling is hierna in hars ingebed. Twee snitte is in die middel van elke kaviteit voorberei, is gepoleer en in die SEM ondersoek. Bindingsagens dikte op sewe plekke op die kaviteitsoppervlakte is gemeet. Beide bindingsagense het hoogsveranderlike en betekenisvolle ($P < 0.05$) bedekking en dikte getoon op verskillende plekke, SMPP in groter mate as Optibond. Poelvorming van SMPP was opvallend in kaviteitshoeke. Verskille in gemiddelde filmdikte was opvallend met Optibond op $221 \pm 130 \mu\text{m}$, en SMPP op $118 \pm 106 \mu\text{m}$. Daar was geen betekenisvolle verskille in die dikte van die agense na kort- of langtermyn berging nie.

cation of the bonding agent to this surface causes the resin to flow into the resultant defects by capillary action (Asmussen, 1985). An excellent micromechanical retention or bonding results due to the increased surface area once polymerisation has taken place. Pretreatment of dentine with a weak acid removes the inorganic component of the exposed dentine to an optimal depth of $10 \mu\text{m}$ (Leinfelder, 1999). The applied bonding agent diffuses into the inter-collagenous spaces which when cured forms a hybrid layer of resin polymer and enmeshed collagenous fibres which forms the basis of dentine bonding. In practice the adhesive fluid is not limited to the etched layer of enamel or the hybrid layer of dentine. Inevitably a layer of cured adhesive of variable thickness is present on the cut tooth surface. The role this layer plays in restoration success is unknown.

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Bonding agents must maintain their tenacity despite the multitude of chemical and physical assaults which challenge this material in the moist oral environment. Ionic, diffusion, dissolution and biological forces brought about by marginal leakage may directly attack the complex adhesive-adherend surface (Hilton and Schwartz, 1995). Recent studies have drawn attention to possible hydrolytic bond degradation at the resin-dentine interface as a consequence of such dynamics (Blunck and Roulet, 1999; Armstrong *et al.*, 2000; Hashimoto *et al.*, 2000). Physical and mechanical forces generated by polymerisation shrinkage, temperature changes and masticatory forces are in part cushioned and absorbed by the bonding agent in its role as an elastic buffer (Opdam, Roeters and Verdonschot, 1997; Staninec *et al.*, 1995). Implicit in this function is an even distribution of load which can only occur if the bonding agent thickness is uniform throughout the interface.

While much has been written about placement techniques which ensure optimal bonding (Swift, Perdigao and Heymann, 1995; Marshall *et al.*, 1997; Leinfelder, 1999) less is known about extrinsic factors which could impact on the durability of the bonding agent itself. To better understand the potential vulnerabilities of the bonding agent as an interfacial material between the tooth cavity surface and restorative material this study was undertaken to:

- determine the adhesive bonding agent cover within the cavity
- assess the film thickness of the adhesive bonding agent along the cavity wall and floor
- examine the retention of the bonding agent on enamel and dentine in specimens stored up to one year in 1% NaCl.

Materials and methods

Ethics clearance to collect teeth for this study was obtained from the Committee for Research on Human Subjects of the University of the Witwatersrand, Johannesburg. Non-carious, human teeth of unknown history were obtained from dental clinics in and around Johannesburg. These were collected and stored in distilled water with thymol at room temperature (20°C). Occlusal cavities were cut in 46 molar teeth using a W & H series 700 air rotor and a tungsten carbide straight fissure bur (No. 57) to expose both enamel and dentine surfaces. The bur was replaced after every 12 cavities to ensure that all specimens were prepared with a sharp instrument. Two specimens were set aside to serve as controls for smear layer covered cavity surfaces. The other 44 specimens were divided into two groups of 22 each and each group treated with one of two adhesive systems according to the manufacturers' recommendations:

*Scotchbond Multipurpose Plus*¹ (SMPP)

- The cavities were etched for 15 sec with 35% phosphoric acid.
- Activator was applied and gently air-dried for 5 sec.
- Primer was applied and gently air-dried for 5 sec.
- One drop of adhesive and catalyst were mixed and applied to the primed surface and allowed to dry (no air-thinning at this stage).

*Optibond*²

- Enamel was etched for 30 sec, dentine for 15 sec with 35% phosphoric acid gel, washed and dried.
- Optibond Prime (Bottle 1) was applied and scrubbed continuously for 30 sec on dentine, dried and light-cured for 20 sec.
- Optibond Light Cure Adhesive (Bottle 2) was applied and light-cured for 30 sec.

Two etched cavities from each group were set aside to permit viewing of the etched surfaces (standard). Another two cavities from each group, treated up to the adhesive stage, were set aside to serve as zero time specimens. The remaining specimens were placed individually in specimen bottles and totally submerged with 1% NaCl plus a few grains of thymol.³ The specimens were aged at room temperature (average 20°C) for the following time periods: 1, 3, 7 and 14 days and 1, 2, 3, 6 and 12 months.

At each time interval two specimens from each group were removed, rinsed with a jet of distilled water and air-dried in Petri dishes. The roots of the specimens were removed with a Hi-Di 679M diamond friction grip bur⁴ in a water-cooled turbine. The tooth crowns were mounted on aluminium specimen stubs using double-sided adhesive tape and DAG (DAG 580 colloidal graphite in alcohol)⁵. After coating the specimens with gold palladium they were viewed in a JEOL 840⁶ scanning electron microscope (SEM).

The degree of bonding agent cover of each cut cavity was assessed on three enamel and three dentine surfaces using a stratified random sampling technique at a magnification of x400 which resulted in a viewing field of 40 x 10³ μm². The total assessment field for each cavity therefore comprised 120 x 10³ μm² for an enamel surface and 120 x 10³ μm² for the dentine surface. The degree of coating on the surfaces was scored as follows: no coat = tooth structure was clearly visible displaying sharp etched edges; thin coat = tooth structure visible but no sharp edges apparent; partly coated = field partly coated with bonding agent; thick coat = surface features completely obscured by bonding agent.

The specimens were examined double blind and the data entered and processed using a SUNSPARC center 2000 computer using SAS (1990) with the dependent variable being bonding agent cover and independent variables

being the bonding agent and time. A Linear Logistic Statistical Analysis (PROC CATMOD) was used to analyse the data with the level of statistical significance set at $P < 0.05$. To examine bonding agent cover over time, specimens were grouped into short-term (0, 1, 3, 7 and 14 days) and long-term (1, 2, 3, 6 and 12 months) classes and bonding agent cover compared between the two groups.

After SEM examination, one specimen from each treatment was embedded in a 5:1 mix of Araldite M (Batch no 200498) and HY956 resin (Batch no 260398)⁷. Two sections, 750 μm thick were prepared midway through the cavity using a slow speed, Isomet, diamond saw⁸ and re-embedded in the same resin. Polishing of the surface of each section was accomplished using an IMPTech 20 DVT grinder polisher⁹ and a succession of wet silicon carbide papers of grit size 180-2500. Final polishing took place using a diamond paste from 3.0-1.0 μm and using DP lubricant. In between diamond polishing the surface was cleaned using a B-220 ultrasonic bath¹⁰ and alcohol. The polished samples were mounted, coated and viewed in the SEM. Bonding agent thickness was measured at 13 points along the cavity margin viz.: rim = edge of each cavity margin within 30 μm of the edge; qrt = a quarter way down each cavity wall; hlf = halfway down each cavity wall; thr = three quarters down each cavity wall; ang = at both floor angles; flr = a quarter way into the cavity floor from each wall and cnt=centrally on the floor (Fig. 1). All measurements were made at right angles to the cut cavity surface except for 'ang' where the thickest measurement from the cavity angle was taken. Thus each section yielded two readings for rim, qtr, hlf, thr, ang, flr and one for cnt.

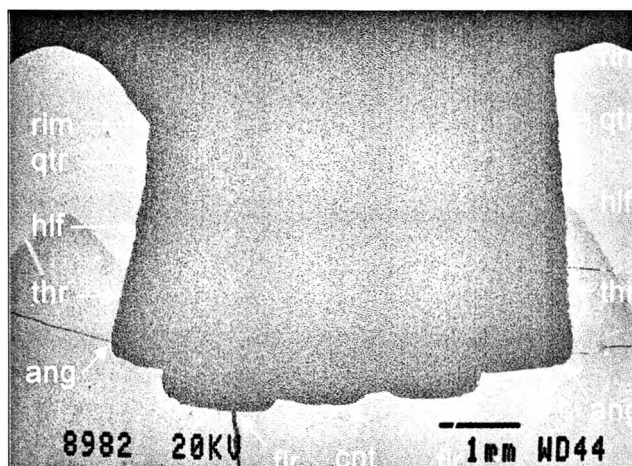


Fig. 1. Micrograph showing the points of measurement for bonding agent film thickness along the cavity margin.

The data were similarly entered and analysed as before using SAS (1990). ANOVA and Tukey's test were used to establish if there was any statistical difference between the bonding agent film thickness between bonding agents and along both the wall and floor of the cavity margin with the

level of statistical significance set at $P < 0.05$. Similarly specimens were grouped into short-term (0, 1, 3, 7 and 14 days) and long-term (1, 2, 3, 6 and 12 months) classes to establish the effect of long-term 1% NaCl storage on bonding agent layer thickness. The dependent variable was bonding agent film thickness and independent variables were the bonding agent, site along the cavity margin wall and floor and time.

Results

Specimen surface

Optibond maintained a uniform cover over the entire cavity surface as indicated by the absence of bur marks on the cavity surface (Fig. 2). In some areas this bonding agent was seen to pull away from the cavity surface because of dehydration. SMPP cover was highly variable in that the adhesive pooled in the angle of the floor and wall while other areas of the cavity surface seemed devoid of bonding agent (Fig. 3). No dehydration artifact in the form of pulling away of the bonding agent was seen in these specimens.

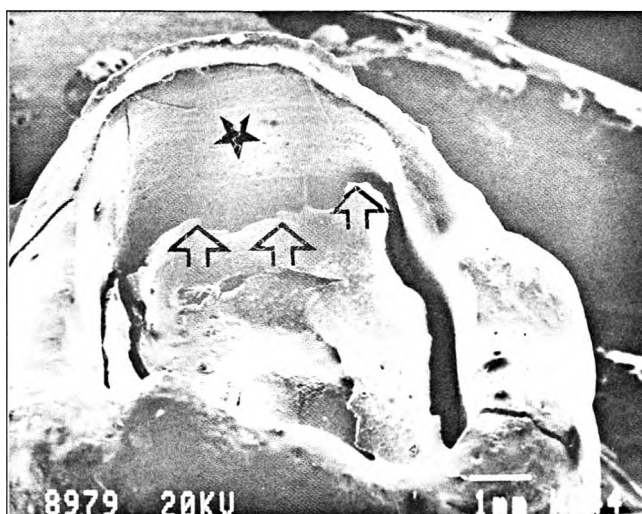


Fig. 2. A two-week Optibond cavity surface. Bur marks are hardly visible on the cavity wall* indicating the presence of bonding agent on the surface. Note the artefactual pulling away of the bonding agent at the cavity angle (arrowed).

Bonding agent cover

There was a total of 120 observations for bonding agent cover for each bonding agent. Statistical analysis showed highly significant ($P < 0.00001$) differences between bonding agent cover for the two different bonding agents (Fig. 4).

Time

No significant difference was apparent in bonding agent

cover over time, indicating that both bonding agents are well retained on both enamel and dentine. Significance

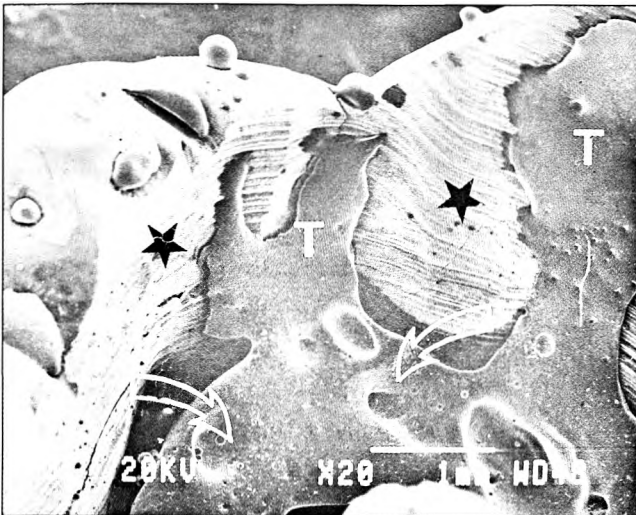


Fig. 3. A three-month SMPP cavity surface. The obvious bur marks on the cavity wall (■) interspersed with tongues of bonding agent (T) and pooling of the material in the cavity angle (arrowed) indicate the irregular cover of SMPP.

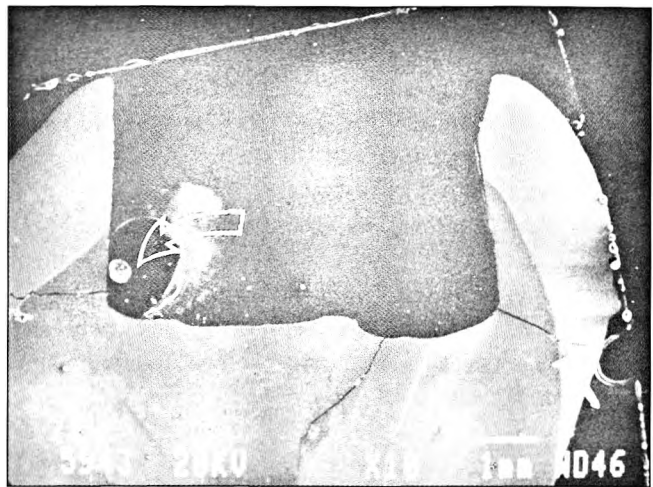


Fig. 5. Sectioned cavity of a six month SMPP specimen. Note the pooling of the bonding agent in the cavity angle (arrowed). At this magnification the thin film of bonding agent is not visible at the cavity margin.

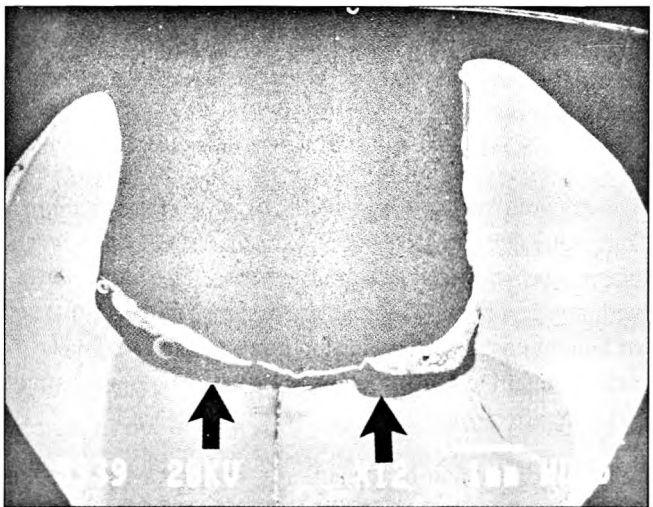


Fig. 6. Sectioned cavity of a one year Optibond specimen clearly showing variable bonding agent distribution along the cavity surface. The Optibond has pulled away along the cavity floor (arrowed) due to specimen dehydration.

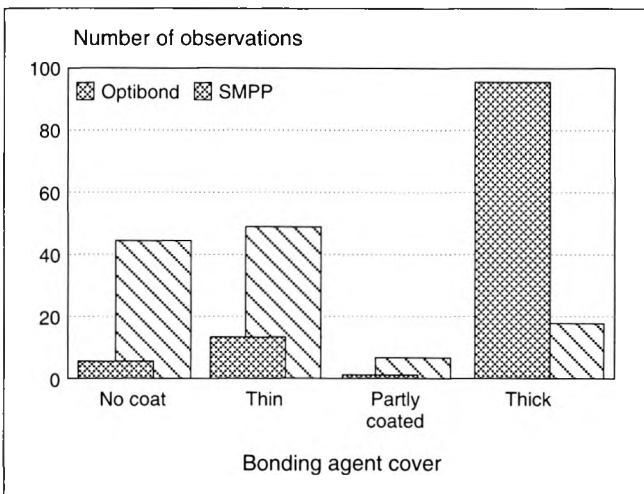


Fig. 4. Differences in bonding agent cover on cavity surfaces.

ranged between $P = 0.9960-0.8938$ for Optibond and SMPP whether assessed against enamel or dentine.

Sectioned specimens

The zero time Optibond specimens proved particularly brittle after SEM examination. The bonding agent layer fragmented during section preparation and was lost to the study.

Bonding agent thickness

There were a total of 20 bonding agent thickness records for sites rim, qrt, hlf, thr, ang and flr and 10 for cnt for SMPP specimens and 18 and 9 respectively for Optibond.

The most striking observation when viewing the sectioned specimens was the variability in film thickness (Optibond between 0 -1150 μm and SMPP 0-1700 μm) of the bonding agents coating the cavity surfaces (Figs 5 and 6). This is best illustrated by a box and whisker plot of the bonding agent cover along the cavity surface (Fig. 7). There was a significant difference in mean layer thickness between the two bonding agents. Optibond had a mean film thickness of $221 \pm 130 \mu\text{m}$ while SMPP was $118 \pm 106 \mu\text{m}$ ($P = 0.0110$; $t = 7.18$).

The cavity site proved to play a role in bonding agent thickness. Optibond tended to increase in thickness as one moved down the cavity wall to a maximum at the cavity angle and then decreased in thickness towards the centre cavity floor. Bonding agent thickness in SMPP specimens

was more variable and with few exceptions comparisons only varied significantly when bonding agent thickness at the cavity angle was compared with other values (Table 1).

Time

With a single exception there proved to be no significant difference between bonding agent thickness in the long and short term in either treatment ($P = 0.3300 - 0.9548$). The only exception was Optibond treated enamel where significance ($F = 5.09$; $P = 0.0384$) was apparent between bonding layer thickness in the short term $125 \pm 110 \mu\text{m}$ and long term $42 \pm 33 \mu\text{m}$.

Discussion

The purpose of this study was not to assess how well the bonding agents penetrated the etched enamel, nor to evaluate the effectiveness of bonding agent infiltration into the collagen matrix of the demineralised dentine. This has been reported on previously in the promotional material supplied by the manufacturers. The objective of the study was to determine the cover and placement thickness of Optibond and SMPP on the cavity surface and to assess the effects of long-term aqueous storage on both these aspects.

Consequently there are three findings in this investigation. This study illustrates the importance of assessing bonding agent cover of cavity walls on the surface in conjunction with sectioned material. Secondly it shows the inconsistency of bonding agent thickness along the cavity wall. Finally neither retention nor thickness of the bonding agents studied were affected by one year storage in 1% NaCl.

Surface examination of the adhesive-treated cavity gave the impression that Optibond formed a uniform layer over the cavity surface in contrast to the apparently patchy cover displayed by SMPP. The sectioned, polished specimens showed that both bonding agents coated the cavity surface unevenly. The highly significant differences between the coating of the bonding agent on the cavity surface appears related more to the type of bonding agent (filled versus unfilled) rather than the actual presence of the bonding material as such. The filled Optibond was highly visible on the cavity surface and this considerably eased the surface score assessment resulting in only seven of the 120 assessment sites being free of bonding agent. Unfilled SMPP was more difficult to assess and score. A rounded and smooth surface appearance of the intertubular dentine indicated the formation of a hybrid layer but this was not always clear cut, hence 12 dentine surfaces were scored as having no coat. Of course, the 'no coat' score could include specimens that were fully infiltrated with SMPP yet had no obscuring surface layer. In the enamel an infiltrated etched layer was indicated by a diffuse film covering enamel. The poor visibility and thin film of SMPP could also account for

the surprising finding that more than half the enamel sites were judged to have no bonding agent cover. However data from the sectioned material indicated that a mean bonding agent thickness of $3.1 \mu\text{m}$ was measured on the enamel surface. What could not be measured was the bonding agent which had penetrated the etched enamel to effect a micromechanical interlocking. Thus the large number of sites which showed no measurable bonding agent indicates only the absence of an observable surface layer and in no way indicates the bonded status of the enamel or dentine. The purpose of the study was to assess and compare bonding agent thickness over and above the surface of the tooth tissue.

For both bonding agents the bonding layer was thinnest at the cavity margin and thickest at the cavity angle. Layer thickness for Optibond showed a progressive increase down the cavity wall to the angle and then tapered off towards the centre of the cavity floor while SMPP showed great film variability. This is further emphasised by the maximum and minimum values shown in the box and whisker plot (Fig. 7) and the significant differences in layer thickness between sites along the cavity wall (Table 1). Peter *et al.* (1997) suggests that pooling of dentine bonding agents at cavity angles arises because of the difference in viscosity between primers and the unfilled adhesives. Air thinning is unable to drive the higher viscosity adhesive through the primed collagen network because of the 'damming' effect of the

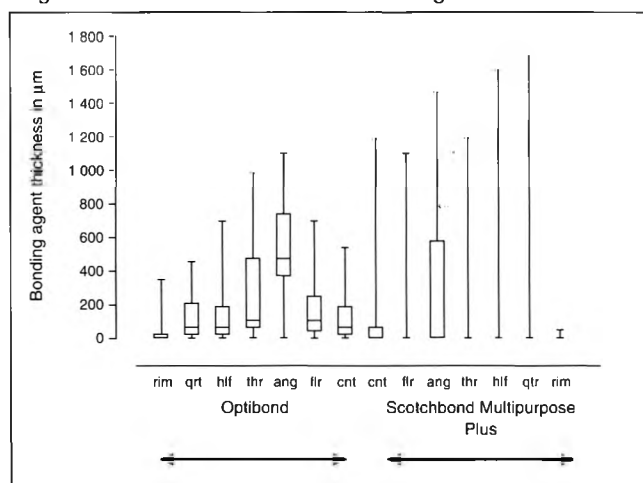


Fig. 7. Box and whisker plot of bonding agent film thickness along the cavity surfaces.

preparation angle. In the case of filled adhesives they feel the increased viscosity and the role of the oxygen inhibition layer is a major cause of thick bonding layers.

While variation in bonding agent thickness has been previously noted, especially at the cavity angle, a systematic analysis of bonding agent thickness along the cavity wall and floor has not been reported. Thickness differences have been ascribed to irregularities in the cavity surface (Griffiths

Table 1. Mean bonding agent film thickness in μm as measured at 13 sites along the cavity wall

Site	Optibond	Scotchbond Multipurpose Plus
Rim	34 \pm 53	3 \pm 7
Quarter	124 \pm 126	104 \pm 232
Half	157 \pm 175	55 \pm 179
Three quarters	277 \pm 229	131 \pm 208
Angle	587 \pm 243	321 \pm 277
Floor	180 \pm 174	87 \pm 196
Center	156 \pm 164	133 \pm 304

The * and - connected by lines indicates that the bonding agent thickness between the two sites is significantly different at $P < 0.05$.

and Watson, 1995); air blowing (Griffiths and Watson, 1995; Opdam *et al.*, 1997); application technique (Opdam *et al.*, 1997); viscosity (Staninec *et al.*, 1995; Opdam *et al.*, 1997); cavity design (Opdam *et al.*, 1997) and incomplete curing of the bonding agent (Staninec *et al.*, 1995). In our study any of the above could apply.

There are two obvious criticisms for the inconsistency of bonding agent layers found in our study. The first would be that of poor technique. Since the operator is highly skilled, this is unlikely and the presented variable bonding agent thickness probably reflects the real situation in clinical dentistry. This is supported by unpublished data of bonding agent thickness measured in seven composite resin restored teeth obtained from full mouth extractions which indicates this layer can vary between 0–250 μm . Both Griffiths and Watson (1995) and Perdigao *et al.* (1996) noticed that air thinning had a tendency to cause pooling of the adhesive into irregularities on the dentine surface and at the angle of the cavities. Additionally Peter *et al.* (1997) report on film thicknesses reaching 254 μm in the inner angle lines of crown preparations. It could secondly be argued that in the absence of composite resin restoration our measured bonding layer thickness could be greater than would actually occur following compressive placement procedures and polymerisation shrinkage. Precuring of the bonding agent is recommended prior to restoration (McCabe and Rusby, 1994; Peter *et al.*, 1997) and while bonding agents do undergo plastic deformation in response to stresses (Wakasa, Yamaki and Matsui, 1995), the results of Ciucchi *et al.* (1997) infer that it seems unlikely that placement of the composite resin would play a role in permanently compressing or thinning out the excessively thick areas of bonding agent.

Long-term contact with water has been implicated in hydrolytic bond degradation at the resin-dentine interface. This has been confirmed by Blunck and Roulet (1999) who quantitatively analysed dentine margins after long-term water storage; by tensile testing on its own (Armstrong *et al.*, 2000) or in conjunction with subsequent examination of the fractured surface (Hashimoto *et al.*, 2000). It is noteworthy that the present study shows no reduction in bonding agent thickness over time which could point to a breakdown of the hybrid layer. However we concur that the rigours of our experimental procedure are not conducive to observing subtle changes which could point to hydrolysis at this interface.

So what should the thickness of a bonding agent between the tooth structure and the composite resin restoration be? Two researchers have been specific about optimal bonding agent film thickness. Burke and McCaughey (1995) feel the ideal bonding agent should have a film thickness of $>10 \mu\text{m}$ if it is to be suitable for use with indirect restorations but it is not clear whether this includes the depth of the hybrid layer as recommended by Leinfelder (1999). Peter *et al.* (1997) quote a range of 50–100 μm as being desirable for bonding ceramic crowns to dentine. In order to gain clarity on this matter bonding layer thickness has been extensively investigated to determine its role on possible restoration longevity. In studies to gauge its effects on bond strength (Retief, Wendt and Bradley, 1989; Langdon, Moon and Barnes, 1994; Staninec *et al.*, 1995; Zheng *et al.*, 2000) the results appear to apply more to the specific bonding agent as such, rather than to the thickness of the bond layer. Opdam *et al.* (1997) report that thick adhesive layers seemed to prevent the formation of interfacial gaps between tooth and restoration and acted as a superior elastic buffer compared to thinner layers, whereas Hilton and

Schwartz (1995) suggest that a thick adhesive layer adversely affects bond strength, increases crack propagation, elevates the thermal co-efficient of expansion mismatch with the tooth and decreases the load bearing and wear component of the restoration. Conversely an adhesive layer should be thick enough to polymerise in the deeper reaches of the hybrid zone to permit optimal dentine adhesion while retaining an uncured layer on the surface to bond to the composite. However it is unclear what is meant by 'thick' or 'thick enough' layer. It seems that the parameters of suitable bonding agent thickness still needs to be determined.

Does an uneven bonding agent layer actually matter? Consensus of opinion is that film thickness should be even along the entire composite resin-tooth interface to ensure consistent bonding and uniform stress distribution. The location of failure sites is often interpreted as reflecting the weakest link in the restoration system. It could be that the high proportion of mixed failures in tensile and shear bond tests (Eick *et al.*, 1993; Staninec *et al.*, 1995; Yoshiyama *et al.*, 1995) may be related to variations in bonding agent thickness.

The variable nature of the bonding agent at the cavity margin in this study is worrying. Staninec *et al.* (1995) noted no discernable bonding resin near the margins with one of the bonding agents in his study. Marginal finish is an important factor in the successful outcome in the restorative treatment. A poor marginal adaptation as a result of insufficient or inadequate bonding will lead to the formation of gaps which will be filled with micro-organisms, cause fractures and loss of material, leading to treatment failure. However all the above is speculation and there seems to be a great need for evidence-based conclusions on the effect of irregular bonding agent thickness on bonded restoration longevity.

Conclusions

The pursuit of overcoming the complexities of bonding restorative material to enamel and dentine appears to have detracted from the obvious effects that the physical thickness of a bonding agent can have on overall restoration success or failure. It seems that further work is needed to define and understand the role of the bonding agent within the restoration process other than that of its function as an adhesive link between tooth and filling material.

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Footnotes

1. 3M Dental Products, St Paul, MN
2. Kerr Manufacturing Co., Romulus, MI
3. BDH Chemicals Ltd, Poole, England
4. ASH, England, UK
5. Acheson Colloids, Prince Rock, Plymouth, UK
6. JOEL Ltd, Tokyo 196, Japan).
7. Jackson Fibreglass, Pty Ltd, Johannesburg South Africa

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