

# Investigation of the impact of acid reflux on dental cements

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## SUMMARY

Laryngopharyngeal reflux (LPR) is a form of gastroesophageal reflux disease (GERD), where stomach acids travel into the oral cavity due to malfunctioning esophageal sphincters. LPR impacts 20% of adults and 10% of children in the United States. Among those affected, 24% of adults and 98% of children have dental damage due to gastric acid, requiring treatments such as fillings, crowns, bridges, or orthodontic appliances. Since dental cements securing these treatments to teeth may degrade when exposed to stomach acid, their performance should be evaluated in LPR patients. While many studies have focused on dietary acids, the effects of stomach acids have received less attention. This study explored the effects of hydrochloric acid, the predominant stomach acid, at pH 2, 3, 4, and 5 over a 30-day period at 37°C on two widely used dental cements: glass ionomer cement (GIC) and resin cement (RC). We hypothesized that dental cements exposed to acidic environments like those found in LPR would exhibit increased degradation as pH levels decrease. Our results showed that GIC had significantly increased sorption and solubility with decreasing pH, resulting in degradation of its ionic matrix. For RC, our results showed that sorption remained unaffected by pH changes, but solubility significantly increased as pH decreased, with no observable degradation. Our study suggests that RC is more resistant to hydrochloric acid-based decay, making it potentially a better choice than GIC for LPR patients. This study underlines the importance of careful dental cement selection when treating LPR patients.

## INTRODUCTION

Gastroesophageal reflux disease (GERD) results in the backflow of acids from the stomach to the esophagus (1). GERD is a condition that affects millions globally. In 2019, there were around 783.95 million cases of GERD worldwide (2). It is estimated that 20% of adults and 10% of children in the United States alone suffer from the condition (1).

Laryngopharyngeal Reflux (LPR) is a special type of GERD, where stomach acids rise into the throat and mouth due to the failure of sphincters (valves) at both the top and bottom of the esophagus (3). Around 71% of GERD patients have LPR, and more severe or longer-standing GERD increases this risk (4). The predominant acid involved in LPR is hydrochloric acid, which is produced in the stomach and helps break down food (5). The pH values of stomach acid range from 1.5 to 2.0 (6). Though the normal pH value for the oral cavity is very close to a neutral 7.0, the oral pH value

for GERD and LPR patients is typically under 5 (7). Patients experience episodes of gastric reflux both during sleep as well as during waking hours. During sleep, reflux episodes typically last from 15-20 minutes, while they last around 1-2 minutes when awake (8). These episodes lower the pH to below 5 for a period of 60 minutes (8).

The corrosive nature of these stomach acids can dissolve dental enamel, causing erosion, which is the progressive loss of tooth surface, in 24% of adults and 98% of children with LPR (9, 10). Although LPR is primarily linked with GERD, many LPR cases go undiagnosed, such as those temporarily caused by medications (sedatives, blood pressure drugs, anti-depressants, and anti-inflammatories) or by medical conditions (obesity, pregnancy, and hiatal hernia) (3). There is a much higher prevalence of dental erosion in LPR patients (70%) than in healthy individuals (10%) (11).

Alongside LPR, oral diseases afflict around 3.5 billion people worldwide, with many requiring dental treatments (12). These include fillings, crowns, bridges, and orthodontic devices (13). Dental cements perform a critical role in these treatments, binding the prosthetic to the dental surface (14). The most common cements used today are glass ionomer cement (GIC) and resin cement (RC) (14). GIC is commonly used for metal crowns due to its reliable properties for retention, fluoride release, and biocompatibility (15). RCs are ideal for zirconia crowns, due to their superior bonding strength and ability to adhere well to zirconia (16). GIC is obtained as a fluoroaluminosilicate glass powder and a polyacrylic acid liquid (17). When mixed, they set through a series of acid-base reactions to produce a brittle, ionic matrix (17). Under acidic conditions, water and hydrogen ions potentially penetrate the GIC matrix, disrupt ionic bonds, and leach critical ions such as calcium and aluminum (18). As a result, GIC may be vulnerable to degradation in low pH environments such as those found in LPR (19).

RC is formed when dimethacrylate resin compounds solidify after several chemical transformations (20). RC consists of a hydrophobic polymer matrix, typically consisting of Bis-GMA or urethane dimethacrylate, reinforced with inorganic fillers (14, 20). The RC matrix consists of strong covalent bonds and may be susceptible to degradation in acidic environments as well (20). Two key metrics in the evaluation of dental cements are their sorption and their solubility. Sorption is the attachment of one substance to another, while solubility is the degree to which a substance dissolves in a solvent (21, 22). The sorption of dental cements in acidic media can cause accelerated degradation, while the solubility of dental cements determines their clinical longevity and effectiveness (23, 24). The presence of acids in the oral cavity can potentially degrade these cements over time. Currently, studies have

focused on external factors (mouthwashes, foods, beverages) and their impact on dental cements. There are some studies on the effects of acidic environments on dental cements (23, 25, 26). A study reported that dental cements could potentially increase the pH of the acid solution itself (25). A second study found that RC was less soluble than GIC after 91 hours of immersion in hydrochloric acid at pH 3.8 (26), while another reported that GIC exhibited higher sorption and solubility than RC after 30 days of immersion in hydrochloric acid at higher pH levels of 5, 7, and 9 (23). These prior studies suggest that GIC may be more vulnerable to acid-induced degradation than RC. However, there are no studies evaluating the degradation of dental cements for the range of pH values potentially encountered in LPR over an extended duration of time.

Our study aimed to determine how exposure to a range of acidic conditions intended to simulate LPR affects commonly used dental cements, with the goal of facilitating research on improving long-term dental restoration outcomes and treatment durability. We hypothesized that dental cements exposed to increasingly acidic environments will exhibit increased degradation as measured by changes in sorption and solubility, with varying degrees of susceptibility depending on the cement type.

We chose hydrochloric acid with a pH range of 2 to 5 for this study, as this range potentially represents oral cavity conditions in a patient with LPR (6,7,8). All samples were immersed in solutions of differing pH values for a 30-day period to simulate *in vivo* exposure to acid reflux. These samples were then placed in an incubator at a constant temperature of 37°C to simulate the average body temperature in the oral cavity (27). Finally, we assessed the sorption and solubility of GIC and RC at the end of 30 days of immersion in hydrochloric acid solutions of varying pH. Our results confirmed that GIC had increased sorption and solubility with decreasing pH, which resulted in degradation. Linear regression analysis further confirmed that pH values significantly influenced both sorption and solubility ( $p < 0.05$ ) in GIC. For RC, sorption remained unaffected by pH changes, but solubility increased linearly as pH decreased, and there was no visible degradation. Regression analysis also confirmed that pH significantly affected solubility ( $p < 0.05$ ) but not sorption ( $p > 0.05$ ) in RC. These findings provided reasonable evidence that resin cement demonstrates greater resistance to acidic conditions than glass ionomer cement, indicating its potential suitability for patients with LPR. However, because this study used *in vitro* immersion conditions that may not fully replicate the acid exposures seen clinically, further *in vivo* research is needed to confirm the long-term performance of these materials.

## RESULTS

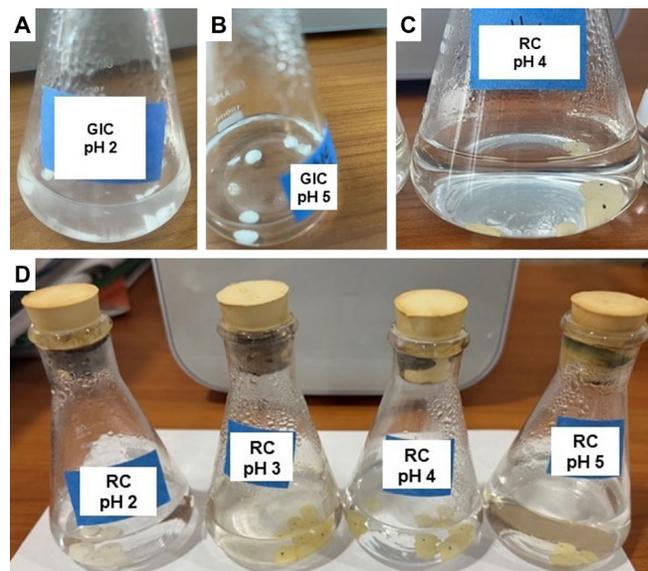
Since GIC and RC are widely used in various dental treatments, understanding their properties in patients with LPR is crucial. Our study examined a total of 40 samples (20 GIC and 20 RC) that were divided into four groups, each with five samples representing each type of cement. We immersed each group in hydrochloric acid solutions of pH 2, 3, 4, and 5, respectively, at 37°C for 30 days. We observed all samples weekly during the study and subsequently at the end of the experiment. We determined sorption and solubility for each group by calculating the percentage difference in weight of each group before immersion, post-immersion, and after 24 hours of desiccation.

At the end of the first week of incubation in hydrochloric acid, we observed that two of the GIC samples in the pH 2 solution were broken (**Figure 1A**). We also observed that the edges around the GIC samples in the solution with pH 3 were jagged, and there were a few flakes that had broken off. However, we did not observe any visual changes in the GIC samples in the solutions with pH 4 and pH 5. When we inspected the samples at the end of the second week, we found that GIC samples in the solutions with pH 4 and pH 5 had also disintegrated (**Figure 1B**). We did not observe any additional changes in GIC samples at the end of the third and fourth weeks.

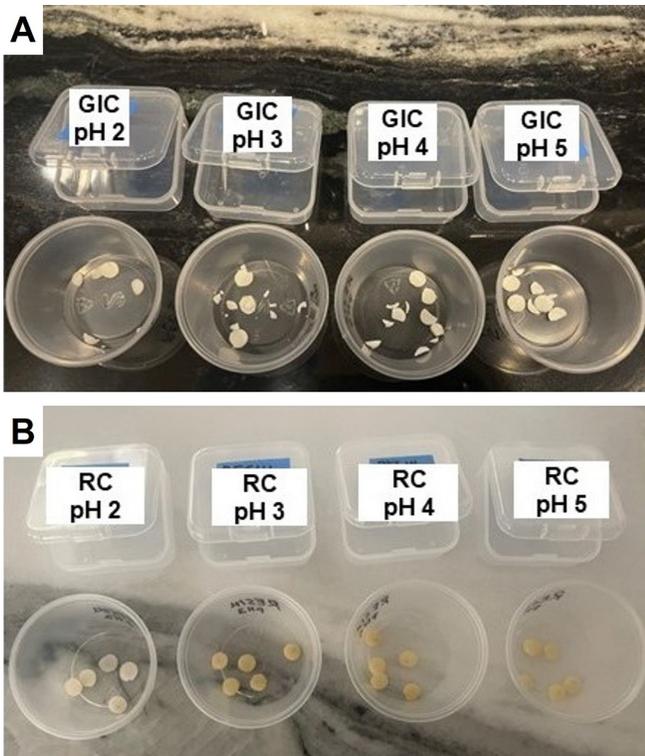
We did not observe any changes in the RC samples when we visually inspected them at the end of one week. However, at the end of the second week, we did see yellow discoloration in the RC samples in pH 4 and pH 5 (**Figure 1C**). We did not see any additional changes at the end of the third week, but after four weeks, we saw that the RC samples in pH 3 also appeared yellowish in color (**Figure 1D**).

We found deterioration in all GIC samples at the end of 30 days when we removed them from the pH solutions (**Figure 2A**). On the other hand, we noted that RC samples appeared intact and unaltered in shape and size, but there was some discoloration at pH 3, 4, and 5 (**Figure 2B**).

We found that the sorption for GIC in hydrochloric acid was  $33.17 \pm 0.038\%$  at pH = 2,  $5.27 \pm 0.060\%$  at pH = 3,  $1.96 \pm 0.018\%$  at pH = 4, and  $0.36 \pm 0.048\%$  at pH = 5 (**Figure 3A**). We saw that the sorption for GIC linearly increased significantly as pH decreased ( $p = 0.0059$ , regression analysis test, **Figure 3A**). Our calculations showed that the sorption for RC in hydrochloric acid was  $0.07 \pm 0.052\%$  at pH = 2,  $-2.78 \pm 1.47\%$  at pH = 3,  $-6.64 \pm 0.18\%$  at pH = 4, and  $-1.82 \pm 0.24\%$  at pH = 5 (**Figure 3A**). We observed that the sorption for RC did not



**Figure 1: Visual observations of glass ionomer cement (GIC) and resin cement (RC) samples.** A) GIC samples in pH 2 at the end of week one. B) GIC samples in pH 5 at the end of week two. C) RC samples in pH 4 at the end of week three. D) RC samples in pH 2, 3, 4, and 5 at the end of week four. GIC and RC samples were immersed in hydrochloric acid solutions of pH 2, 3, 4 and 5 and placed in an incubator at 37°C for 1 week. n=5 per cement type per pH value.



**Figure 2: Glass ionomer cement (GIC) and resin cement (RC) after 30 days of immersion in hydrochloric acid at various pH. A) GIC samples after 30 days in different pH solutions. B) RC samples after 30 days in different pH solutions.** GIC and RC samples were immersed in hydrochloric acid samples of pH 2, 3, 4 and 5 and placed in an incubator at 37°C for 30 days. n=5 per cement type per pH value.

correlate to changes in pH ( $p=0.56$ , regression analysis test, **Figure 3A**).

We calculated the solubility and found that for GIC in hydrochloric acid, the solubility was  $65.83 \pm 0.029\%$  at pH = 2,  $28.01 \pm 0.037\%$  at pH = 3,  $19.86 \pm 0.023\%$  at pH = 4, and  $11.11 \pm 0.037\%$  at pH = 5 (**Figure 3B**). We noted that the solubility for GIC also increased as pH decreased ( $p=0.014$ , regression analysis test, **Figure 3B**). We found that the solubility for RC in hydrochloric acid was  $2.28 \pm 0.049\%$  at pH = 2,  $1.84 \pm 0.046\%$  at pH = 3,  $1.55 \pm 0.045\%$  at pH = 4, and  $1.16 \pm 0.033\%$  at pH = 5 (**Figure 3B**). We observed that the solubility for RC also increased, as pH decreased though less steeply than GIC ( $p=0.0027$ , regression analysis test, **Figure 3B**).

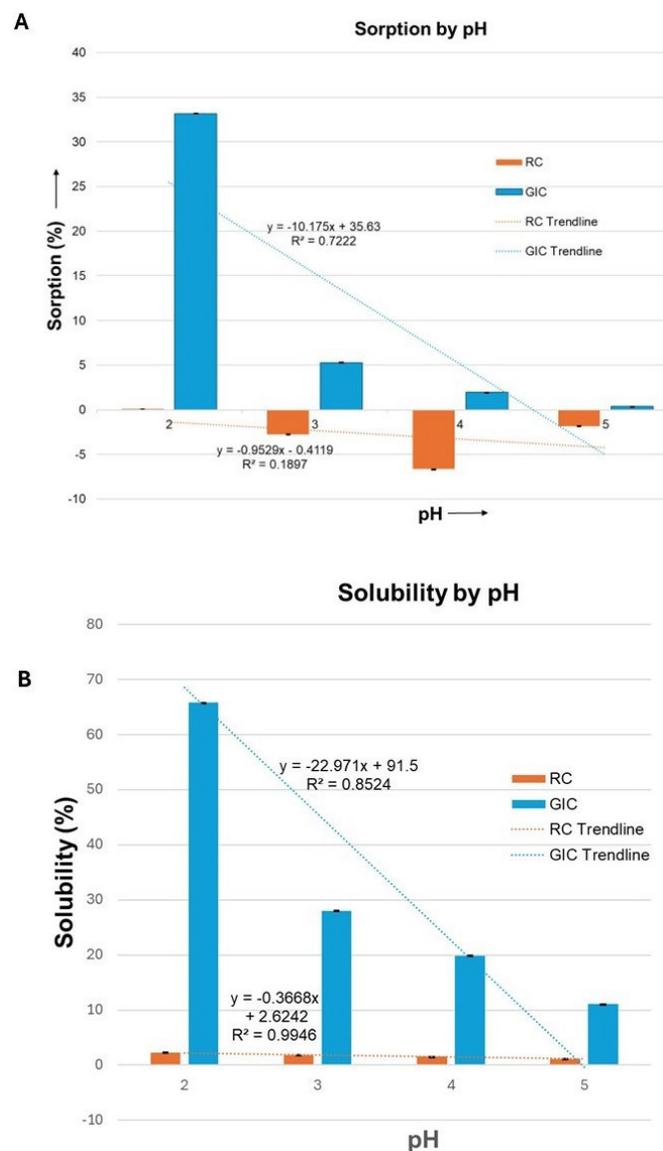
### DISCUSSION

The original purpose of our experiment was to determine the effects of pH values similar to those found in patients with LPR on common dental cements. Our study found that the degradation rate has a direct relationship with pH, in that the lower the pH, the faster the breakdown and the greater the overall deterioration. For example, the GIC samples in pH 2 degraded more rapidly than those in pH 3, 4, and 5 (**Figure 1A**). However, all GIC samples eventually disintegrated (**Figure 2A**). There was an increase in both sorption and solubility for GIC as the pH decreased (**Figure 3**).

The degradation of GIC may be related to the demineralization of its ionic matrix structure, under acidic

conditions (18). GIC sorbs water and hydrogen ions, which facilitates ion exchange and accelerates acid attack (28). This degradation can potentially reduce its integrity and durability in low pH environments.

RC may show enhanced stability in acidic environments due to its strong covalent bonds that resist disruption from hydrogen ions in acidic solutions (20). The color change we observed in RC is likely due to surface reactions, such as oxidation or slight hydrolytic degradation. At lower pH, the acidity is strong enough to cause some damage to the composite resin's structure, leading to matrix softening but not enough to promote oxidation, which is necessary for discoloration (29). In contrast, pH values that are closer to the natural pH of saliva allow for mild oxidation and surface degradation. This makes the composite resin more susceptible



**Figure 3: Sorption and solubility of glass ionomer cement (GIC) and resin cement (RC) at varying pH. A) Bar graph showing sorption (%) as pH changed from 2 to 5. B) Bar graph showing solubility (%) as pH changed from 2 to 5.** \*denotes  $p \leq 0.05$ . Error bars represent standard deviation. Dotted lines show trendlines and equations. n=5 per cement type per pH value.

to visible discoloration (30). However, these changes are superficial and do not affect the material's structural integrity, making RC a more reliable choice for prolonged exposure to low pH.

The clinical performance of dental cements is largely influenced by their stability and resistance to degradation in the oral environment. Our study revealed a potential threat to the integrity of commonly used dental treatments in patients experiencing even temporary episodes of LPR. Short or long-term exposure to reflux acids can compromise the bond between the restoration and the tooth. GIC, while offering benefits like fluoride release and biocompatibility, may be less durable in patients with LPR. In contrast, RC offers superior acid resistance, making it more suitable for LPR patients, though its aesthetic properties may need further improvement to minimize discoloration over time. The findings from our study encourage dentists to consider the reflux history of patients when selecting materials and planning treatments, ultimately leading to better long-term outcomes and more personalized care.

This study does have limitations. Future studies can be performed with additional cement types such as zinc phosphate cement and zinc polycarboxylate cement (14), larger sample sizes, and a broader range of pH values to further the results of this experiment. During this study, we tested a limited number of samples and combined replicates during weighing, as our equipment could not detect small differences in weight. More sensitive equipment would allow for each replicate to be weighed separately, allowing for a more accurate calculation of standard deviation and better assessment of variability between samples. In addition, the acid solution was not buffered in the experiment. The pH values of the solutions could have changed due to cement disintegration or exposure to CO<sub>2</sub> in the air. A follow-up study with an acid buffering solution will ensure a stable pH environment and ensure that pH fluctuations do not influence results.

The oral cavity is a complex environment subject to several physiological variations and is difficult to simulate *in vitro* (31). In our study, we immersed our samples for 30-day in hydrochloric acid solutions of varying pH. A 30-day immersion period reflects a worst-case scenario to model accelerated aging of dental cements in low pH conditions. A future study can wash out and replace the acid solution periodically to model real world reflux conditions, where each reflux episode lowers the pH for approximately one hour. An investigation into the combined effect of acid and non-acid components of refluxate, such as pepsin and bile salts, on different dental cements would improve our understanding of material behavior under conditions that more accurately reflect the complex environment of LPR (32). The extent to which RC is prone to visible discoloration can be studied by comparing the cement samples before and after immersion against a standard tooth shade guide, or by measuring the difference in saturation or luminosity through a photo editing tool. Existing research on enhancing GIC's acid resistance through modifications, such as incorporating nanoparticles or resin components, offers promising directions for improving its performance, and to optimize its durability in such conditions.

In conclusion, our study demonstrates that acidic conditions significantly impact the degradation and color stability of dental materials, with GIC being more susceptible to structural breakdown than RC. While RC showed better stability, its

discoloration at higher pH levels underscores the need for careful material selection in acidic environments. The differing responses of GIC and RC samples to acidic environments highlight how contrasting chemical compositions and structural properties can impact their stability in acidic environments. Our findings can help clinicians make more informed choices when treating patients with conditions such as LPR or GERD, reducing the risk of treatment failure. Ultimately, this research can contribute to improving the longevity and aesthetic outcomes of dental treatments in low pH environments.

## MATERIALS AND METHODS

### Sample Preparation

We created 20 GIC samples by placing GIC mix (Fuji, Cat# 901007) into 10 mm diameter x 1mm deep molds (Creatzone, Cat# B0F3Y19WFJ) and air curing at room temperature for over 24 hours. Additionally, we prepared 20 samples of RC (Kerr Dental, Cat# 33873) by dispensing the mixture into the molds directly and light-curing the samples according to instructions provided by the manufacturer. We randomly divided samples of each cement type into 4 groups for immersion in solutions of pH 2, 3, 4, and 5, respectively.

### Weighing

Using an analytic balance (Fisher Science Education, ALF 104), we weighed each group three times and recorded an average weight,  $W_1$ , for each group.

### Preparation of Acidic Solutions

We created solutions of pH 2, 3, 4, and 5 by mixing a stock solution of 0.1M hydrochloric acid (Aldon, Cat# IS28128) with varying amounts of distilled water. We validated pH values by using a digital pH meter.

### Immersion Protocol

We immersed each group of samples in 40 mL of the corresponding pH solution and placed them in a closed, temperature-controlled incubator at 37°C for 30 days. The incubator did not have a controlled humidity and CO<sub>2</sub> level. We briefly removed the flasks from the incubator at the end of each week for visual inspection and pH validation.

### Sorption and Solubility Testing

After 30 days, we removed all sample groups and recorded their weights,  $W_2$ . After that, we placed the sample groups in a desiccator for 24 hours. We weighed the dried samples and recorded a final weight after dessiccation,  $W_3$ . We used recorded weights and calculated sorption (%) as  $(W_1 - W_2)/W_1 \times 100$  and solubility (%) as  $(W_1 - W_3)/W_1 \times 100$ , where  $W_1$  represents the initial weight of the sample,  $W_2$  the weight after 30-day immersion in the acid solution, and  $W_3$  the final weight after drying (33).

### Statistical Analysis

We conducted a statistical linear regression analysis to study the effect of pH by material on sorption and solubility. All the data was analyzed using the data analysis toolpack in Microsoft Excel (Microsoft 365, Excel Version 2506). A  $p$ -value  $\leq 0.05$  was considered statistically significant.

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