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Influence of Selected Restorative Materials on the Environmental pH: In Vitro Comparative Study

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Abstract: In dental caries treatment, it is worth using such restorative materials that may limit plaque accumulation. The pH of the filling seems to be an important factor affecting the potential bacterial colonisation. Our study aimed to assess how selected restorative materials influence the environmental pH. A total of 150 specimens (30 of each: Ketac Molar, Riva LC, Riva SC, Filtek Bulk Fill, and Evetric) were placed in 100 sterile hermetic polyethene containers with saline and stored in 37 °C. The pH of each sample was measured using the electrode Halo HI13302 (Hanna Instruments, Poland) at specific points in time for 15 days. The initial pH levels were significantly lower for glass ionomer cements (3.9–4.7) compared to composites (5.9–6.0). With time, the pH increased for samples with glass ionomer cements (by nearly 1.5), whereas it decreased for samples with composites (maximally by 0.8). In the end, all materials were in the pH range between 5.3 and 6.0. The highest final pH was obtained with Ketac Molar at about 5.9. Double samples had lower pH values than single samples, irrespective of the type of material. In conclusion, immediately after application, restorative materials decreased the environmental pH, especially light-cured glass ionomer cements. For glass ionomers, within two weeks, the pH increased to levels comparable with composites.

Keywords: dental material; restorative material; composite; glass ionomer; resin-modified glass ionomer; pH; acidity; oral environment; dental filling; dental restoration

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1. Introduction

Acid demineralisation of enamel due to the metabolic activity of bacterial plaque is the first stage of the caries process, leading to the formation of a cavity [1–3]. The destructed dental tissues are restored with various materials; however, on the contact surface of the tooth and the filling, there is usually a niche that favours the plaque accumulation and may increase the risk of secondary caries. Therefore, it is worth using such restorative materials that may prevent bacterial adhesion or limit the development of bacterial plaque. The pH of the filling seems to be an essential factor influencing the potential bacterial colonisation [4]. Numerous in vitro studies show that at acidic pH, the surface of the composite material is more stable than the surface of glass ionomer cement [4–6]. Thus far, few researchers have analysed the potential effect of the filling materials on the environmental pH. However, conventional glass ionomer cements (GICs) can buffer lactic acid and release fluoride, which appears to be very beneficial in a clinical approach [7–9].

It is believed that there are no clear limits to the number of fillings a dentist can perform in one visit. If necessary, it is recommended to perform several fillings in the same area within the conduction anaesthesia. However, other factors can affect the amount of time a patient spends in the dental chair. Most often, apart from the time limitations of the doctor and the patient, the general health condition and ailments related to the temporomandibular joint can pose a limitation. After two hours with an open mouth, it is recognised that there may be irreversible changes in the joint [10]. There are no previous articles on how the pH in the oral cavity changes after tooth restoration depending on the number of fillings.

The literature reports the hydrolysis process of fillings under low and high pH conditions in vitro. The carious bacteria fermentation and gastric acid attack are most often simulated by lactic acid and hydrochloric acid, respectively [11,12]. Both composite materials and glass ionomer cements undergo hydrolysis at acidic pH [4,13,14]. However, GICs seem to exhibit more favourable properties than resin-based materials, among others: chemical adhesion to enamel and dentin in the presence of moisture, resistance to microleakage, good marginal integrity, dimensional stability at high humidity, coefficient of thermal expansion like tooth structure, biocompatibility, fluoride release, rechargeability with fluoride, and less shrinkage than resins upon setting with no free monomer being released [15]. In the case of composite materials, the key is proper preparation of the cavity edge, gradual material application, and a meticulous polishing procedure [1,16]. However, even in vitro, a microleakage can be observed in the most carefully placed composite [17]. Certain limitations can also be seen in the bonding systems. All adhesive systems are somehow unstable and susceptible to hydrolytic degradation, which may be responsible for the partial failure of the material adhesion to the tooth, leading to marginal leakage [18,19]. The literature shows that resin restoration degradation is a complex process involving the hydrolysis of the resin and the dentin collagen fibril phases within the hybrid layer. Inhibition of the collagenolytic activity, as well as the use of cross-linking agents, are the two main strategies to increase the resistance of the hybrid layer to enzymatic degradation [20,21]. There is still no answer as to whether the effect of the restorations' pH on the oral environment could be a helpful factor in reducing secondary caries. It is known that despite the continuous modernising of the filling materials, the enamel still has a more significant buffer capacity than the composite or glass ionomer cement [22,23].

Our study aimed to analyse the potential pH effect of the most used dental materials, i.e., composites and glass ionomers, on the oral environment modelled in vitro. We tried to answer the following questions:

1. How do restorative materials influence the environmental pH within the first two weeks after their application?
2. Can a larger number of one-time fillings affect the surrounding pH more?
3. Which filling materials are more stable in terms of pH?

2. Materials and Methods

2.1. Materials Used in the Study

In this in vitro study, we used five dental restorative materials. Detailed characteristics of the materials are presented in Table 1.

Table 1. Detailed characteristics of the dental restorative materials used in the study.

Material	Material Group	Manufacturer	Acronym	Composition	Lot Number
Filtek Bulk Fill, Shade A3	composite	3M/ESPE, Seefeld, Germany	FBF	Organic matrix: AUDMA, UDMA, 1,12-dodecane-DMA 20 nm silica; Filler fraction (wt%/vol%) 76.5/58.4, Fillers: 4–11 nm zirconia, ytterbium trifluoride filler consisting of agglomerate 100 nm particles	N867070

Table 1. *Cont.*

Material	Material Group	Manufacturer	Acronym	Composition	Lot Number
Evetric, Shade A3	composite	Ivoclar Vivadent, Schaan, Liechtenstein	ER	Organic matrix: Bis-GMA, Bis-EMA, UDMA; Filler fraction (wt%/vol%) 80–81/55–57, Fillers: barium glass, ytterbium trifluoride, mixed oxide, copolymers (size 40–3000 nm)	Y20235
Ketac Molar Easymix, Shade A3	glass-ionomer cement	3M/ESPE, Seefeld, Germany	KM	Liquid: polyacrylic acid 20–30%, tartaric acid 10–15%, water Powder: fluoroaluminosilicate glass 90–95%, polyacrylic acid 5–10%	7870998
Riva SC, Shade A3	glass-ionomer cement	SDI Limited, Victoria, Australia	RSC	Liquid: polyacrylic acid 20–30%, tartaric acid 10–15%, water Powder: fluoroaluminosilicate glass 90–95%, polyacrylic acid 5–10%	B1912041
Riva LC, Shade A3	resin-modified glass ionomer cement	SDI Limited, Victoria, Australia	RLC	Liquid: polyacrylic acid 20–30%, tartaric acid 5–10%, HEMA 20–25%, dimethacrylate cross linker 10–25%, acid monomer 10–20% Powder: fluoroaminosilicate powder 95–100%	J2011171

2.2. Specimen Preparation

All specimen preparation was completed by one operator to reduce variability. A total of 150 specimens (30 from one material) were prepared using metal moulds with 6 mm diameter and 2 mm thickness. All materials were inserted into the mould and intentionally overfilled. Then, the mould was sandwiched between transparent Mylar strips to expel excess material. The uncured resin-modified glass ionomer and composites were light-cured for 40 s, according to the manufacturer's instructions (light intensity of 1200 mW/cm^2, Translux Wave, Kulzer GmbH, Hanau, Germany). Conventional glass ionomer cement (CGIC) was prepared according to the powder-liquid mixing ratio indicated by the manufacturers. After 30 min, the CGIC specimens were removed from the moulds.

2.3. Specimen Storage and pH Evaluation

The samples were placed in 100 sterile hermetic polyethene containers (ApteoCare, Sanmed, Bydgoszcz, Poland; lot: 06/KS/2021/S) and incubated in 37 °C. Each type of material was divided into 20 containers. In the first 10, there was one sample of the filling material and in the next 10, two samples. To each container, 5 mL of saline (Polpharma, Stargard Gdanski, Poland; lot: 1280620) was added.

The pH of each sample was measured using the electrode Halo HI13302 (Hanna Instruments, Olsztyn, Poland) by immersing it into the central part of the solution. Between procedures, the electrode was cleaned and recalibrated. All measurements were made in duplicate and then averaged. The pH evaluation was performed at the following time points: after 1, 6, 12 and 24 h and after 3, 6, 9, 12 and 15 days from the specimen preparation.

2.4. Statistical Analysis

The determined pH values were analysed using a two-way repeated-measures analysis of variance (in the model with the interaction). The pH changes were compared separately at specific points in time depending on the kind of used dental restorative material and the number of the incubated samples. The significance level was defined as $\alpha = 0.05$. The statistical analysis was performed using Statistica 13.3 (Statsoft, Cracow, Poland).

3. Results

For a better interpretation, all results are presented in the form of graphs. Figures 1–3 show the differences in pH changes over time depending on the dental materials used.

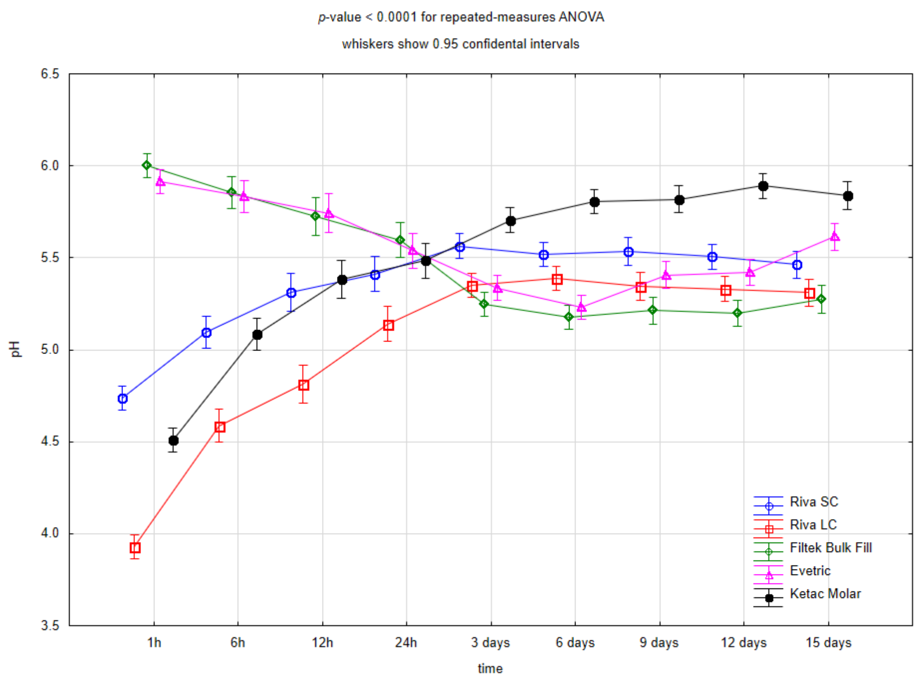

Figure 1. Repeated-measures analysis of variance for pH values in the individual time points depending on the dental restorative material (without subdividing by number of samples).

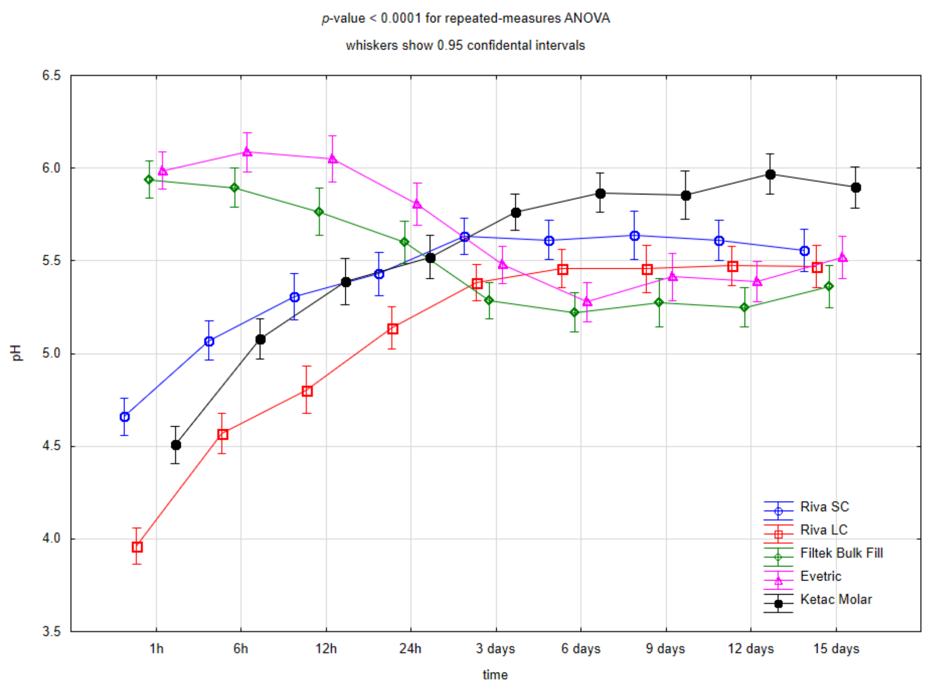

Figure 2. Repeated-measures analysis of variance for pH values in the individual time points depending on the dental restorative material for the one sample.

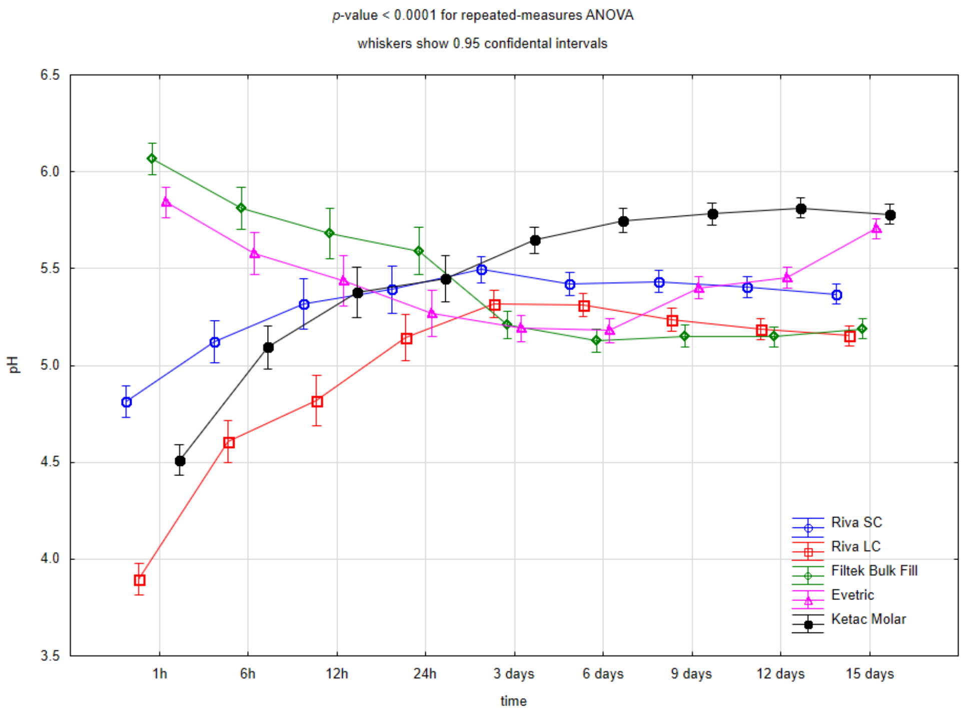

Figure 3. Repeated-measures analysis of variance for pH values in the individual time points depending on the dental restorative material for the two samples.

In general, all materials of a given group showed similar trends in pH change over time, regardless of the number of samples. Glass ionomer materials showed a significant increase in initial pH level from acidic to more neutral. The lowest initial pH level was found for the light-cured glass ionomer. However, for this material, the final pH level was higher by nearly 1.5. A similar increase was found for Ketac Molar. In contrast, for composite materials, a decrease in pH level by less than 1 was observed. At the endpoint, all materials had pH levels close to each other between 5 and 6, with Ketac Molar having the highest value. Moreover, after about a week, this material had a significantly higher environmental pH than all the others.

The changes in pH over time for individual materials depending on the number of the samples are presented in Figures 4–8.

For glass ionomer materials, already after three days, significant differences between pH levels can be seen depending on the number of samples. In the case of dual samples, lower values were obtained. In contrast, for Evetric composite material, significant differences were observed in pH level depending on the number of samples during the first three days. Additionally, the two samples showed significantly lower values, although higher variability was determined for the measurements of composite materials. On the other hand, Filtek Bulk Fill was the only material without pH variation depending on the number of samples, although there were changes in pH over time.

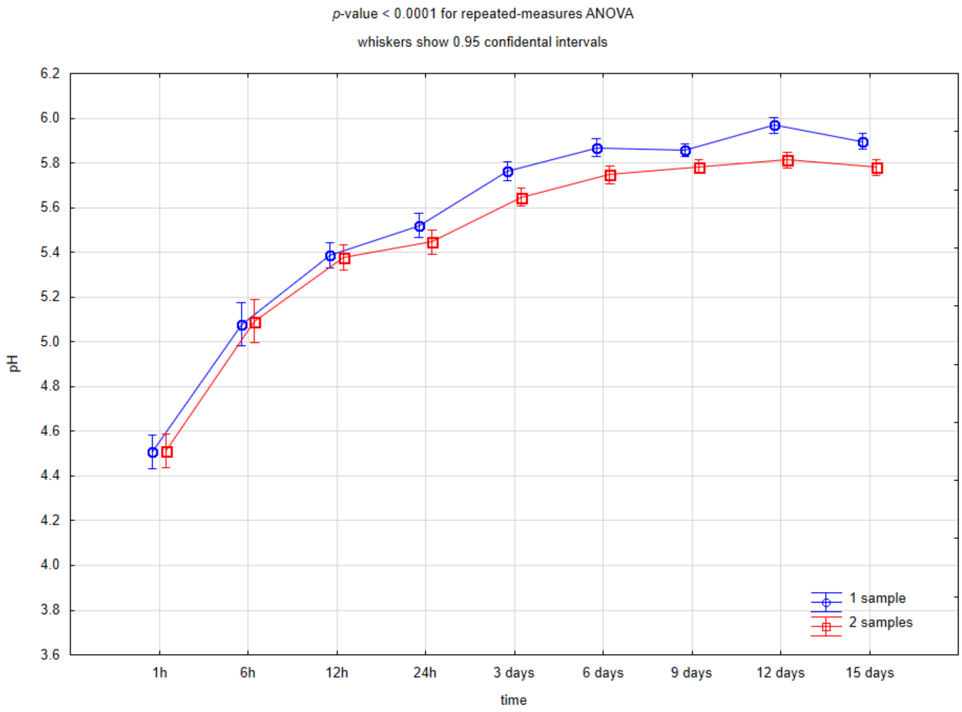

Figure 4. Repeated-measures analysis of variance for pH values in the individual time points depending on the number of the samples for Ketac Molar Easymix.

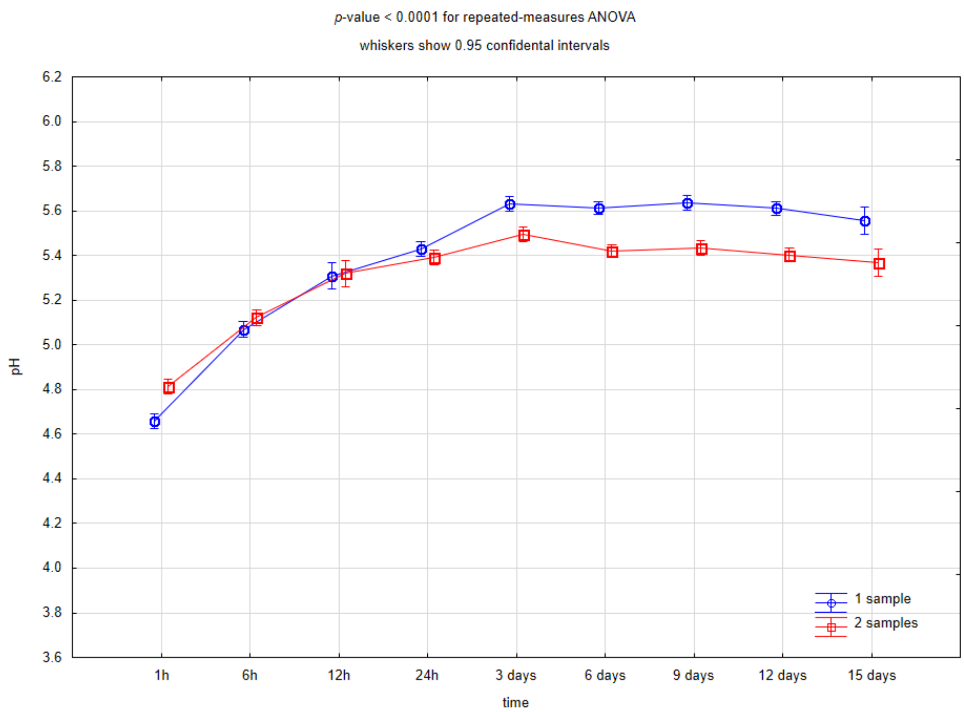

Figure 5. Repeated-measures analysis of variance for pH values in the individual time-points depending on the number of the samples for RIVA SC.

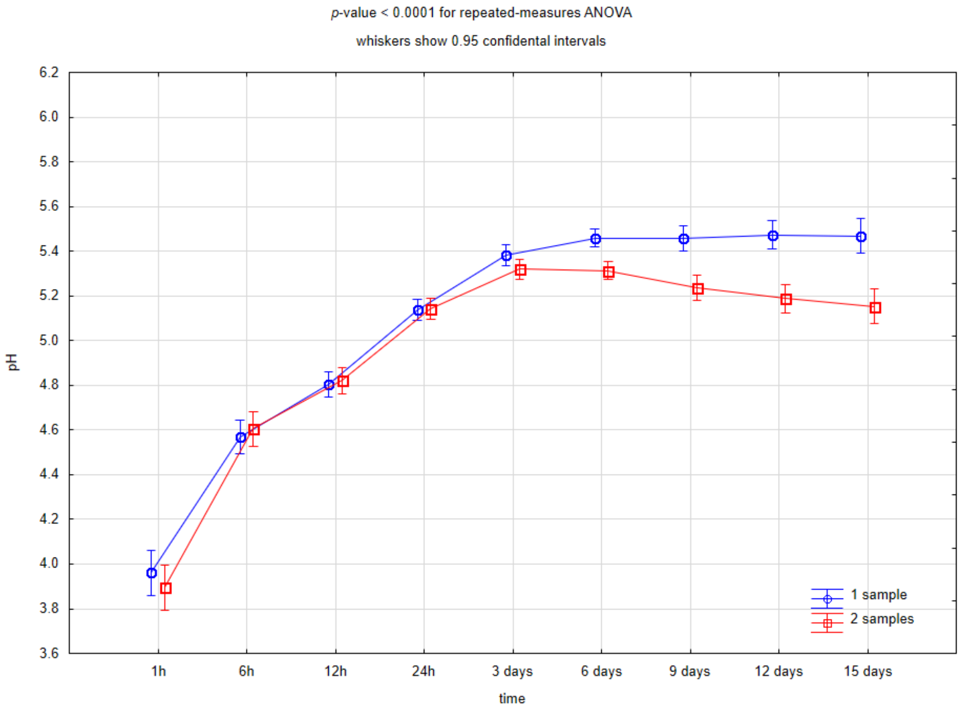

Figure 6. Repeated-measures analysis of variance for pH values in the individual time points depending on the number of the samples for RIVA LC.

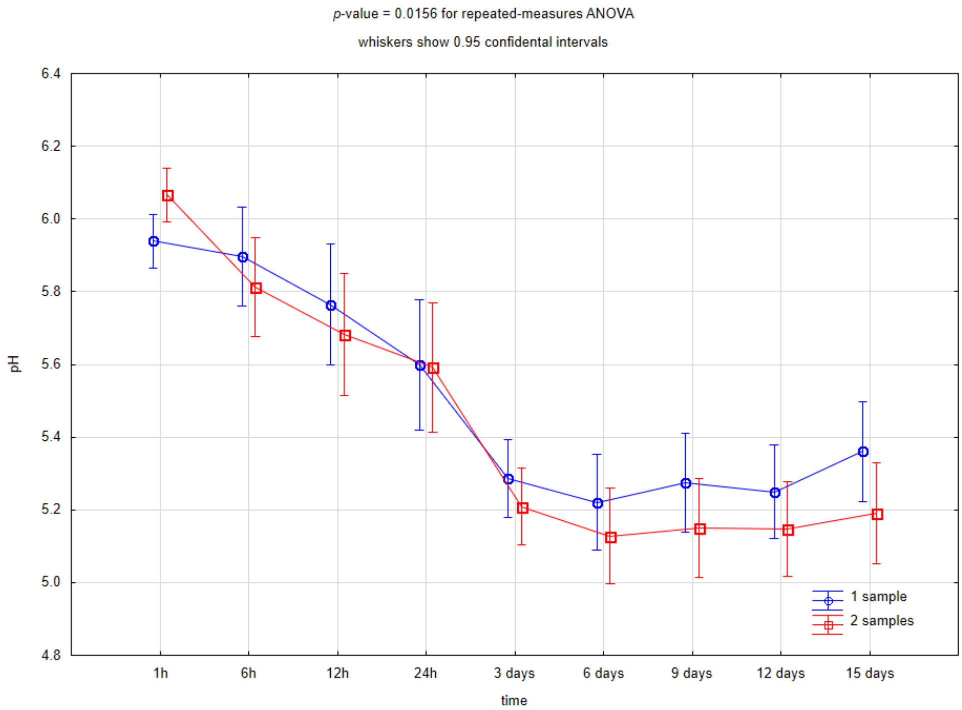

Figure 7. Repeated-measures analysis of variance for pH values in the individual time points depending on the number of the samples for Filtek Bulk Fill.

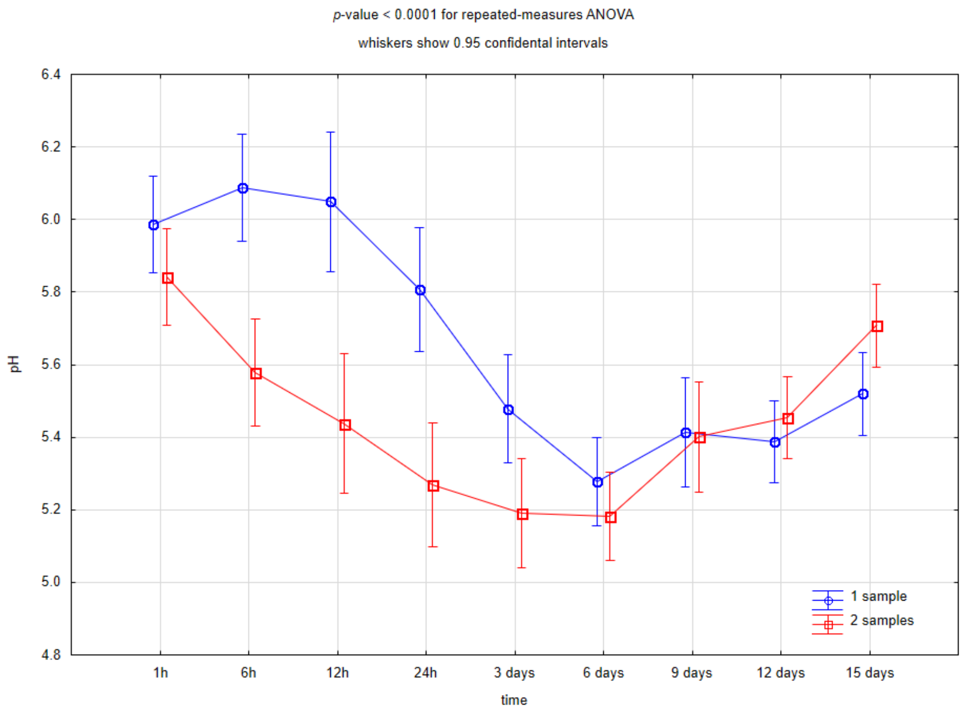

Figure 8. Repeated-measures analysis of variance for pH values in the individual time points depending on the number of the samples for Evetric.

4. Discussion

Unlike many researchers, we have chosen saline as a research medium to create an oral environment [13,18,24]. It should be noted that the use of distilled water, even at human body temperature, does not reflect oral conditions [25]. Several authors have already used saline with interesting findings [26–28]. Researchers also use artificial saliva to determine the pH of dental materials [29,30]. However, its composition is very variable; the search for the best composition is still ongoing [31]. Apart from the eating situation, the oral cavity contains maximally about 1.1 mL of saliva as this amount forces the swallowing reflex [32]. For our analysis, we used 5 mL of saline so that the pH electrode was completely immersed in the solution.

In our study, we found significant differences in the effects of different materials on the environmental pH. After 15 days, all tested materials reached a pH between 5 and 6 on the pH scale. It seems that this fact may have some clinical implications.

It is well known that glass ionomer cements in the initial setting phase are characterised by a low pH that can irritate the pulp [13,15]. Therefore, it was surprising that they finally reached a pH higher than composites. Interestingly, conventional glass ionomers acidified the environment to a lesser extent than those modified with resin during the entire 15-day observation period. It is believed that this is a consequence of the lack of any buffering capacity of composite materials [4,33]. Many researchers indicate this fact as the main cause of secondary caries in materials based on synthetic resin [34,35]. We also observed that a single sample of the GIC lowered the environmental pH less than dual samples. The highest final pH about 5.9 was achieved by Ketac Molar. According to other researchers, it may even reach a pH close to 7 [13]. The first three days of material maturation seem to be crucial, when the pH increase was faster. The pH variation of Riva SC was similar but with a lower amplitude of the pH value at about 0.8 (compared to 1.4). This behaviour can be explained by releasing unreacted acrylic (or another organic acid) and its calcium salt, a weak acid, and conjugate base, which constitute a typical chemical buffer [35]. Some researchers state that it may result from the acid groups being bound to polymer molecules with limited diffusivity [2]. The most acidifying material was Riva LC which had the lowest initial pH of below 4.0 (below the critical point for enamel). In the case of two samples, it was also the most acidic material after 15 days with a pH of about 5.2. It is commonly believed that RMGICs are more composites than glass ionomers [36,37], which was confirmed by the findings of our experiment.

The research methodology on composite materials focuses largely on subjecting them to cyclical changes in pH [38,39]. However, there is no clear answer as to whether these materials are chemically stable at a neutral pH. Despite the daily and routine placement of composite restorations, dentists' knowledge about the composite properties and the main factors of the polymerisation process is not adequate. It can lead to incorrect curing protocol, resulting in the incomplete polymerisation as well as relying on residual monomers and pH changes [40,41]. Moreover, chemical degradation of composite resin results in the release of final products, such as methacrylic acid which could acidify the environment [42,43].

Contrary to other studies [35,44], composite materials used in our research (Filtek Bulk Fill and Evetric) acidified the environment. Despite the initially relatively high pH of about 6, we observed a significant decrease to about 5.3 during the next 3 days of measurements. Around day 6, the pH started to rise slightly, and on day 15, it reached about 5.5. The Evetric composite, containing the Bis-EMA resin, turned out to be less acidifying in our study. Some researchers suggest that Bis-EMA, due to its hydrophobicity and high conversion character, is characterised by lower water sorption and lower solubility [45]. Another theory claims that UDMA composites are less hydrophilic than Bis-GMA-based composites. Also, it is suggested that the main difference between UDMA and Bis-GMA is their flexibility. Therefore, systems containing carboxylic or phosphate groups as functional monomers are more hydrophilic than the resins containing the Bis-GMA/TEGDMA monomer system [41,46]. It is believed that differences in the water absorption of the polymer network can be observed depending on the type of monomer. TEGDMA, Bis-GMA and UDMA seem to be the most hydrophilic [41].

Our study showed that medium-sized fillings are capable of inducing pH changes in the aquatic environment. Interestingly, the acidifying effect was visible in the case of resin-based materials, and two fillings lowered the environmental pH more than one. The literature shows that the lower the cross-linking of the composite, the higher the water absorption. It follows that the polymerisation quality of the composite may affect the pH stability [46]. In clinical practice, the irradiation of a portion of the material is not always optimal—a lower degree of conversion may adversely affect the quality of the filling and its subsequent durability [47]. We are planning further analyses to assess the possibility of changes in acidification by modifying the exposure time of the samples, the distance from the polymerisation lamp, and the diameter of the lamp optical fibre. The soaking of the filling materials in saline would also affect the dimensional, mechanical, and chemical properties. Therefore, it would also be advisable to evaluate the surface roughness of the samples or the release of chemicals such as residual monomers and metal ions.

5. Conclusions

Restorative materials lowered the environmental pH immediately after application, especially light-cured glass ionomer cements. Within two weeks, the pH level for glass ionomers rose to values comparable to composite materials. Moreover, dual samples reached lower pH levels than single samples. Self-cured materials showed larger amplitudes of pH changes than light-cured ones.

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