

# The Effect of Daily Simulated Acidic Beverage Consumption on the Surface Roughness of Resin Luting Cements

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## Abstract

The purpose of this study was to evaluate the influence of acidic beverages on the surface roughness of resin cements. Eight groups of resin cement specimens, four each of conventional resin cement and self-adhesive resin cement, were prepared. Baseline linear surface roughness, measured by a contact profilometer, was determined for each specimen. Laboratory simulation of high beverage intake was performed on the specimen using chilled cola soft drink, lemon tea or orange juice for 10 days, and surface roughness again determined. The pH value and titratable acidity of each chilled beverage were also determined. Treatment with cola soft drink, lemon tea and orange juice significantly increased the surface roughness of the cements, suggesting that daily consumption of acidic beverages may roughen the surfaces. The lower the pH of the beverage is, the higher potential to roughen the cement surface increases.

**Key words:** Acidic beverage; Erosion; Resin cement; Surface roughness

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## Introduction

Tooth-colored restorations have gained in popularity for both patients and dentists. Although resin composite restorations offer such advantages as aesthetic and less invasive preparation,<sup>1</sup> they have several disadvantages based on their properties and restoration technique. Restoration with proper approximal contours, approximal contacts<sup>2</sup> and marginal ridges is difficult to create using a direct technique.<sup>3</sup> Other complications are postoperative sensitivity<sup>3</sup> and microleakage due to polymerization shrinkage.<sup>4</sup> In certain clinical situations, an indirect restoration, either resin composite or ceramic, might be more suitable in order to obtain a higher quality restoration.<sup>5</sup>

There are two main categories of ceramic restorations in dentistry: metal-ceramic and all-ceramic. Although the latter does not have a metal substructure, resulting in a more translucent and tooth-colored restoration,<sup>6</sup> marginal gap size is a significant drawback. A systematic review reported that the gap size between all-ceramic restorations and tooth structure ranged from 17 – 265  $\mu\text{m}$ ,<sup>7</sup> which is larger compared to the 30- $\mu\text{m}$  gap size found for gold restorations.<sup>8</sup> Because of the large gap size before cementation, resin-based cements are commonly used to lute all-ceramic restorations due to the cement's low solubility and good adhesion.<sup>9</sup>

In the oral environment, resin cements will inevitably encounter acid challenge from the metabolism of oral microorganisms and ingested drinks. This can

result in changes in physical properties such as translucency, hardness and surface roughness.<sup>10</sup> Increased surface roughness of luting cements at the marginal can adversely affect the restoration and adjacent teeth. The periodontal tissue can also be affected. A rough luting cement surface promotes microorganism accumulation, which can initiate nearby dental caries and promote gingival inflammation.<sup>11-13</sup>

Many studies have focused on the effects of acid on the physical properties of dental restorative materials, such as microhardness, surface roughness and erosion.<sup>10,14-22</sup> However, these studies simulated an acid challenge in a manner that did not imitate daily beverage consumption,<sup>10,14-22</sup> in that the specimen were continuously immersed in acid solutions or acidic drinks and did not consider the role of saliva, which can neutralize beverage acidity.<sup>23,24</sup>

The aim of this study was to investigate the effect of acidic beverages on resin cement surface roughness using a laboratory simulation of daily consumption. The null hypothesis was that the acidic beverages have no effect on surface roughness of resin luting cements.

## Materials and Methods

### Resin cements and beverages

The details of the resin cements and beverages (including artificial saliva) used in this study are presented in Tables 1 and 2, respectively.

**Table 1** Resin cements used in the study

Code	Material/Manufacturer	Type	Ingredients*	Lot No.
NX	NX 3 Nexus (clear)/ Kerr Corp., Washington, DC, USA	Conventional	<ul style="list-style-type: none"><li>• 20 – 40 % Methacrylate ester monomers</li><li>• Inert mineral fillers</li><li>• Activators</li><li>• Stabilizers</li><li>• Radiopaque agent</li></ul>	4851154

Table 1 (Continued)

Code	Material/Manufacturer	Type	Ingredients*	Lot No.
R2	RelyX Unicem 2 (translucent)/ 3M ESPE, St. Paul, MN, USA	Self-adhesive	<p><i>Base Paste:</i></p> <ul style="list-style-type: none"> <li>• 45 – 55 % Glass powder, surface modified with 2-propenoic acid, 2 methyl-3-(trimethoxysilyl) propyl ester and phenyltrimethoxy silane</li> <li>• 20 – 30 % 2-propenoic acid, 2-methyl-,1,1'-[1-(hydroxymethyl)-1,2-ethanediyl] ester, reaction products with 2-hydroxy-1,3-propanediol dimethacrylate and phosphorus oxide</li> <li>• 10 – 20 % Triethylene glycol dimethacrylate (TEGDMA)</li> <li>• 1 – 10 % Silane treated silica</li> <li>• &lt; 3 % Sodium persulfate</li> <li>• &lt; 3 % Oxide glass chemicals (non-fibrous)</li> <li>• &lt; 0.5 % Tert-butyl peroxy-3,5,5-trimethylhexanolate</li> </ul> <p><i>Catalyst Paste:</i></p> <ul style="list-style-type: none"> <li>• 55 – 65 % Silane treated glass powder</li> <li>• 20 – 30 % Substituted dimethacrylate</li> <li>• &lt; 5 % 1-benzyl-5-phenyl-barbic-acid, calcium salt</li> <li>• &lt; 5 % Silane treated silica</li> <li>• &lt; 5 % Sodium p-toluenesulfinate</li> <li>• &lt; 5 % 1,12-Dodecane dimethacrylate</li> <li>• &lt; 2 % Calcium hydroxide</li> <li>• &lt; 2 % Methacrylated aliphatic amine</li> <li>• &lt; 0.5 % Titanium dioxide</li> </ul>	518892

\* as reported in their respective Material Safety Data Sheets (MSDSs)

Table 2 Acidic beverages and artificial saliva used in the study

Liquid	Product	Manufacturer	Composition
Cola soft drink	Coca Cola	Thainamthip Manufacturing Co., Ltd., Bangkok, Thailand	Carbonated water, 10 % sugar, flavors

**Table 2 (Continued)**

Liquid	Product	Manufacturer	Composition
Orange juice	Tipco tangerine juice	TIPCO F&B Co., Ltd., Phra Nakhon Si Ayuthaya, Thailand	100 % tangerine juice
Lemon tea	Lipton ice tea	Sermsuk Beverage Co., Ltd., Chon Buri, Thailand	10.5 % sugar, 0.13 % instant powdered tea, 0.02 % lemon-flavored powder
Artificial saliva	Artificial saliva	Department of Pharmaceutical Sciences, Faculty of Dentistry, Chulalongkorn University, Bangkok, Thailand	KCl BP 0.75 g, MgCl <sub>2</sub> BP 0.07 g, CaCl <sub>2</sub> BP 0.199 g, K <sub>2</sub> HPO <sub>4</sub> USP 0.965 g, Sodium CMC BP 6.0 g, KH <sub>2</sub> PO <sub>4</sub> 0.439 g, 70 % Sorbitol BP 36 g, Paraben 18.20 ml, Deionized water to reach 1,200 ml

BP = British Pharmacopoeia, USP = United States Pharmacopoeia, CMC = Carboxymethyl cellulose

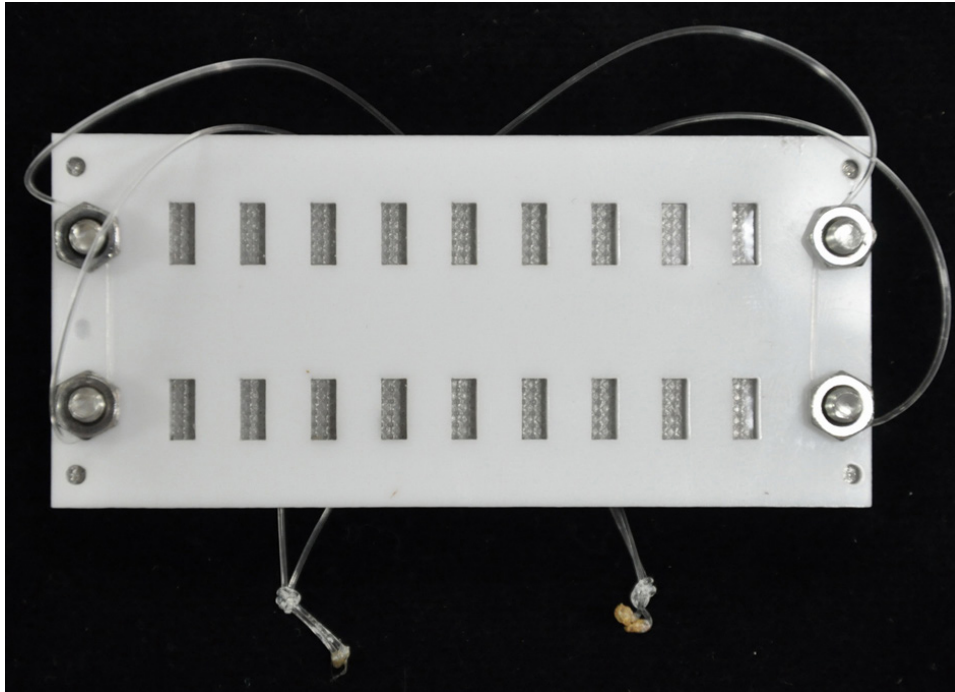
### Specimen preparation

Eight specimen blocks were prepared. Each block consisted of a base plate of #304 stainless steel covered its surface with a plastics film, and an upper plastics plate with 2-mm deep rectangular cavities, 2 mm x 5 mm. The two plates were bolted together, and nylon loops attached to the bolts for carrying the block during drinking simulation (Fig. 1). Four blocks received NX, while the rest received R2. Each cement was mixed according to its manufacturer's instructions and placed into the cavities, in a (37 ± 1) °C chamber (THCC 575, King Mongkut's Institute of Technology Ladkrabang, Bangkok, Thailand). To minimize porosity in the cements, NX was mixed using an automix syringe, and R2 was hand-mixed with circular motion. The cement was loaded into the cavities, covered with a celluloid strip and a 1-mm thick transparent glass slide. A 1-kg mass was placed on the slide for 10 seconds to eliminate excess cement. The weight was removed, the cement in each cavity was polymerized using a LED light curing unit (Curing Light 2500, 3M ESPE, St. Paul, MN, USA) for

20 s according to the manufacturer's recommendation, and the glass slide and the celluloid strip were removed. The light intensity of the curing unit was measured with a radiometer (Optilux Radiometer, Kerr Corporation, Orange, CA, USA) every 10 uses to ensure that an intensity more than 600 mW/cm<sup>2</sup> was maintained. The specimen surfaces were observed using a stereomicroscope (ML 9300, MEIJI, Japan) at 40x magnification to identify defective specimens, and any such were excluded. Thirteen specimens were simple randomly selected as tested specimen according to the sample size calculation from a pilot study. Each block was kept in artificial saliva at (37 ± 1) °C for 24 h.

### pH and titratable acidity determination

The pH of each cold drink was measured with a pH meter (Type 420A, Orion Research Inc., Boston, USA). Titratable acidity was determined by the volume of 0.2 M, (25 ± 1) °C NaOH which needed to be added to 50 ml of the cold drink to raise its pH to 7.0. Three measurements were repeated and the mean calculated.



**Figure 1** Customized block used in the study

#### Surface roughness measurement

After 24-h storage, the block was rinsed with distilled water, air-dried with air from a triple syringe for 1 min and kept at room temperature ( $25 \pm 1$  °C) for at least 1 h prior to insertion into a jig positioned on a contact profilometer (TalyScan 150, Taylor Hobson Limited, England). The jig was fabricated to ensure that the block was at exactly the same position at each measurement. A stylus with 2- $\mu$ m diameter diamond tip scanned a 1 x 4 mm area located at the center of each selected specimen's surface. The X- and Y-axis spacing parameters were set at 5  $\mu$ m with a scanning speed of 3,000  $\mu$ m/s.<sup>26</sup> Two hundred-and-one linear profiles were traced, and 16 profiles were randomly selected. The mean surface roughness (Ra) was calculated for each specimen from these selected profiles.

#### Drinking simulation

Each specimen block was alternately immersed

in 45 ml of cold beverage (5 s) followed immediately by immersion in warm artificial saliva (10 s), and the cycle repeated 10 times in a ( $37 \pm 1$ ) °C chamber. The cold drinks were chilled to ( $4 \pm 1$ ) °C for at least 1 hour and the artificial saliva was kept in a ( $37 \pm 1$ ) °C chamber for at least 30 minutes prior to use. The block was stored in artificial saliva in a ( $37 \pm 1$ ) °C incubator (Contherm 1200, Contherm, New Zealand) for 3 h before a second and third simulation cycles were performed. After three drinking simulations, the block was kept in artificial saliva at ( $37 \pm 1$ ) °C for 18 h and the simulation cycle repeated on the next day (Fig. 2). The negative control groups were immersed only in artificial saliva, and the artificial saliva was replaced three times daily, twice before the 3-h storages and once before the 18-h storage. The simulation cycle was repeated for 10 days.<sup>10</sup> After the 10-day drinking simulation, Ra measurement was repeated using the same 16 linear profiles as described above.

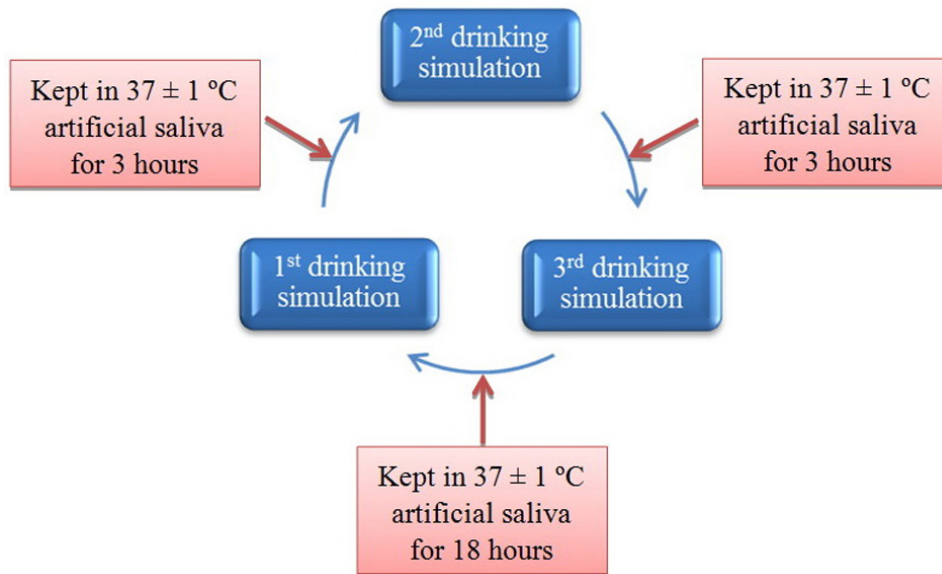


Figure 2 Daily drinking simulation protocol used in the study. The protocol was repeated for 10 days.

### Statistical analysis

Statistical analyses were conducted using computer software (IBM SPSS Statistics 17.0, International Business Machines Corp., Armonk, NY, USA). Sample distribution and homogeneity was analyzed. A normal and homogeneous distribution were confirmed, therefore the data were analyzed using the two-way ANOVA parametric test. Multiple comparisons were made using the Dunnett T3 post hoc test. In addition, the paired T-test was also used. Statistical significance was set at  $p < 0.05$ .

## Results

### pH and titratable acidity

The results of the pH analysis indicated that cola soft drink had the lowest pH, followed by lemon tea, orange juice and artificial saliva (Table 3). The order of titratable acidity of the beverages was cola soft drink, orange juice, lemon tea.

Table 3 Average pH value and amount of NaOH used to determine titratable acidity

Liquid	pH value	Required amount of 0.2 M NaOH (ml)
Cola soft drink	2.6	19.7
Orange juice	3.6	17.8
Lemon tea	3.1	9.5
Artificial saliva	7.0	-

### Surface roughness measurement

The results of the surface roughness measurements before and after the drinking simulation are shown in Table 4. The surface roughness of specimens before the

drinking simulation were not significantly different ( $p = 0.567$ ), thus the surface roughness of the samples determined after the simulation were analyzed as ultimate outcomes.

**Table 4** Average surface roughness ( $\mu\text{m}$ ) (standard deviation) of each group before and after the drinking simulation ( $N = 13$ )

Group	Sequence	Cola soft drink	Orange juice	Lemon tea	Artificial saliva
NX	Before treatment	0.016 (0.002) <sup>a</sup>	0.016 (0.001) <sup>a</sup>	0.016 (0.001) <sup>a</sup>	0.016 (0.001) <sup>a</sup>
	After treatment	0.029 (0.005) <sup>b</sup>	0.019 (0.002) <sup>c</sup>	0.026 (0.002) <sup>d</sup>	0.018 (0.001) <sup>c</sup>
R2	Before treatment	0.017 (0.002) <sup>a</sup>	0.016 (0.002) <sup>a</sup>	0.016 (0.001) <sup>a</sup>	0.016 (0.001) <sup>a</sup>
	After treatment	0.028 (0.004) <sup>b</sup>	0.019 (0.002) <sup>c</sup>	0.025 (0.002) <sup>d</sup>	0.017 (0.001) <sup>c</sup>

The same superscript letter denotes no statistical difference between the groups.

After the drinking simulation, all the beverages, including artificial saliva, significantly increased the surface roughness of the resin cements ( $p < 0.001$ ). Cola soft drink had the greatest roughening beverage effect, followed by lemon tea. Orange juice and artificial saliva significantly increased the roughness of the tested materials but to a significantly lesser extent than that of cola soft drink and lemon tea. Two-way ANOVA revealed the influence of the beverage type on the increase of surface roughness ( $p < 0.001$ ), while cement type did not ( $p = 0.299$ ). In addition, there were no significant interactions between these two factors ( $p = 0.889$ ).

### Discussion

The present study investigated the ability of cola soft drink, lemon tea and orange juice to roughen the surface of conventional and self-adhesive resin cements using a laboratory simulation of daily drinking. We found that all the tested beverages were able to

increase the surface roughness of the resin cements, the ability being related to the pH. Beverages with lower pH caused a greater increase in surface roughness. The pH value of the beverages used in this investigation was comparable (within  $\pm 0.25$ ) to those used in previous studies<sup>15,27-30</sup> which validates the use of these beverages in our study.

Our specimen preparation technique produced smooth surfaces; hence, additional polishing was not required.<sup>23-25</sup> However, lack of polishing leaves a resin-rich and oxygen-inhibited layers on the top of the surface. Although these layers may not represent the actual properties of the resin-based materials, in clinical practice it cannot be removed from some specific sites, including approximal and subgingival areas. In addition, as dentists always try to eliminate excessive cement before complete polymerization is achieved; additional polishing may not be required after cementation. In the present study the specimen surface can represent these clinical situations.

Previous studies that focused on the effect of acids or acidic beverages on the erosion of dental materials<sup>10,14-22</sup> did not simulate realistic daily drinking behavior. These studies continuously immersed the materials in erosive liquids for a long period of time, without alternating with saliva as would occur *in vivo*. Drinking behavior generally consists of taking in a small amount of beverage that is briefly held in the oral cavity and then swallowed before the next drink is taken. In addition, there is typically a pause between drinking cycles, where the saliva in the oral cavity washes away the beverage which is retained on tooth structure and restorative materials, and also neutralizes the beverage's acidity.<sup>23,24</sup> The present study was designed to repeatedly alternate specimen immersion between a cold beverage and warm artificial saliva in order to mimic real-life beverage consumption behavior and the effects of saliva. Many of the protective effects of saliva, including its buffering capacity or acquired pellicle are not easily reproduced in laboratory studies.<sup>24</sup> In addition, the drinking consumption protocol in the present study was designed to replicate high beverage intake.<sup>10</sup> Therefore, our protocol might slightly overestimate the results of a typical drinking pattern.

For surface roughness measurement, two hundred-and-one linear profiles were traced, and 16 profiles were randomly selected because our pilot study determined that this amount of profiles were sufficient to obtain an average Ra within  $\pm 5\%$  error at a 95% confidence interval. When comparing the surface roughness before and after the drinking simulation within each group, all tested drinks increased the surface roughness of the materials. This can be explained by the sorption properties of resin-based materials,<sup>23</sup> which is a complex process and usually occurs in the resin matrix. Due to the plasticizing property of water, immersion of the resin-based materials into water-based liquids can facilitate polymer chain movement. When water is absorbed much more than the space provided in the matrix, the polymer chains are displaced from each other, causing volumetric expansion.<sup>31</sup> In addition, water can cleave the polymers

by hydrolysis of the ester group of the dimethacrylate monomers.<sup>23,24</sup> The material surface can become rougher because the damaged polymers and unpolymerized monomers are dissolved and washed away. The reason for the increase in surface roughness observed in our study of the specimens immersed only in the artificial saliva might be similar to that previously described, as the artificial saliva is a neutral water-based solution. In addition, precipitation of some ions in the artificial saliva on the resin cement surface might facilitate the roughening process. As dental materials in the oral cavity are always in contact with saliva, the negative control group was designed such that the specimens were only immersed in the artificial saliva. Because of this observation, the roughness measured after the drinking simulation in the present study is a combination of the roughening potential of the acidic drink and artificial saliva.

Comparing the surface roughness between the tested groups after the drinking simulation, we found that the roughening potential of an acidic beverage was related to its pH; the lower the pH, the greater the increase in surface roughness. Several previous studies<sup>19,23,32</sup> attributed such results to the buffering capacity of the solutions, which was believed to be a better roughening indicator because it represented the total acidity of the solutions. However, our results contradicted this explanation, even orange juice, with a higher titratable acidity than that the lemon tea, it was less able to increase surface roughness. Although beverages with higher buffering capacity could better resist pH change induced by saliva than those with lower buffering ability,<sup>32</sup> pH, which represents the active acidity, should be more important when a brief immersion in acid occurs. During typical drinking behavior, a beverage is only briefly retained in the oral cavity before swallowing; therefore the pH of a drink might be a more reliable indicator of its roughening potential. In addition, the types of acids in each beverage and their ability to react with the materials may also have influence on the roughening ability of the acidic drinks. Coca Cola has phosphoric and carbonic acids as predominating acidic



ingredients, while there is citric acid in orange juice and lemon tea. Although this might be a confounding factor, the main purpose of our study is to mimic a real-life situation as closely as possible. We elected to use commercially available acidic beverages which have different types and amount of acids, instead of laboratory preparations of simulated acidic drinks with different pH values.

Although there were differences in composition of tested cements in the present study, which may create the different in cements' properties, the type of resin cement was not a significant factor influencing the increase in surface roughness change. This may be due to the short period of drinking simulation. Silva *et al*<sup>33</sup> reported that long-term immersion of resin-based cements in organic acids increased cements' degradation. Han *et al*.<sup>22</sup> reported a different resistances to surface roughness change among the tested resin composites. In that study, tested conventional and flowable resin composites were categorized into two groups regarding the amount of filler content. The types of fillers used in resin composite with relatively high filler contents are barium glass and silica. The amount of fillers in their study ranged from 61.6 – 78.2 w/w %. For the resin composites categorized as relatively low filler contents, the fillers are glass, silica, zirconia, and fluoroaluminosilicate, and the percentage of fillers ranged from 42.7 – 48.3 w/w %. After a 14-day immersion in acidic beverages, they found different resistances to surface roughness change among the tested resin composites. They suggested that the amount of filler in each resin composite influenced resistance to roughening; the more filler in the material, the less the roughness increased. The reason for the different results between their study and ours may be due to different in type and properties of material. More important, there was a different in experimental designs, as our actual immersion time in the acidic beverages was much shorter.

Our results indicate that the ability of saliva to dilute or wash the acids away during alternate immersions should be taken into account in future studies. In addition,

a future study should employ a prolonged drinking simulation period, which might better clarify the effect of resin cement type on resistance to roughening.

## Conclusion

This study demonstrates the ability of acidic beverages to increase the surface roughness of the resin cements used in this study. The drink with lower pH value had more ability to increase surface roughness more than that with higher pH value. The present study also shows that the type of resin cement had no significant influence on the ability to resist change in surface roughness.

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