

## ORIGINAL RESEARCH

# The effects of gastric acid on pediatric restorative materials: SEM analysis

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**Abstract**

In this study, we aimed to demonstrate changes in the surface roughness and microhardness of three different restorative materials routinely used in pediatric dentistry (composite, compomer and resin-modified glass ionomer cement (RMCIS)) in response to continuous daily exposure to gastric acid. Twelve samples of each of type of restorative material were prepared. Eleven of the specimens were included in the gastric acid cycle. The microhardness and surface roughness of ten samples were measured before and after the cycle. Another sample included in the cycle was compared with the sample not included in the cycle by scanning electron microscopy (SEM). There was a significant difference between the groups in terms of roughness scores following gastric acid cycle ( $p = 0.039$ ). RMCIS material possessed the highest roughness value. A significant difference was identified in terms of microhardness levels before and after the gastric acid cycle ( $p = 0.001$ ). The most significant change was observed in the compomer material. SEM analysis, performed after the gastric acid cycle, revealed that most cracks were identified in RMCIS material; this was followed by compomer and composite materials, respectively. Our analysis indicates that the restorative materials used frequently in pediatric dental procedures, show increased surface roughness and reduced microhardness when exposed to gastric acid.

**Keywords**

Gastric acid; Microhardness; Surface roughness; Composite; Compomer; Resin modified glass ionomer cement

## 1. Introduction

An important aspect of the desired properties of a restorative dental material is to be physically and chemically similar to tooth tissue, to provide high levels of adaptation to tooth tissue, and to provide functionality by remaining in the mouth for an extended period of time [1]. The microhardness and surface roughness of dental materials are important features of tooth tissue integrity. Many internal and external factors can influence the structure of dental materials. Rough surfaces promote plaque build-up and material wear more easily than smooth surfaces. An increase in the surface roughness of restorative materials represents a key risk factor for microbial colonization and future oral diseases. In addition to occlusal stress and trauma to the teeth during chewing movements, there are many other factors that can affect the surface roughness of restorative materials, including the abrasive effects of toothpaste and tooth brushing, factors such as poor restoration polishing, the continuous intake of cold and hot various foods and beverages into the oral environment, and acidic erosion caused by food and beverages. Among other external factors that contribute to increased surface roughness [2, 3].

In addition to external factors, internal factors, can also af-

fect the properties of restorative materials. The most dominant of these factors is gastroesophageal reflux (GER) disease, a condition that occurs when stomach contents flow uncontrollably into the esophagus [4]. The pH of gastric acid ranges from 1–1.6. The regular presence of gastric acid in the oral environment is an important factor that can easily reduce the pH of the mouth below 5.5; this is a critical pH value with regards to the solubility of enamel. Research has shown that gastric acid can cause tooth erosion if it comes into contact with the teeth several times per week for at least 1–2 years [5]. The palatal surfaces of the upper teeth are said to be the most susceptible to erosion caused by GER. The occlusal surfaces of the upper premolars and molars, as well as the palatal surfaces, become eroded as the factors causing erosion continue. Furthermore, erosion can affect the occlusal and buccal surfaces of the lower teeth in severe cases [6].

Damage induced by acid attacks can cause damage to both dental tissues and restorative materials. Consequently, the surface polishing of restorative materials used inside the mouth should be of a specific quality and checked regularly. In children, dental erosion caused by GER is a rare condition. This is because children have a shorter history of GER and reflux is limited to the esophagus [7]. When erosion is detected early,

conservative treatment methods can be applied. The primary goal of erosion treatment is to provide maximum protection for both the anterior and posterior teeth while minimizing changes to the tooth structure [8]. It should be noted, however, that low pH gastric fluid entering the mouth as a result of reflux affects not only the natural teeth but also the surfaces of restorative materials [9].

The purpose of this study was to evaluate the changes in surface roughness and microhardness of three different restorative materials routinely used in pediatric dentistry (resin modified glass ionomer cement, compomer and composite resin) when constantly exposed to gastric acid during the day. Our null hypothesis was that prolonged contact with gastric acid would not change the surface roughness and microhardness of restorative materials.

## 2. Materials and methods

### 2.1 Study design

A composite resin (Palfique Estelite Paste, Tokuyama, Japan), a compomer (Nova Kompomer, Imicryl, Konya, Turkey), and a resin modified glass ionomer cement (Riva Light Cure HV, SDI Dental, Victoria, Australia) were tested in this *in vitro* study. Surface hardness and surface roughness values were compared between these materials. Table 1 lists the properties of the restorative materials used.

### 2.2 Sample preparation

Data from the three groups were compared by two repeated measures analysis of variance (ANOVA). Based on power analysis performed using G\*Power version 3.1 software (Heinrich Heine University, Düsseldorf, Germany), we determined that a minimum of 30 samples were required with 10 samples in each subgroup to achieve an error margin of 0.05, and effect ( $f$ ) of 0.50, and a power of 0.90 at the  $(1-\beta)$  level. In addition, two additional samples from each group were prepared for SEM analysis. Disc-shaped ( $10 \times 3$  mm) standardized specimens were created from restorative materials using a specially designed mold. The restorative materials were then polymerized for 20 seconds in accordance with the manufacturer's instructions using an LED light source (Guilin Woodpecker, Guangxi, China) and a wavelength of 420–480 nm and power intensity of 1400 mw/cm<sup>2</sup>. All samples had one surface standardized underwater with 800, 1000 and 1200 grit sanders, respectively (Tor VM, Moscow, Russia).

Surface hardness and surface roughness were measured on 10 samples from each group before and after a gastric acid cycle. One of the two unmeasured samples received no treatment; the other was subjected to the gastric acid cycle (Fig. 1).

### 2.3 The artificial saliva and gastric acid cycle

Similar studies in the literature were used to create gastric acid solutions and artificial saliva solutions [10–12]. Table 2 lists the contents of the solution. During the gastric acid cycle, we used 5 mL tubes containing gastric acid, distilled water, and artificial saliva. After 60 seconds in gastric acid, the samples

were purified in distilled water for 5 seconds before being returned to artificial saliva for 30 minutes [10, 11]. Over a period of ten days, this application was repeated six times at one-hour intervals.

### 2.4 Surface roughness evaluation

Surface roughness measurements (Ra) were taken before and after the gastric acid cycle using a surface roughness instrument Mitutoyo® (Surftest SJ-210, Tokyo, Japan). Measurements were taken from three different areas on each surface, and mean values were recorded, as well as mean Ra (m) value for each sample.

### 2.5 Surface microhardness evaluation

The Vickers microhardness test was used for microhardness testing, with surface hardness measurements taken with a THV-1De before and after each sample was exposed to the gastric acid cycle. The surface microhardness was measured at room temperature ( $23 \pm 1$  °C) using a 300 g weight for 20 seconds, and the values were recorded as Vickers hardness units. Three measurements were taken in the central region, each no closer than 1 mm to the other or the edges. For each sample, the mean of these three results was accepted as a single value.

### 2.6 Scanning electron microscopy (SEM) analysis

One sample from each group was subjected to the gastric acid cycle. The Hitachi SU-1510 SEM was used for the analysis (Hitachi High-Technologies Corp., Tokyo, Japan). The surface topography of the samples was assessed using images at  $\times 500$  and  $\times 5000$  magnification. Prior to SEM analysis, the samples were induced to be conductive by coating them with gold/palladium.

### 2.7 Statistical analysis

The SPSS 26 statistical package program (SPSS Inc., IBM, New York, USA) was used to analyze the data and  $p < 0.05$  represented statistical significance. The normality of data distributions was first checked by applying the Kolmogorov-Smirnov and Shapiro-Wilk tests. If the  $p$ -value obtained from the Kolmogorov-Smirnov test was  $< 0.05$ , then non-parametric testing was considered appropriate because the sample size of the subgroups was  $< 30$ . The restoration materials were compared using the Kruskal Wallis H test, and their pre-cycle and post-cycle measurements were compared using the Wilcoxon Signed Rank test.

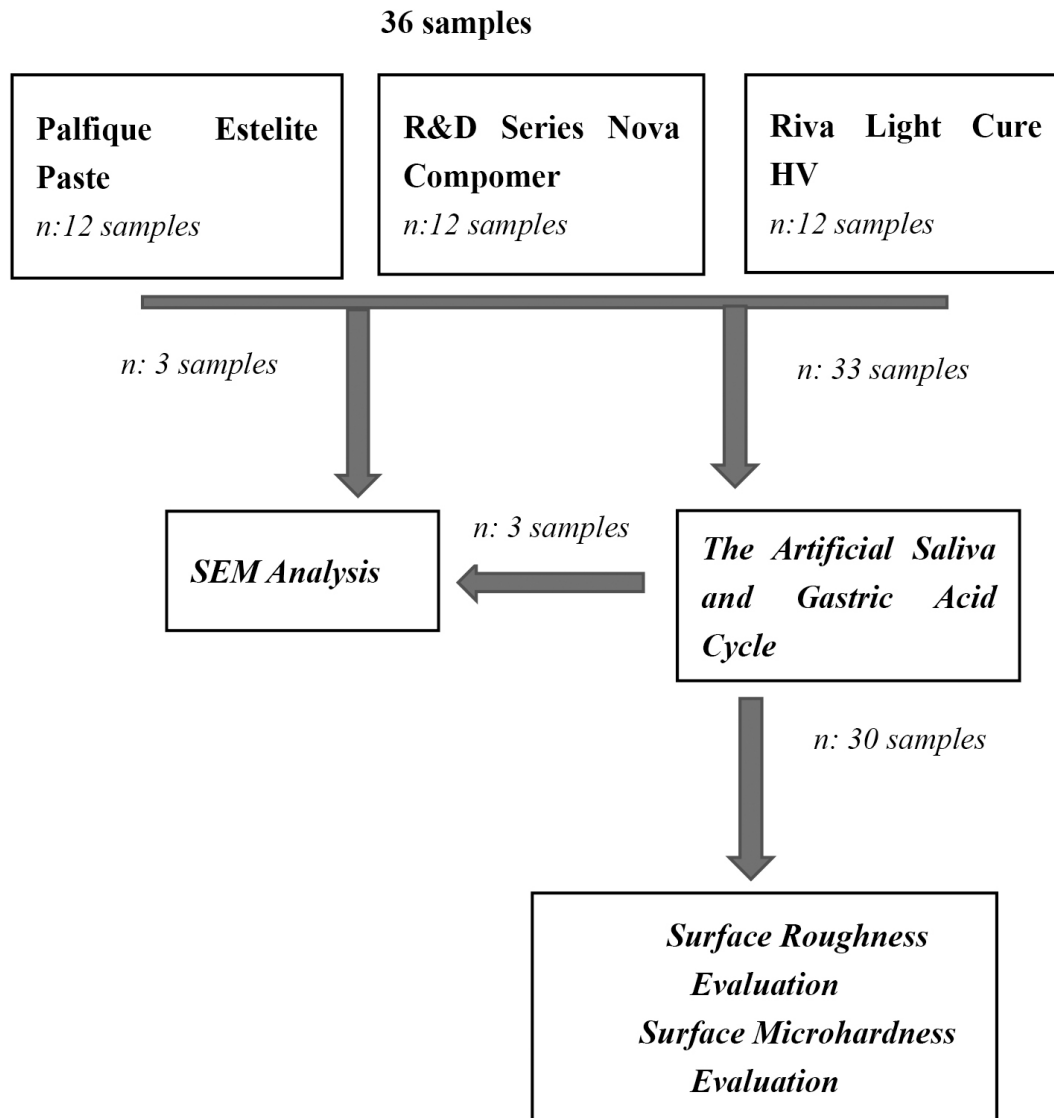
## 3. Results

### 3.1 Results of the surface roughness test

Prior to the delivery of gastric acid, analysis showed that the compomer material had the lowest roughness value; this was significantly lower than the other materials. Similar to the assessment of roughness values following gastric acid administration, a statistically significant difference was discovered between the three restorative materials ( $p = 0.039$ ). The

**TABLE 1. Characteristics of the restorative materials used in the study.**

Material	Material Type	Contents	Manufacturer	Lot No.
Palfique Estelite Paste	Microhybrid Composite	Contains 58% silica-zirconia filler and silica-titania filler by weight. Bisphenol A polyethoxymethacrylate (Bis-MPEPP), Triethylene glycol dimethacrylate (TEGDMA), and 1,6-bis (methacryl-ethoxycarbonylamino) trimethylhexane comprise the monomer matrix (UDMA).	Tokuyama (Tokyo, Japan)	041E91
R&D Series Nova Compomer	Compomer	UDMA, Carboxylate modified dimethacrylate, TEGDMA, Dimethacrylate resins, Yttrium trifluoride, alumino-fluoro-silicate glass, Catalysts, Stabilizers, Pigments.	Imicryl (Konya, Türkiye)	23A254:701
Riva Light Cure HV	Enhanced Resin-Modified Glass Ionomer Cement	Polyacrylic acid, tartaric acid, dimethacrylate binders and acid monomer are all examples of methacrylate monomers.	SDI Dental (Victoria, Australia)	K2202034EA

**FIGURE 1. Workflow of samples belonging to each of the groups used in the study. SEM: Scanning Electron Microscopy.**

**TABLE 2. The contents of the solutions in the study.**

Solution	Contents	pH
Artificial Saliva	20 mM 4-(2-hydroxyethyl)-1-piperazineethanesulfonic acid, 30 mM potassium chlorid (KCL), 4 mM mono potassium phosphate (KH <sub>2</sub> PO <sub>4</sub> ), and 0.7 mM calcium chloride (CaCl <sub>2</sub> )	6.8
Artificial Gastric Acid	Hydrochloric acid (HCl) 0.06 M (0.103% solution in deionized water, 1.5 mg/mL pepsin	1.6

RMCIS restoration material differed notably from the other groups and had the highest roughness value (Table 3). There was no statistically significant difference between the three restorative materials regarding differences in roughness values before and after gastric acid administration ( $p = 0.555$ ). However, the RMCIS material experienced the most remarkable improvement in roughness.

**TABLE 3. Evaluation of material roughness prior to and during delivery of gastric acid.**

	Surface Roughness		<i>p</i>
	Before	After	
Palfique Estelite Paste	0.010 ± 0.002	0.011 ± 0.004	0.394
R&D Series Nova Compomer	0.008 ± 0.001	0.011 ± 0.002	0.006
Riva Light Cure HV	0.012 ± 0.003	0.015 ± 0.004	0.047
<i>p</i>	0.002*	0.039*	

\* $p < 0.05$ .

### 3.2 Results of microhardness tests

Both before and after the delivery of gastric acid, we found that the RMCIS material had the lowest microhardness values which were significantly lower than the other materials ( $p < 0.001$ ) (Table 4). The assessment of microhardness when the restorative materials were exposed to gastric acid revealed a statistically significant difference between the three restorative materials ( $p = 0.001$ ). The material used in composites experienced the greatest change in terms of microhardness.

### 3.3 Results of SEM analysis

In the study, we evaluated the surface topography of the control and experimental groups at  $\times 500$  and  $\times 5000$  magnifications. All sample groups showed microcracks and pores. In addition, we noted that the groups that had experienced the gastric acid erosive cycle had higher surface irregularity and microcracks (Figs. 2,3,4). The RMCIS group had the greatest number of microcracks; this was followed by compomers and then composite resin.

## 4. Discussion

The selection of restorative materials is among the most important factors in pediatric dentistry. Generally, pediatric restorations are performed quickly due to difficulties associ-

**TABLE 4. Evaluation of material microhardness prior to and during delivery of gastric acid.**

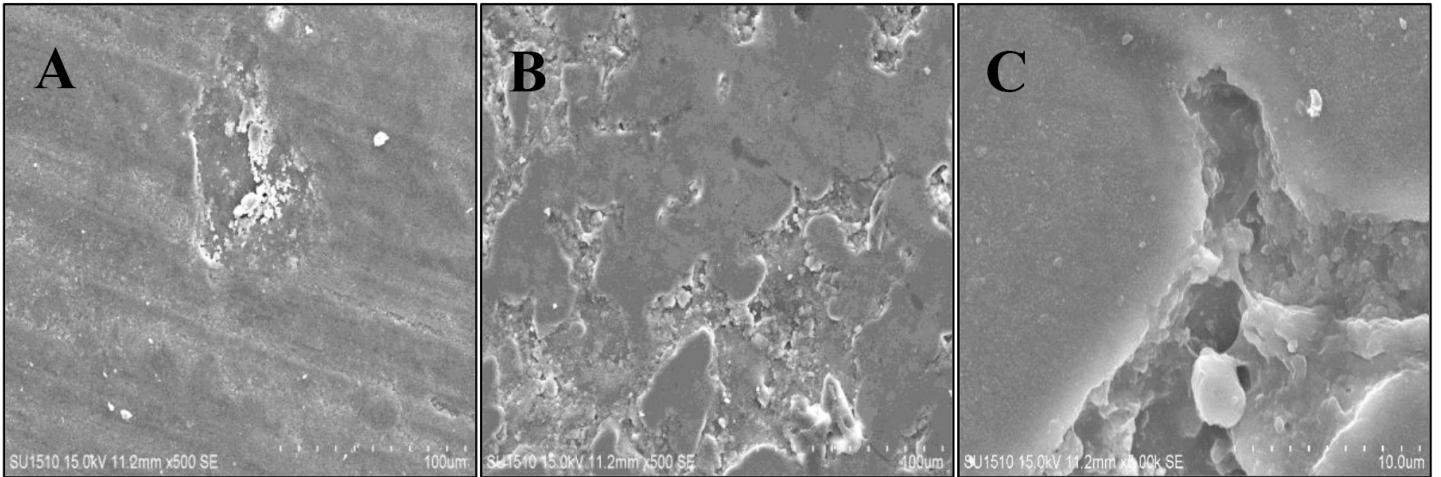
	Microhardness		<i>p</i>
	Before	After	
Palfique Estelite Paste	59.830 ± 1.076	45.450 ± 2.080	0.001*
R&D Series Nova Compomer	52.560 ± 1.449	29.920 ± 1.707	0.001*
Riva Light Cure HV	29.070 ± 3.224	14.250 ± 1.953	0.001*
<i>p</i>	0.001*	0.001*	

\* $p < 0.05$ .

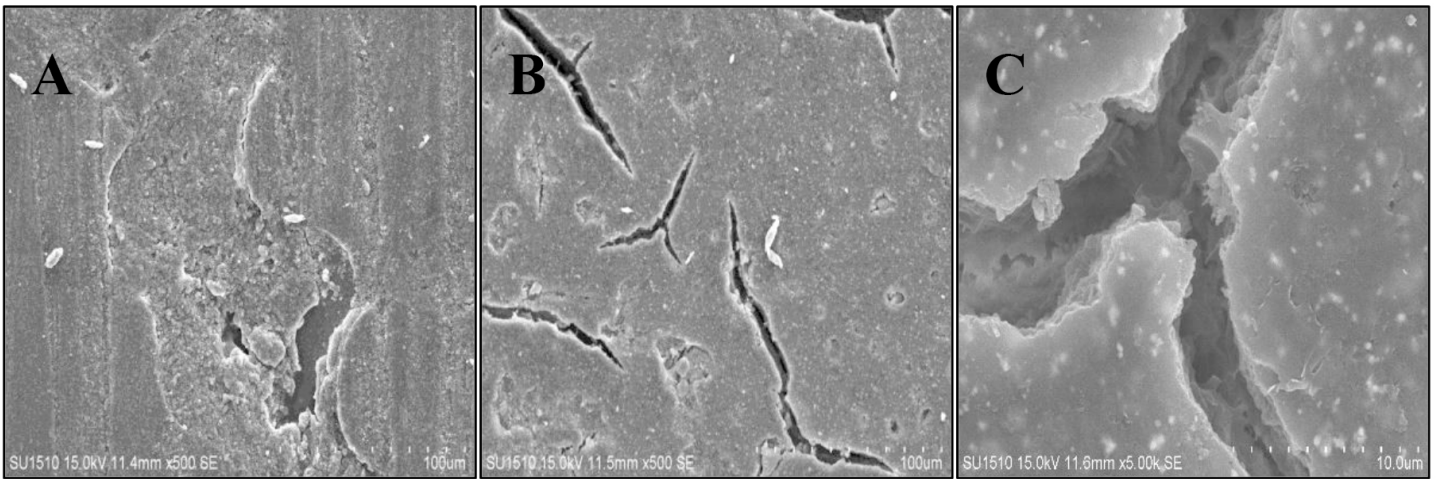
ated with young patients. Nevertheless, when possible, the best esthetic treatment should be performed in order to retain self-esteem and confidence in dental treatments [13]. It is well known that further expensive and complex procedures can be avoided if the restoration fails by choosing the correct restorative material for the indication. One of the most popular restorative materials in composite and primary teeth is glass ionomer.

The term “gastroesophageal reflux” (GER) refers to the overflow of gastric acid or other gastric enzymes into the esophagus. Studies have demonstrated an association between localized dental erosions and GER, which is typically encountered in children and adolescents. The loss of a tooth’s hard structures due to chemical processes and not bacterial activity, is referred to as erosion. The hardness and roughness of restorative materials, as well as teeth, are also affected by the low pH of gastric acid that GER introduces into the oral environment [4, 5]. In this study, we investigated the effect of restorative materials, which are often used in pedodontics, on the surface roughness and microhardness of restorative materials as a result of exposure to gastric acid solution by simulating the oral environment. Our results rejected our null hypothesis that prolonged exposure to gastric acid would not affect the surface roughness and microhardness of restorative materials.

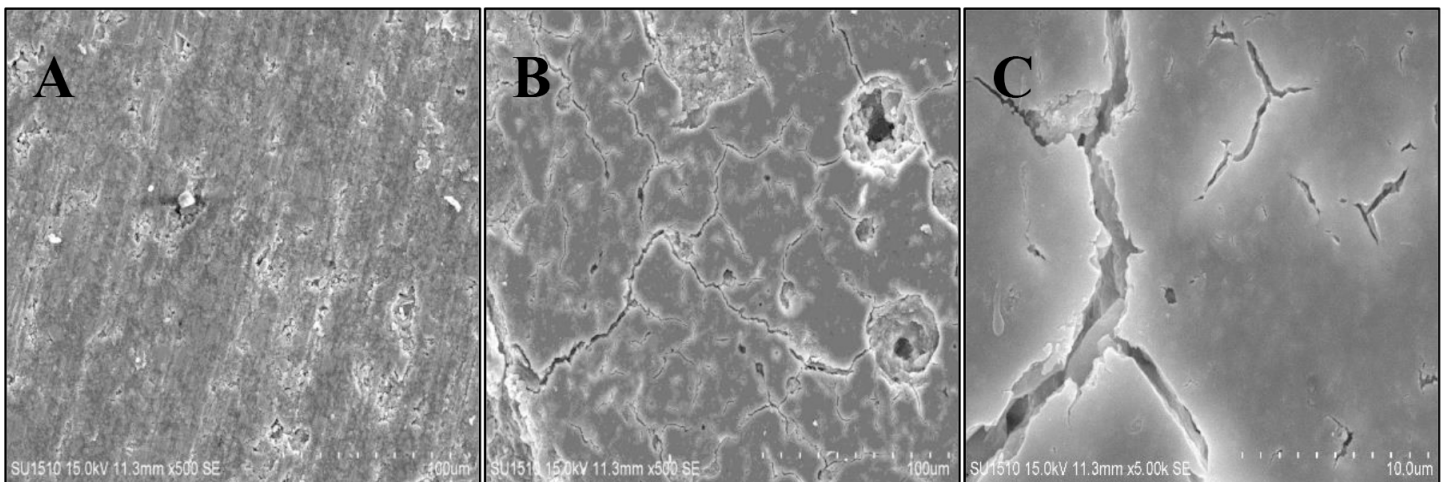
By subjecting glass ionomer cement and composite resin sample groups to the erosive cycle of citric acid for 1 minute (6 times each day for 10 days), Yu *et al.* [10] examined the erosive susceptibility of restorative materials. After each cycle, the materials were cleaned in distilled water for 5 seconds before being kept in artificial saliva for at least 30 minutes. The composite was demonstrated to be the best erosion-resistant material in our study which assessed the loss of material prior to the next cycle [10]. The composite material in our study



**FIGURE 2. Images of composite material during the gastric acid cycle. (A)** SEM image before composite gastric acid cycle. **(B)** SEM image after composite gastric acid cycle ( $\times 500$ ). **(C)** SEM image after composite gastric acid cycle ( $\times 5000$ ).



**FIGURE 3. Images of compomer material during the gastric acid cycle. (A)** SEM image before compomer gastric acid cycle. **(B)** SEM image after compomer gastric acid cycle ( $\times 500$ ). **(C)** SEM image after compomer gastric acid cycle ( $\times 5000$ ).



**FIGURE 4. Images of RMCIS material during the gastric acid cycle. (A)** SEM image before RMCIS gastric acid cycle. **(B)** SEM image after RMCIS gastric acid cycle ( $\times 500$ ). **(C)** SEM image after RMCIS gastric acid cycle ( $\times 5000$ ).

was chosen because it was least impacted by the gastric acid cycle; the waiting and cycle times were adopted by reference to appropriate articles [10, 11].

Rough surfaces increase the probability of plaque formation more than smooth surfaces, but they also increase the probability of abrasion, reduce the brightness of the restoration, and raise the probability of discoloration. Due to its low pH, the gastric acid cycle can also cause material structures to become rough and discolored [14, 15]. Following the gastric acid cycle, we discovered a statistically significant difference between the materials; the compomers showed the greatest change in roughness in our within-group comparisons. Composite resin material demonstrated a better performance when compared to other materials. After being exposed to gastric acid for 24 hours, the microhardness and surface roughness of four different indirect composite resin materials were examined *in vitro*. The four groups of composite resin samples showed decreasing microhardness values and increasing roughness values [16]. In accordance with the results of this study, our analysis revealed an inverse association between surface roughness and microhardness values.

Microhardness is one of the most significant factors that should be taken into account when assessing the mechanical characteristics of dental materials. Low surface microhardness values are significantly correlated with inadequate wear resistance and a propensity for scratching, both of which can impair fatigue strength and result in the failure of tooth restorations [17, 18]. In our study, RMCIS, which had the lowest microhardness level at the beginning of the experiment, changed from having the lowest microhardness level to having the highest microhardness level at the conclusion of the gastric acid cycle. The composite material, however, experienced the greatest quantitative change, and the gastric acid cycle resulted in significant material effects.

SEM analysis was performed on two samples from each group in this study. We found that all of the experimental groups that had received gastric acid had visible cracks and gaps; these findings are similar to those reported previously. A study published in 2020 examined the impact of teeth brushing and gastric acid solution on several ceramic materials. The prepared samples were maintained in an artificial gastric acid solution for 9 days, six times a day (50 mL, 0.2% (w/v) sodium chloride (NaCl) in 0.7 (v/v) HCl, combined with 0.16 g pepsin powder, pH = 2). Following treatment, the teeth were cleaned with deionized water and set aside to dry. Furthermore, samples in a brushing cycle group underwent 100 brushing cycles over nine days. The samples were then analyzed for morphological alterations by optical microscopy and SEM. According to the findings of this earlier study, all samples exposed to gastric acid exhibited negative effects with regards to their brightness and surface roughness [19].

In this *in vitro* study, we investigated the surface roughness and hardness variations of three distinct restorative materials while simulating the oral environment with artificial saliva and GER with artificial gastric acid. However, because this study was carried out *in vitro*, it did not precisely replicate the oral environment. A main limitation in this situation is the inability to account for salivary flow rate and buffering capacity. However, the strength of our study is the inclusion

of the most frequently used restorative materials for pediatric patients and the unambiguous demonstration of the effects of gastric acid on the appearance and functionality of the materials using SEM images and experiments.

## 5. Conclusions

Dental materials are subjected to a variety of stress and abrasion factors in the oral environment. When considering the integrity of tooth tissue, it is important to evaluate the microhardness and surface roughness of the materials used. The results of our study demonstrate that restorative materials, which are often used in dental operations, change when exposed to gastric acid; surface roughness increases and microhardness decreases. These variations are more obvious in compomer and RMCIS materials. These modifications to the surface structures of materials will reduce the time these materials are exposed to the oral environment and make it easier for plaque to adhere to the product. In addition, the development of caries is more likely. Clinicians should take this condition into account when choosing restorative materials for pediatric patients.

## ABBREVIATIONS

SEM, Scanning Electron Microscopy.

## AVAILABILITY OF DATA AND MATERIALS

All data are presented in the article.

## AUTHOR CONTRIBUTIONS

MAI—contributed to design, manuscript draft; HO—contributed to study conception, and data checking; HNO and MK—contributed to data collection and all authors reviewed the manuscript.

## ETHICS APPROVAL AND CONSENT TO PARTICIPATE

Ethical approval of this study was obtained from the Ethics Committee of Necmettin Erbakan University, Faculty of Dentistry, Non-Pharmaceutical and Medical Device Research (2023/265).

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## CONFLICT OF INTEREST

The authors declare no conflict of interest.

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