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Water sorption and mechanical behaviour of cosmetic direct restorative materials in artificial saliva

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Abstract

Objectives: To evaluate the water sorption and mechanical behaviour of a compomer in comparison with those of its nominal forerunners, a filled resin restorative material and a conventional glass ionomer cement.

Method: Compomer (Dyract AP) (D-AP), filled resin (SureFil) (SF), and glass ionomer (ChemFlex) (CF) (all Dentsply, Addlestone, UK) restorative materials were tested. Forty bar specimens ($26 \times 1.5 \times 1.0$ mm^3) of each material were prepared according to the manufacturer's instructions and randomly distributed into eight groups: dry air (22% RH), saturated water vapour (WV) (100% RH), and five in artificial saliva (AS) at pH6, all at 37°C, as well as untreated control (UC) (23°C, 50% RH). Water sorption was assessed gravimetrically; flexural strength and elastic modulus were determined in three-point bend. The control group was tested at 24 h; AS groups were separately tested after 0.5, 1, 3, 6 and 9 months; the other two at 9 months.

Results: Mass gain for SF, D-AP and CF in AS was up to 0.17%, 1.2% and 7.0%, respectively. CF showed a marked decrease of strength in AS compared with other groups, followed by a gradual slight rise to a peak at 3 months. Unlike SF and CF, whose flexural strength remained relatively stable, that of D-AP showed a sharp decline from the 1 month peak ($P = 6 \times 10^{-7}$) after 6 months in AS. D-AP also showed a slight decline in flexural modulus from a peak, that of SF was quite stable, while CF showed no peak. The values of flexural strength for both CF and D-AP at 9 months were significantly lower in AS than WV, but SF showed no such difference.

Significance: Materials intended for service in the mouth must be stored in a realistic medium if the results of testing are to be interpretable. Dyract AP, a compomer, does not seem suitable for application in stress bearing areas as is currently recommended by its manufacturer. The rapid decline in flexural strength after 1 month of exposure to AS and its progressive fall in flexural modulus suggest a progressive deterioration of the material and this necessitates re-examination of the chemistry of compomers, if the behaviour is typical of the class.

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Keywords: Compomer; Glass ionomer cement; Filled resin restorative material; Water sorption; Flexural strength; Flexural modulus

1. Introduction

Glass ionomer cement (GIC) and filled resin restorative materials (FRRMs) are well-established cosmetic restorative materials. Their biocompatibility, tooth-like appearance and relative ease of use led to a wide acceptance of both materials [1]. The advantages of GICs over FRRMs are their apparent cariostatic property due to sustained release of fluoride [1–3] and a long-term adhesion to tooth structure [4–6], although their handling, mechanical strength and appearance are inferior. The two types of material have distinctly different compositions and setting mechanisms. Ordinary glass ionomer cement when set is the product of an acid-base reaction between a basic fluoroaluminosilicate glass powder and a polycarboxylic acid, in the presence of water [7]. The setting reaction starts immediately on mixing the two components, the glass powder and the aqueous polyacid solution. The hydrogen ions of the acid attack the glass, releasing metal ions that combine with the carboxylic acid groups of the polyacid, thus cross-linking the polymer chains, causing the mixture to harden [7]. The relatively low mechanical strength, brittleness and high surface wear rate of GICs [8,9], as well as the emergence of better alternative restorative materials, has led to their current usage being restricted to the restoration of cavities of classes III and V, especially in patients exhibiting high caries activity [10]. FRRMs are also essentially two-phase composite materials, a high strength dispersion phase (the particulate filler) and a lower strength polymer matrix [11]. Setting of FRRMs is due to a free radical addition polymerization of methacrylate-based monomers, now

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principally initiated by exposure to visible light. FRRMs, unlike GICs, are currently used in the restoration of virtually all types of cavity.

Attempts to combine the desirable properties of FRRMs and GICs led to the development of so-called resin-modified glass ionomers [12] and more recently to that of 'compomers' [13]. This term is derived from COMPOsite and ionoMER, the material supposedly being, or suggesting, a combination of FRRM and GIC technologies [10]. Compomers contain the essential components of GICs but do not permit the acid-base setting reaction [5], indeed no such setting reaction should occur because of lack of water [14]. It is claimed that compomers, in contrast to FRRMs, are designed to be water tolerant in that they are 'required' to absorb water to enhance setting of the material through the acid-base reaction after the first-occurring polymerization of the resin [15]. This claim is maintained by manufacturers such as Dentsply, whose recent product Dyract AP is said to be for all classes of cavities, and 'specifically recommended for use in stress bearing occlusal surfaces', apart from Class I and II cavities of width greater than 2/3 of the intercuspal distance until continuing studies confirm 'unrestricted use' [16,17].

The aim of the present study was specifically to test the claims made of a compomer's ability to withstand an aqueous environment, and suitability for use in stress bearing areas, in comparison with representatives of the classes of materials from which compomers are notionally derived, FRRMs and GICs. This was done through evaluation of the water sorption, flexural strength and flexural modulus of elasticity after exposure to an artificial saliva.

2. Materials and methods

A compomer, Dyract AP (D-AP) (batch 9806001378, shade A3), a glass ionomer cement, ChemFlex (CF) (batch 9810000350, shade A3) and a filled resin restorative material, SureFil (SF) (batch 980519, shade A) (all Dentsply, Addlestone, UK) were studied. Bar specimens ($26 \times 1.5 \times 1.0$ mm^3) of each material were fabricated using a knife-edged split aluminium mould (alloy 6061) (Fig. 1). The intentions for using this design were:

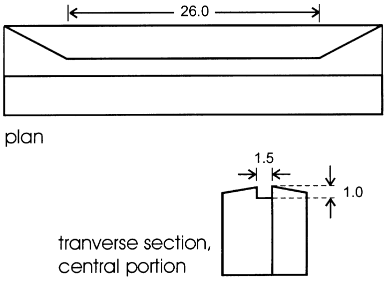

Fig. 1. Principle details of knife-edge split mould used for fabricating test specimens.

1. One-shot preparation of specimens ready to be tested without further finishing.
2. To ensure that the bottom surface of the specimens was cured sufficiently in a single exposure. Based on findings from previous work [18], 1 mm was chosen.
3. To fabricate specimens whose width and span met the assumptions of the derivation of the flexural strength and elastic modulus equations for the three-point bend test [19]. A slot of 1.5 mm breadth and 26 mm length was considered appropriate for a span of 16 mm.
4. A split mould was used to minimize stresses exerted on the specimens during their retrieval.
5. The knife-edge permitted an insignificant flash of excess material at the specimen edges and ready separation from the overflow.
6. To allow filling of the mould with minimal risk of air entrapment and resistance to flow, the ends were open and the side walls were divergent.

For SF and D-AP, excess material was placed into the rectangular slot of the mould, covered with a Mylar strip, backed by a glass microscope slide, and placed under a small load in a levelling press (Leitz, Wetzlar, Germany) for 3 min to permit flow to come to an end. Removing the load and slide, specimens were cured by placing the tip (8.0 mm diameter) of the light guide in contact with the Mylar strip and irradiating each specimen at five overlapping spots for 40 s per spot with a visible light curing lamp (Luxor, Imperial Chemical Industries, Macclesfield, UK). The light intensity was estimated at $405 \pm 5\ \mathrm{mW \cdot cm^{-2}}$ (Demetron 100, Demetron, Danbury, CT, USA). For CF, the liquid and powder were mixed according to the manufacturer's instructions to form a paste which was quickly placed into the mould. The material was allowed to set under the load of the levelling press for 5 min.

The mould was then opened and specimens retrieved using great care to avoid bending stresses. Even so, GIC specimens showed a slight tendency to curl on standing after removal from the mould, so were sandwiched between two glass slides and under a load of about 0.2 N for 1 h. Specimens were inspected on retrieval from the mould; any showing evident defects were discarded and replaced. Forty specimens of each material were thus prepared, randomly distributed into eight groups of five. For each material, one group was used as an untreated control (UC), i.e. exposed only to the laboratory environment (23°C, 50% RH), one group was exposed to water vapour (WV) at 100% relative humidity (RH) (i.e. saturated air over deionized water), one to low humidity (LH) air, ~22% RH (i.e. laboratory air raised to 37°C), and the remaining five groups were immersed separately in artificial saliva (AS) [20] at pH6; all treatments except UC were at 37°C, commencing 24 h after preparation. The AS was replaced every 5 days.

Water sorption was monitored gravimetrically frequently until saturation and at various times thereafter. The initial

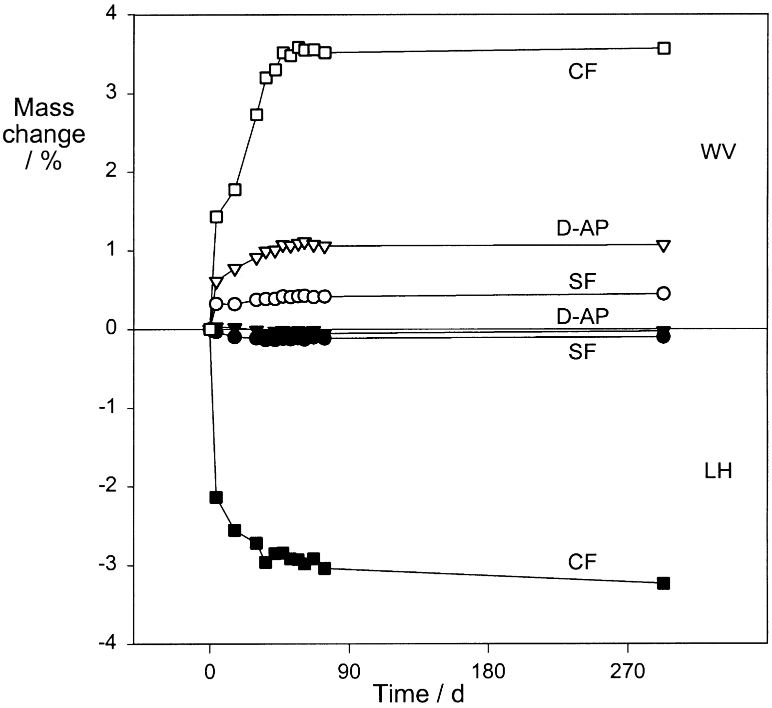

Fig. 2. Percentage mass change on exposure to water vapour at 100% RH (WV) [open symbols] and low humidity (22% RH) (LH) [solid symbols] at 37°C for ChemFlex glass ionomer cement (CF), Dyract-AP compomer (DAP) and SureFil filled resin restorative material (SF).

mass values (m_1) were obtained as soon as preparation was complete. Those specimens immersed in AS were retrieved with tweezers, blotted with a filter paper until free from visible moisture and waved in the air (23°C, 50% RH) for 5 s before being weighed as a group to a precision of 0.01 mg using a digital balance (H542, Mettler, Greifensee, Switzerland). New mass values (m_2) were recorded. Water sorption, as a percentage mass change, was then calculated from $100[(m_2 - m_1)/m_1]$.

Specimens were tested at 23°C in three-point bend using a universal testing machine (1185, Instron, High Wycombe, UK), at $0.50\ \mathrm{mm \cdot min^{-1}}$ cross-head speed, to determine the flexural strength and flexural elastic modulus. For the control group (UC) this was done at 24 h; for the AS groups this was done wet at about 0.5, 1, 3, 6 and 9 months after preparation; for the WV and LH groups, this was at about 9 months immediately after weighing. The polished support rods were of diameter 2 mm set 16 mm apart; the central load was applied with a rod also of diameter 2 mm. Data acquisition was done in software (LabVIEW version 5.1, National Instruments, Austin, TX, USA) on a PC, as was the programmed calculation of ultimate flexural strength, F [21]:

$$F = \frac{3PL}{2bd^2}\left[1 + 6\left(\frac{\Delta y}{L}\right)^2 - \frac{4d\Delta y}{L^2}\right]$$

and flexural modulus of elasticity, E:

$$E = \left(\frac{\Delta P}{\Delta y}\right) \times \frac{L^3}{4bd^3}\left[1 + 2.85\left(\frac{d}{L}\right)^2 - 0.84\left(\frac{d}{L}\right)^3\right]$$

where P is the load to fracture, L the support span, b and d the specimen breadth and depth, and $\Delta P/\Delta y$ the gradient of the (steepest) linear portion of the load-deflection curve [19]. The breadth and depth of each specimen was checked, on the two pieces, immediately after testing and the average value was used for the calculations.

All treatment times were recorded to the calendar day. Statistical calculations were done in software (SigmaPlot v6, SPSS Science, Chicago, IL, USA). Student's t-tests were done on specific selected within-material comparisons. To avoid confusion, no arbitrary corrections were applied to correct probabilities for multiple testing as either results were entirely unambiguous or no decisions hinged on the outcome: even the most conservative protection would not alter the conclusions.

3. Results

The mass variation in WV and LH is shown in Fig. 2. In each case, at least 60 days was required for an approach to an equilibrium value with marked differences evident both between materials and between conditions, although the initial change rate was very rapid.

All three materials rapidly reached a more or less stable mass (nominal saturation) on immersion in AS in 3–5 days, there being a clear distinction between them: SF 0.17%, D-AP 1.1% and CF 6% (Fig. 3). After this point, the mass for D-AP showed a slow and slight continued increase up to 100 days, with a slow decline thereafter. The AS value at

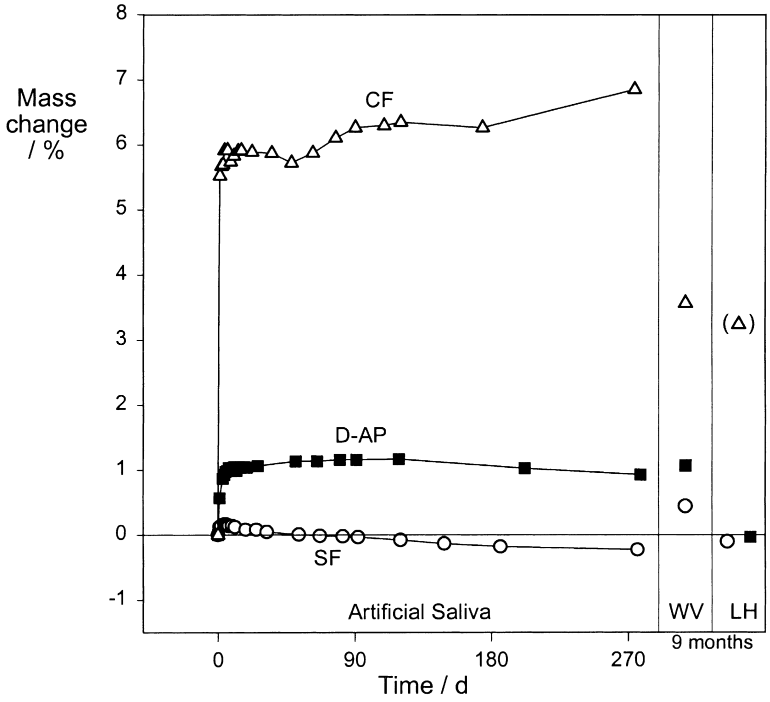

Fig. 3. Percentage mass change on exposure to artificial saliva. Key and conditions as for Fig. 2 (except symbols). WV and LH values at 9 months from Fig. 2. Point in parenthesis for CF represents a negative value, i.e. mass loss.

119 days (1.2%) was very similar to that obtained in WV at 9 months (1.1%). On the other hand, D-AP in LH was essentially unchanged at 9 months at 0.03%. For SF there was a gradual but progressive decline to −0.2% at 9 months while in WV at that time a gain of 0.4% was recorded, compared with −0.1% in LH. The mass changes for CF were less stable but with a tendency to increase. In WV only about half the AS gain was found at 9 months, i.e. 3.6%, while in LH a loss of about 3.2% was found.

The UC strength values for SF ($P = 0.43$) and D-AP ($P = 0.17$) were not distinguishable from the first AS results, but CF showed a substantial fall on immersion ($P = 3.5 \times 10^{-4}$) (Fig. 4). In AS, the patterns of behaviour varied. For SF, no trend is detectable for the duration of the immersion ($P = 0.26$), but the curvature of the plot for CF is significant (quadratic term, $P = 0.026$), with a maximum at about 3 months. After its maximum at 1 month, DAP showed a sharp decline up to 6 months ($P = 6 \times 10^{-7}$); the 6 and 9 month values were not distinguishable ($P = 0.20$). In Fig. 4, the value for the tensile strength of dentine is indicated [22] as no direct flexural strength value seems to have been reported, although because failure is initiated in tension in that mode the values are expected to be comparable.

The WV and the final AS strength values were statistically indistinguishable for SF ($P = 0.97$), but the LH value was significantly greater than that for AS ($P = 1.5 \times 10^{-4}$), i.e. by 16 MPa or some 13%. CF in WV was not different from AS ($P = 0.159$), but in LH was substantially stronger ($P = 1.5 \times 10^{-5}$), i.e. by more than a factor of 3. While D-AP in WV was stronger by some 19 MPa than in AS at 9 months ($P = 0.0033$), in LH the difference was increased to 70 MPa ($P = 4 \times 10^{-6}$), the value being similar to that seen at 1 month in AS ($P = 0.037$).

For flexural elastic modulus, none of the UC values are distinguishable from the first value obtained in AS: CF, $P = 0.47$; SF, $P = 0.23$; D-AF, $P = 0.39$ (Fig. 5). The apparent steady decline for CF from the start does not attain significance under any of various models ($P > 0.05$), although for both D-AP ($P = 0.017$) and SF ($P = 0.0025$) the increase at 1 month from the first recorded value is significant. However, while for SF the value was clearly constant from then on, for D-AP it just as clearly diminished (quadratic regression, $P = 1.1 \times 10^{-4}$). The value for CF in WV was significantly higher than for AS at 9 months ($P = 0.0056$), and the LH value much more so ($P = 6.6 \times 10^{-4}$), but although this distinction against the AS value could not be made for SF in WV ($P = 0.25$), for SF in LH it was plain ($P = 3.6 \times 10^{5}$). Both comparisons were significant for D-AP: in WV, $P = 0.0052$, in LH, $P = 1.9 \times 10^{-6}$. In Fig. 5, some reported values for the dynamic and static elastic moduli for dentine in flexure are indicated for comparison [23].

4. Discussion

The implicit sensitivity of the materials tested here to environmental humidity (Fig. 2) is of great import for testing protocols. Plainly, it cannot be assumed that dry storage

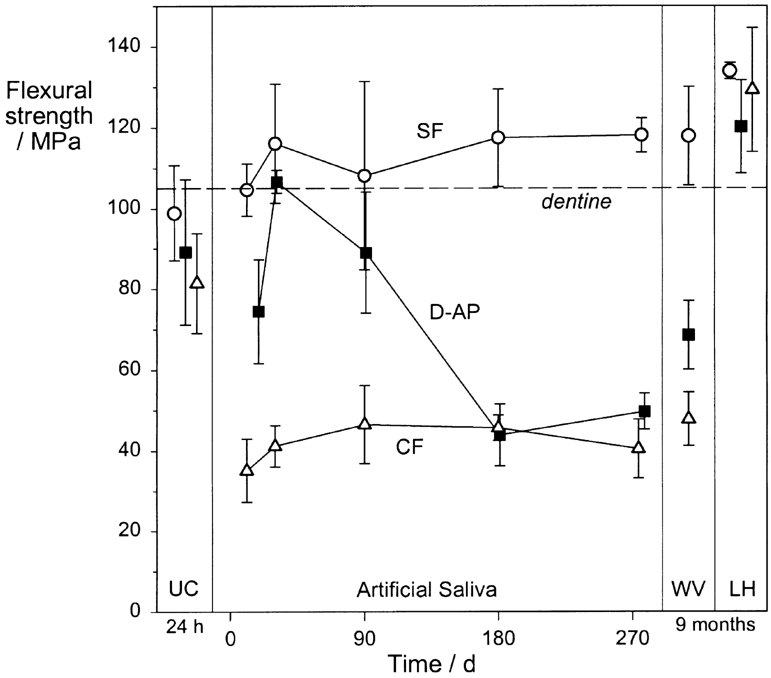

Fig. 4. Flexural strength (three-point bend). Key and conditions as for Fig. 3 except UC: untreated control (50% RH, 23°C, at 24 h). Broken line represents value of tensile strength for dentine, shown for comparison [21].

is unassociated with change, yet equally the presence of water vapour in the surroundings with a potential greater than in the material must lead to its absorption. While this has been known for a long time in the polymer industry, it does not seem to be generally appreciated in dentistry, where the tacit assumption appears to be that contact with liquid is essential. These implications will need careful consideration in many contexts.

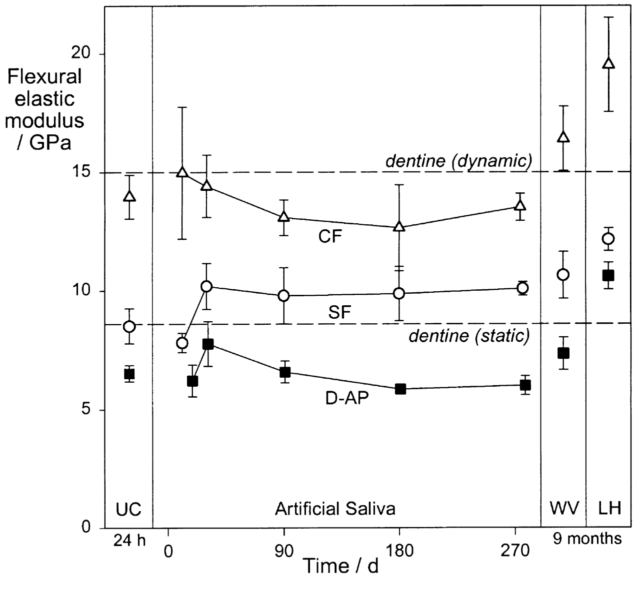

Fig. 5. Flexural modulus of elasticity (three-point bend). Key and conditions as for Fig. 4. Broken lines represent values of static and dynamic flexural elastic moduli for dentine, shown for comparison [22].

The observation that CF had the highest water sorption value in AS (6–7%) was no surprise and agrees with other workers' findings [24]. Glass ionomer cements (GICs) are hydrophilic [25] and absorb water that continues to take a mediating role in the acid-base reaction, the process important for the development of the material's properties. The low value for SF is also reasonable for an FRRM with a substantial filler loading. The intermediate position of D-AP could be attributed to the generally greater volume fraction of resin in compomers [26], but this seems inadequate as an explanation for a seven-fold increase over the FRRM. Instead, an alternative explanation such as hydration of the acid-soluble glass core is more probable as well as the necessarily greater solvation of the more polar resin.

The very small loss of mass for SF in LH is indicative of the presence of little volatile material, thus the loss in AS is further confirmation of the 'corrosion' of FRRMs [27,28] but under what might otherwise be considered benign conditions. The importance of this loss is amplified by the gain shown in WV, such that the actual dissolved mass in AS is implied to be of the order of 0.54% in 9 months, although on this timescale and for this sample size there is no detectable effect on strength beyond that of the water itself (Fig. 4). The corresponding discrepancy for D-AP is 0.14%, although the similar slopes to the curves from 4–9 months suggest that a similar process is occurring. The great loss of mass seen for CF in LH was also not unexpected, but it is noteworthy that this was not associated with deterioration in either strength or stiffness, in fact quite the converse, very high values being obtained for both properties (Figs. 4 and 5). However, the WV absorption by CF was surprisingly low in comparison with the value obtained in AS, suggesting that other processes are at work due to the chemistry of the AS or the physical presence of liquid water and thus the dissolution of material that affects subsequent sorption. That there are several concurrent processes occurring at different rates is suggested by the complicated form of the CF in AS curve, but further work is required to tease these out.

The gradual increase in the flexural strength of the GIC, taking about 3 months to reach a maximum, may be attributed to its low rate of setting [9,29,30], the reaction may continue over a period of weeks or even months [31]. The dramatic decline in strength on immersion in AS is of great importance, suggesting marked surface deterioration. On immersion, apart from dissolution effects, the high osmotic potential of the matrix leads to the large absorption of water (Fig. 3), so a decline in flexural modulus of CF would therefore be the predicted natural consequence, but unconfirmed with these data. Whether this is the case needs further more detailed study with greater sensitivity.

Compomers differ from GICs in at least two respects: first, the glass particles are silanated to bond covalently the particles to the polymer matrix; and second, the solid matrix is formed primarily as a result of photopolymerization [14]. The monomers incorporated in compomers are essentially modified methacrylates, for example urethane dimethacrylate, as well as bifunctional monomers carrying carboxylic acid groups as well [14]. These are therefore capable of reacting via free radical addition polymerization and, in principle, with cations that may be liberated from the glass core via an acid-base reaction. However, to date, the exact mechanism of setting of compomers still seems to be poorly understood. Nevertheless, since compomers contain no water in their composition [14] and cannot set without exposure to light [5], the initial setting of the material is due to photopolymerization. Any contribution of the acid-base reaction to overall setting must occur after exposure of the material to water. Were this to be the case, the conventional prediction would be that this exposure, i.e. in saliva, should lead to an improvement in mechanical properties (and indeed this appears to be the design principle of this class of materials), despite the possible countereffects of dissolved water as a plasticizer from the point of view of the resin. Thus, it might be tempting to attribute the observed initial increase in flexural strength of D-AP (Fig. 4) to this acid-base reaction, but the possible contribution of post-irradiation polymerization to this increase further complicates the system. However, the fact that mere exposure to WV caused a substantial decline from the LH value (Fig. 4) undermines the argument that exposure of compomers to water improves their mechanical properties, even if the acid-base reaction occurs to any extent.

It might be argued that in water vapour alone insufficient water was available for the acid-base reaction to occur properly. However, the fact that in AS the strength further declined to about 50 MPa suggests hydrolytic damage. This may in part be attributed to the pH of the medium resulting in a more aggravated attack on the glass. This is consistent with observations of a higher fluoride release from compomers in an acidic than in a neutral environment, which effect was also attributed to enhanced hydrolytic degradation [32,33]. It is to be noted that the activity (chemical potential) of water in saturated vapour is necessarily identical to the activity in the liquid at the same temperature, so the ultimate water sorption must be identical in the two circumstances in the absence of other effects. The AS has too low a salt concentration to affect the water activity appreciably, it can only be very slightly lower. Hence, the water vapour test gives an unambiguous result: water is detrimental to D-AP, AS more so.

If indeed D-AP, or any compomer, has a silanated glass powder, it is difficult to understand the rationale for it, on purely chemical grounds. Thus, if it is intended that the glass be hydrolysed to provide a source of cross-linking ions for the acidic moiety of the resin, then it is a necessary consequence that the bonding of matrix to core is compromised if not actually destroyed. The siliceous gel known to be formed [34] is necessarily weak as an anchorage for such attachment, assuming it survives reaction, in any case weaker than such a bond to a glass (although even this is subject to hydrolytic breakdown with time [27,28]). It is this bonding, and the consequent matrix constraint, that is the fundamental basis of all composite materials' design where an improvement in stiffness and strength is desired.

In fact, it is difficult to understand the rationale for a compomer in the first place. An FRRM has a fully covalently bonded matrix, where the C–C bond energy is about 350 kJ/mol. Ionic coordination bonds can only be about one tenth as strong as this at best, similar to the strongest hydrogen bonds (whose range is roughly 4–40 kJ/mol). Large metal cations could only diffuse through the resin matrix at an appreciably useful rate if this matrix were particularly hydrophilic and contained a sufficient proportion of water dissolved in it to solvate those ions. This water must however cause the material to swell, to soften, and to weaken. It is therefore necessary that any additional cross-linking compensates for this diminution in properties.

Thus, whether or not any acid-base reaction occurs in a compomer, it is not expected to be associated with an improvement mechanically for reasons of matrix-core bonding degradation, water sorption plasticizing effects, and weaker cross-links as well as reduction in the volume fraction of the original glass—which is harder and stiffer than the acid-base reaction products.

In the light of this analysis, it is not difficult to explain the dramatic fall in flexural strength seen for D-AP after only 1 month of exposure to the AS (Fig. 4). For comparison, in trying to explain the chemistry of resin-modified glass ionomer, it has been suggested that it may undergo a phase separation to yield hydrophobic and hydrophilic domains [35]. The water is envisaged as migrating preferentially to the hydrophilic sites, causing localized dissolution and leakage of ionic species, with consequent property changes [36].

However, it is to be noted that resin-modified GIC remains a primarily aqueous, acid-base reaction setting system. It might be of interest to establish whether an equivalent process occurs in compomers, but it appears to be a secondary issue in the face of the points made above. Even so, it is of some interest to establish whether the present observations are unique to D-AP or, as seems to be expected [37], represent behaviour typical of compomers.

It is considered desirable that both the flexural strength and elastic modulus of a restorative material matches that of the tissue it replaces [38,39]. The flexural strength of SF compares very well with that of dentine, while its flexural modulus, although lower, is within the likely appropriate range and sufficiently stable (Figs. 4 and 5) and so it might be an appropriate material for use in stress-bearing areas. Although the observed loss in mass for SF may be attributed to leaching of filler and uncured components of the resin, in agreement with previous studies [27,28,40–42], this was not reflected in a deterioration in mechanical properties over the 9 months of the study (Figs. 4 and 5). In contrast, the flexural strength of CF in AS was low in comparison with dentine and, as is commonly recommended [1,43], this type of material is not suitable for load bearing. The rapid decline in flexural strength seen for D-AP, however, and its ultimately low value in comparison with dentine (Fig. 4), as well as its low, progressively declining modulus of elasticity, makes it least suitable in this context, particularly on a long-term basis.

Further crucial points emerge from this study. Firstly, the inappropriateness of testing any material stored under conditions that do not relate to the service environment is clear if results are to be at all interpretable. Indeed, if only the UC or LH values of strength were inspected (Fig. 4), one might conclude that all three materials tested here were essentially indistinguishable and quite satisfactory in comparison with the dentine value as a criterion. That this would be a false conclusion is plain. Although the possible effect on flexural modulus is not so marked (Fig. 5), it is nevertheless obvious that erroneous conclusions could be drawn. Secondly, the use of water alone as a means of mimicking oral conditions emerges as inadequate. It is evident that the chemistry of the oral environment must be taken into account, although the effects differ according to the type of material being tested. Thirdly, results depend on exposure time. Although there is no indication of a single, universally acceptable time for testing (nor is it clear than one could exist), it is certainly the case that early values may not reflect later behaviour. Testing at multiple times is therefore indicated to avoid surprises and to detect trends. There is no evidence in the literature that these points are generally appreciated.

5. Conclusion

The behaviour and properties of D-AP do not support the application of the material in stress bearing areas, as is currently recommended by the manufacturer. The present data indicate an early and progressive deterioration of the material. This and theoretical considerations necessitate a re-examination of the assumed chemistry of setting and the design principles of compomers as a class, as well as reconsideration of their suitability as restorative materials or, at the very least, the indications for use.

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