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Are inert glasses really inert?

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ARTICLE INFO

Article history:

Received 7 September 2022

Received in revised form

8 September 2022

Accepted 8 September 2022

Keywords:

Inert glass

Degradation

Leaching

Ion release

ABSTRACT

Objectives: The aim of this study was to investigate the degradation of inert glass fillers which are commonly used in conventional resin-based composites to provide radiopacity, reduce the polymerization shrinkage and improve the mechanical properties.

Methods: 75 mg of five different glass powders (1 μm) was immersed separately into 50 mL of acetic acid (pH 4) and tris buffer (pH 7.4) for up to 4 weeks. At each time point the glass powder was filtered and dried for characterization using ATR-FTIR and XRD to assess the degradation behavior and crystallization. ICP-OES, ISE and pH measurements were performed on the supernatant solutions to monitor the pH and ion release.

Results: Although FTIR and XRD analysis showed no significant glass degradation or crystallization upon immersion, there was a substantial release of ions from the inert fillers, especially from BABFG and CDL. Barium release for these fillers were 270 and 165 ppm respectively. G018–373 glass presented the lowest ion release followed by GM27884 and BABG. The ion release was more pronounced in acidic conditions compared to neutral conditions apart from the fluoride release.

Significance: Inert glasses are not as inert as previously thought. This may result in leaching of ions, potentially causing toxicity, reduction in mechanical properties, increased wear and subsequent failure of the composite material. The ions released from the inert glass may interfere with other glass fillers such as bioactive glass fillers, inhibiting degradation of the bioactive glass, beneficial ion release from the bioactive glass, pH neutralization and apatite formation.

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1. Introduction

Structural fillers have been added into resin-based composites (RBCs) since the 1950s, where quartz fillers were incorporated into a methylmethacrylate based resin matrix to reduce the high polymerization shrinkage and thermal

expansion coefficient, improve the mechanical properties and esthetics [1]. However, quartz is not radiopaque, has a low refractive index and a high hardness, so can cause abrasive wear to the tooth. Therefore, silica-based glass fillers such as barium or strontium based aluminoboro-silicates are now more commonly employed [1,2]. The addition of heavy metals such as barium or strontium in the glass structure provides radiopacity, and these glasses have a higher refractive index in the range of 1.49–1.57, making it easy to

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Table 1 – The compositions, D₅₀ particle sizes, silane levels and refractive indices of the five inert glasses being investigated.

Inert Glass Name	Approx. Composition / mol%	Particle Size	Silane Level	Refractive Index
BABFG	52.9 % SiO ₂ , 14.8 % BaO, 9.4 % B ₂ O ₃ , 5.3 % Al ₂ O ₃ , 17.6 % F	1 µm	3–4 %	1.531
BABG	66.29 % SiO ₂ , 17.15 % BaO, 10.30 % B ₂ O ₃ , 6.25 % Al ₂ O ₃	1 µm	3–4 %	1.553
GM27884	69.34 % SiO ₂ , 12.35 % BaO, 10.88 % B ₂ O ₃ , 7.43 % Al ₂ O ₃	1 µm	4.6 %	1.528
G018–373	80.98 % SiO ₂ , 1.36 % Al ₂ O ₃ , 6.20 % CaO, 1.23 % Cs ₂ O, 0.74 % K ₂ O, 1.07 % La ₂ O ₃ , 2.24 % Na ₂ O, 3.35 % SrO, 2.82 % ZrO ₂	1 µm	2.9 %	1.530
CDL	61.67 % SiO ₂ , 11.22 % BaO, 9.78 % B ₂ O ₃ , 6.68 % Al ₂ O ₃ , 6.60 % F, 4.05 % Na ₂ O	1 µm	3.4 %	1.525

match with resins to improve depth of cure and provide good esthetics of the composite material.

Barium alumino-boro-silicate glasses are now the most widespread particulate fillers used in dental composites. These glasses are silylated with methacrylate functional silanes in order to chemically couple the glass particles to the resin. This improves the mechanical properties, particularly the flexural strength and reduces the abrasive wear behavior [3]. Over the last thirty years there has been a progressive move towards smaller glass particle sizes, although some materials still use particles of 2–10 µm in conjunction with nanosized fillers. These glasses are highly crosslinked since they contain mainly network formers such as boron oxide, silicon dioxide and aluminum oxide which is an intermediate oxide. This suggests that these types of glasses are likely to be resistant to degradation under physiological conditions so are often referred to as “inert glasses” [3–7]. However, there is lack of evidence to how ‘inert’ these glasses are. Therefore, if these glass fillers are degradable and are not inert, this could potentially result in the release of cytotoxic ions, wear of the composite material as well as reduction of the mechanical properties, which are crucial when designing a dental composite.

Early studies by Soderholm et al. [8] in the 1980s did indicate that there was some degradation and ion release in distilled, then further investigated this in artificial saliva alongside distilled water [9,10]. However, deionized water tends to be unstable and this study did not consider the leachability in acidic conditions, which are crucial in the oral environment. In these pioneering studies there is often little information on the glass compositions or their particle size. It is likely that the particle size was significantly larger than those present in modern day composites. Moving to smaller particle sizes with their increased surface area is likely to result in much faster glass degradation.

One of the significant motivations for the present study is the increasing interest in degradable bioactive type glass in composites. In many of these studies mixtures of inert glasses and bioactive/reactive type glasses have been used and in some of these studies it has been assumed that all of the ion release comes from the bioactive glass. Al-eesa et al. found for example a small amount of fluoride release from an inert glass composite of approximately 10, 15 and 4 ppm in TB, AS7 and AS4 respectively [4]. In this study however other elements from the inert glass were not measured since it was assumed there would be none. Similarly, a study by Tiskaya et al. measured only the calcium, phosphate, silicon and

fluoride release from a commercially available dental compomer even though the composition of this material also contained a barium alumino-silicate glass [11].

Since the only studies that have previously investigated the degradation of “inert” glasses were performed in alkaline conditions, this present study investigates the degradation behavior of four barium alumino-boro-silicate “inert glasses” and one lanthanum silicate glass in both acidic and neutral media. All of the glasses were silylated by the suppliers and have a nominal particle size 1 µm. The lanthanum containing glass was included in the study since the manufacturer claims this glass to be more chemically stable than conventional barium alumino-boro-silicate glasses and has been labeled in their brochure as “dental glass resist”. In the present study, the dissolution behavior of inert glass powder was investigated by immersing into tris buffer at pH 7.4, rather than distilled water (since it has a more consistent pH) and into acetic acid buffered at pH 4.0 to mimic more aggressive acidic conditions associated with dental plaque or consumption of an acidic drink. The most reactive glass that showed the highest ion release was also investigated in the form of composite disks and compared to a composite containing a less degradable glass.

2. Materials and methods

Five inert glasses were used to investigate their inertness. BABFG and BABG were manufactured by Ferro and Esstech Inc. (Table 1) respectively, GM27884 and G018–373 were manufactured by Schott, where the latter is the lanthanum silicate glass, and the CDL labeled glass was manufactured by Cera Dynamics Ltd. All glasses contained a silane level between 2.9% and 4.6%.

2.1. Preparation of the immersion media

Acetic acid (AA) and tris buffer (TB) solutions were used for the powder immersion study to mimic acidic and neutral conditions respectively. To prepare 2000 mL of 0.1 M AA, 11.44 mL of 1 M acetic acid was added to 800 mL deionized water and stirred using a magnetic stirrer. The pH was adjusted to 4.00 using KOH (Sigma-Aldrich) and the solution was diluted using deionized water to a total volume of 2000 mL and stored in an incubator at 37 °C. To prepare the TB, 15.090 g of tris(hydroxymethyl) aminomethane (Sigma-Aldrich) powder and 44.2 mL of 1 M HCl (Sigma-Aldrich) was

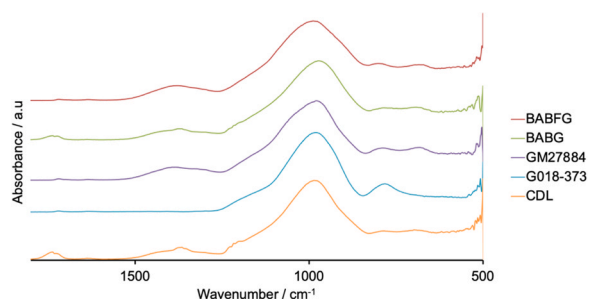


Fig. 1 – The ATR-FTIR spectra of the five glasses before immersion.

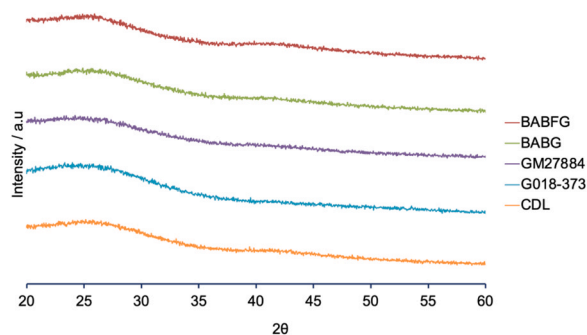


Fig. 2 – The XRD diffraction of the five glasses before immersion.

added to 800 mL of deionized water and stirred using a magnetic stirrer. The mixture was then placed in an incubator at 37 °C overnight. The pH of this solution was adjusted to 7.30 the following day using 1 M HCl and was diluted using deionized water to a total volume of 2 L and stored in an incubator at 37 °C.

2.2. Immersion study

2.2.1. Powder immersion

75 mg of the five glasses ($n = 5$) were immersed in duplicates into centrifuge tubes containing 50 mL of TB ($n = 70$) and AA ($n = 70$) separately, equating to a total of 140 tubes. The tubes containing the mixture was placed in a shaking incubator at 37 °C for multiple timepoints: 3 h, 6 h, 1 day, 3 days, 1 week, 2 weeks and 4 weeks. At each timepoint, the mixture was filtered using filter paper and placed on a petri dish and dried overnight in incubator at 37 °C and stored in sealed polyethylene bags in a desiccator for characterization. The supernatant solution was kept for ion release and pH measurements.

2.3. Characterization techniques

2.3.1. Powder characterization

The dried glass powder was characterized before and after immersion using attenuated total reflectance-Fourier transform infrared spectroscopy (ATR-FTIR, Perkin Elmer, Frontier FTIR, Massachusetts, USA) and X-Ray diffraction (XRD, Panalytical, X'Pert Pro) to observe glass degradation and crystal phase formation respectively.

2.3.2. Solution immersion

The supernatant solution was analyzed using an Ion Selective Electrode (ISE, ELIT 221, Nico2000 Ltd, London, UK) at each time point to quantify the fluoride release from the glasses that contain fluoride (BABFG and CDL). Inductively coupled plasma-optical emission spectrometry (ICP-OES, Thermo Fisher iCAP 7400 Duo, Massachusetts, USA) was used to detect the release of silicon, barium, boron, aluminum, calcium, sodium, lanthanum and strontium ions. pH measurements (Seven2Go Pro pH meter, Mettler-Toledo Ltd, Leicester, UK) of the supernatant solutions were measured, to detect any changes in pH after immersion of the glasses.

3. Results

3.1. Powder immersion study

3.1.1. ATR-FTIR

The FTIR spectra of the five original glasses (Fig. 1) shows distinct vibrations found at 975 cm^{-1} , which corresponds to the Si-O-Si and Si-O-B in the glass structure of these glasses [12]. The signals at $780\text{--}790\text{ cm}^{-1}$ also correspond to Si-O-Si bonds. All glasses presented additional signals at 670 cm^{-1} and 1400 cm^{-1} (BO_3 vibrations) apart from the G018–373 since this glass does not contain boron and lacks this signal. No changes were observed after immersion and no crystalline phases were detected.

3.1.2. XRD

The XRD patterns of the five original glasses (Fig. 2) shows a halo at approximately 30° suggests that all the glasses were amorphous. No changes were observed after immersion and no crystalline phases were detected.

3.1.3. Ion release

The silicon release data (Fig. 3) suggests that for every glass the Si release was greater in AA than in TB apart from the G018–373 glass which had a higher Si release in TB than AA. BABFG and CDL released significantly more Si in AA than the other glasses with G018–373 releasing the least. In TB, G018–373 and CDL glasses released the most Si, whereas GM27884 and BABG released the least. The release of Si both in TB and AA is approximately linear with square root time suggesting a diffusion controlled release according to Fick's Law. The slope of the regression line corresponds to the rate of degradation for each glass where the highest degradation rate was observed for the BABFG and CDL glasses in AA. The lowest degradation rate was observed for BABG in TB and G018–373 in AA.

Ba release was observed to be greater in AA than in TB (Fig. 4). There was substantial Ba release from the BABFG glass of up to 275 ppm and 86 ppm in AA and TB respectively. CDL released the next most Ba in AA followed by BABG and GM27884 (164, 102 and 34 ppm respectively). G018–373

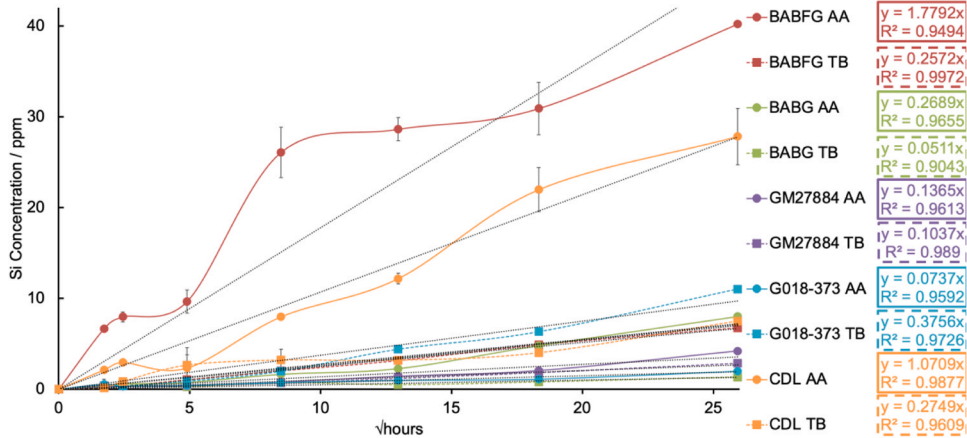


Fig. 3 – The silicon concentration in solution of the glasses upon immersion into AA and TB. The linear regression lines are plotted in black dashed lines and the equation of the line and R² values are stated on the right side of each legend.

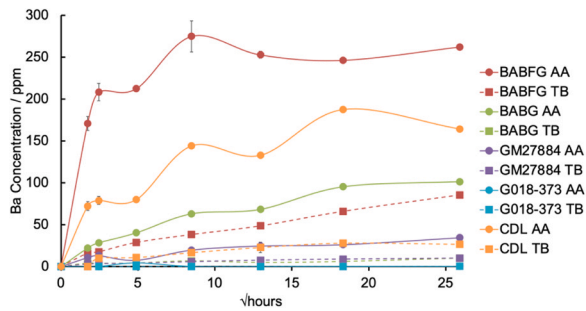


Fig. 4 – The barium ion concentration in solution of the glasses upon immersion into AA and TB.

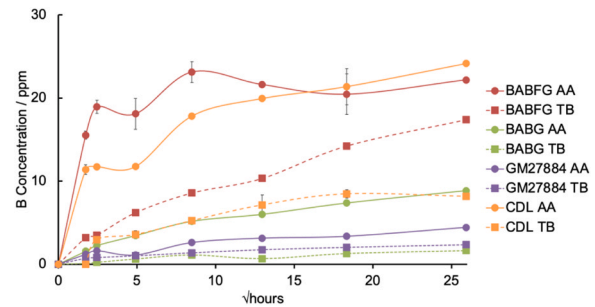


Fig. 6 – The boron concentration in solution of the glasses upon immersion into AA and TB.

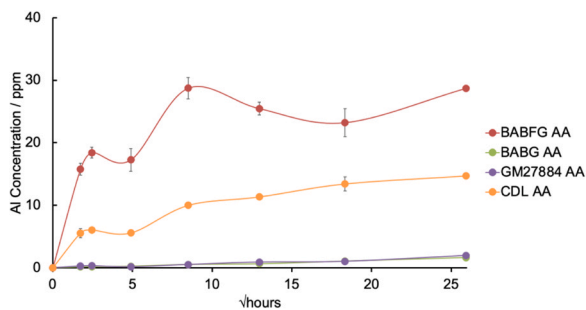


Fig. 5 – The aluminum concentration in solution of the glasses upon immersion into AA and TB.

released virtually no Ba since there was no BaO in the composition of those glasses.

There was no significant Al release with Al being below the detection limit in TB from any of the glasses (Fig. 5). BABFG and CDL released significantly more Al than Schott GM27884 and BABG, whilst G018–373 released no Al in AA.

BABFG and CDL released more B than the other glasses both in TB and AA (Fig. 6). Schott G018–373 released no B

either in TB or AA, since it is not present in the glass composition. B release was much higher in AA than in TB for all glasses.

There was more release of Ca in AA than TB for each glass and G018–373 presented the highest calcium release in both AA and TB up to 11 and 12 ppm respectively, followed by CDL in AA. GM27884 and BABG in TB showed the least Ca release. Fig. 7.

The G018–373 glass released La in AA but there was no release of La found in TB (Fig. 8). No other glass released La since there was no La in their compositions, and the amounts of La released for the G018–373 glass is less than 2 ppm.

G018–373 released significant amounts of Sr in both AA and TB of approximately 6.9 and 7.4 ppm respectively (Fig. 9). There was a small amount of Sr release for GM27884 in AA at 1 day, but overall all glasses showed almost no Sr release apart from G018–373.

The sodium release was highest for CDL in AA and TB at approximately 27 and 14 ppm respectively, followed by G018–373 (Fig. 10). Both of these glasses contain sodium in their glass composition. There was very little sodium release for all other glasses.

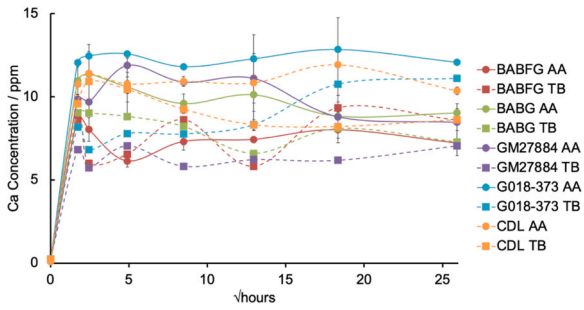


Fig. 7 – The calcium ion concentration in solution of the glasses upon immersion into AA and TB.

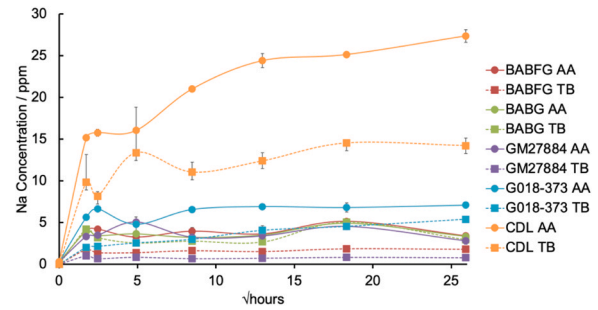


Fig. 10 – The sodium concentration in solution of the glasses upon immersion into AA and TB.

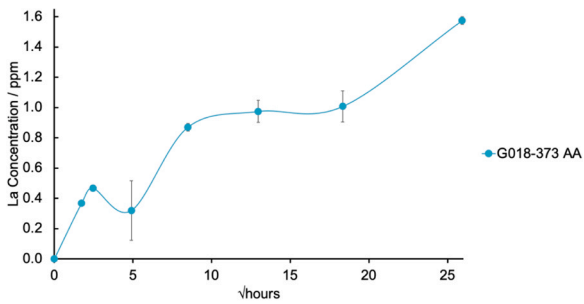


Fig. 8 – The lanthanum ion concentration in solution of the glasses upon immersion into AA and TB.

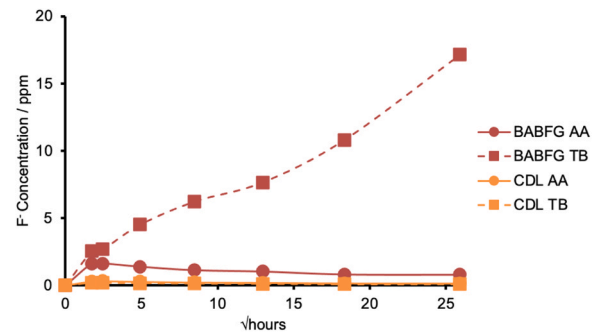


Fig. 11 – The fluoride ion concentration in solution of the glasses upon immersion into AA and TB.

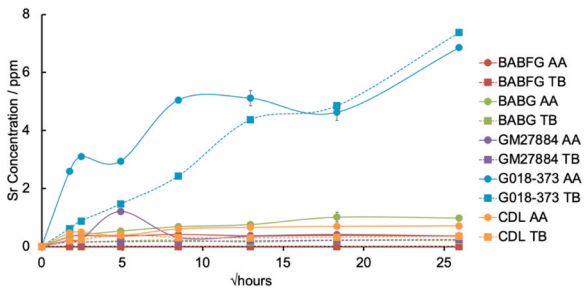


Fig. 9 – The strontium ion concentration in solution of the glasses upon immersion into AA and TB.

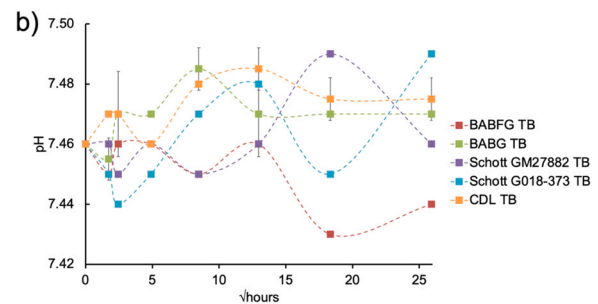
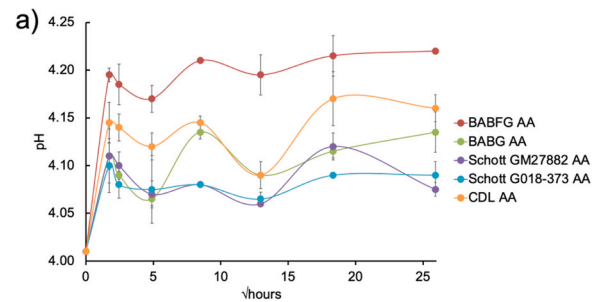


Fig. 12 – The pH changes upon immersion into a) AA and b) TB.

Fluoride release measurements were obtained using an ion selective electrode and BABFG showed the highest free fluoride in solution at approximately 17 ppm, whereas in AA there was less than 1 ppm fluoride (Fig. 11). CDL glass released less than 0.5 ppm fluoride in both AA and TB.

3.1.4. pH

Fig. 12a and b show the pH of the immersion solutions AA and TB for the different glasses as a function of time. There is a small pH rise in AA for all the glasses but even with BABFG that gave the greatest rise the pH increase was only 0.2. The

pH rise correlates with the Ba release in AA. In the case of TB the pH rise was < 0.1 for all the glasses even BABFG that exhibited significant ion release.

4. Discussion

The fact no changes were observed by FTIR and XRD post immersion might indicate little or no glass degradation has occurred. However, the ICP-OES results showed large amounts of B, Al and Ba in solution for all glasses with the exception of G018–373, since this glass composition did not contain BaO and B₂O₃ in the glass composition. This latter glass contained much less Al₂O₃ than the other glasses, so released less Al compared to the others and was much more stable than the other glasses. The release figures for Si, Al, B, Ba, Ca, La, Sr and Na are all greater in AA than in TB for all the glasses investigated. The F release was higher for the BABFG that contains a substantial quantity of fluorine.

In AA unlike TB, the Al-O-Si bonds of the glass network can be acid hydrolyzed in a similar way to that which occurs in aluminosilicate glasses used to form glass ionomer cements [13,14]. This effect not surprisingly is most marked with regard to Al release, where the Al release upon immersion was below the ICP-OES detection limit (0.5 ppm) in TB, whereas significant Al release was observed after immersion in AA. Al₂O₃ is an intermediate oxide and takes part in the glass network it therefore cannot be released without acid hydrolysis of Al-O-Si bonds. The aluminum will form AlO₄ or possibly AlO₃F tetrahedra locally charge balanced by metal cations. Since the dominant metal cation in the current glass compositions is Ba²⁺, it is likely to be the dominant cation charge balancing AlO₄⁻ tetrahedra. Thus, a glass undergoing acid hydrolysis of Al-O-Si bonds will release not only Al³⁺ but also the associated charge balancing cation, Ba²⁺. Therefore, in AA there is not only release of Al but also increased release of Ba, B and Si.

Of particular interest is the BABFG glass which contains 17.6 mol% fluorine (the source of fluorine is unknown) which also releases about 17 ppm fluoride in TB whilst only releasing < 5 ppm in AA, although it would have been expected that F release would be enhanced in acidic conditions. However, similar results by observed by Al-eesa et al. who measured the fluoride release of a composite in TB as well as an acidic medium and found that the release was 2.5 times higher in TB than the acidic media [4]. It is possible the fluoride released is not being detected because its present as a complex rather than as the free ion, or its precipitating from solution most likely as a metal fluoride. It is likely fluorine is added to the BABFG glass not to give fluoride release but to reduce the refractive index, since fluorine is known to reduce the refractive index and to decrease melt temperature [15].

The fact that all the glasses resulted in only small pH increases upon immersion and especially in TB despite significant ion release indicates that a simple ion exchange process, where H⁺ ions are exchanged for metal cations that occurs for soda lime silica glasses is not the dominant mechanism for degradation of the aluminoboro-silicate glasses studied here. Soderholm et al. [8–10,16] explained the leaching process using the model first developed by Charles [17] involving alkaline corrosion which is applicable to soda

lime silica glasses but not to the glasses studied here. Consequently, this researcher did not consider acid leaching as it was viewed that glass degradation took place under alkaline conditions rather than acidic conditions, hence why only artificial saliva and deionised water at pH 6.7 was used. It can be seen here that degradation rates in AA are much faster than in TB, which is supported by previous studies [18].

The high amounts of ion release that occur in AA have some significant implications for current restorative dental composites. Barium is classified as a heavy metal, whilst insoluble barium salts like BaSO₄ are often added to dental products to confer radiopacity. Soluble Ba²⁺ cations have been deemed to present cytotoxic properties in a toxicology report which states that “the Ba²⁺ ion and the soluble compounds of barium (notably chloride, nitrate, hydroxide) are toxic to humans”, suggesting it may have an adverse effect on dental tissues [19]. In addition, the surface of dental composites are often exposed to low pHs associated with dental plaque or acidic beverages in the mouth. Such acidic pHs will result in the potential leaching of the glass filler particles at the composite surface which will result in a consequent loss in strength, hardness and an increase in abrasive wear and roughness [18,20,21].

Conversely, the release of fluoride found with BABFG is significant especially in TB and could potentially protect composites made with this glass from secondary caries. Current testing protocols for measuring the flexural strength of dental composites reflect the ISO standard and measure the strength after only 24 h immersion in water [22]. A similar scenario exists with abrasive wear testing. These tests performed after only short-term immersion times in water would not reflect any significant glass degradation that would occur over a much longer time frames in the oral environment.

The substantial ion release from the BABFG glass in powder form could also affect the degradation behavior when incorporated into a composite. This could result in increased wear when used as a restorative composite and subsequent reduction in mechanical properties over time, leading to fracturing of the composite.

In recent years there has been considerable interest in incorporating reactive fillers such as bioactive glasses and alkasite glasses into resin-based composites in combination with so called inert glasses. It has been implicitly assumed in all the studies to date including our own studies that the ions released come only from the reactive glass and not from the inert glasses here may also be complex interactions between the ions released from inert glasses and bioactive type glasses. In addition, the not so inert glasses may compete for water with the bioactive type glass and retard its dissolution, therefore it is important that in future studies the dissolution of the inert glass fillers is considered.

This paper can be criticized for not measuring the ions released from the glasses when incorporated into a resin-based composite. However, our [supplementary data \(Figure S1\)](#), where composite disks were immersed into physiological solution, demonstrate degradation of the composite

containing BABFG fillers and significant release of both B and Ba, particularly under acidic conditions.

5. Conclusion

Although some ion release can be desirable in the oral environment (e.g., Ca, Sr and B), inert glasses are not as inert as previously thought, particularly in acidic conditions which were not investigated in the past. This may result in leaching of ions, potentially causing toxicity (due to barium release), reduction in mechanical properties, increased wear and subsequent failure of the material. The ions released from the inert glass may interfere with other glass fillers such as alkasite and bioactive glass fillers, inhibiting degradation and beneficial ion release, pH neutralization and apatite formation.

Appendix A. Supporting information

Supplementary data associated with this article can be found in the online version at doi: [10.1016/j.dental.2022.09.004](https://doi.org/10.1016/j.dental.2022.09.004).

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