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# Effects of Ethanol on the Surface and Bulk Properties of a Microwave-Processed PMMA Denture Base Resin

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## Abstract

**Purpose:** This study evaluated the effect of different concentrations of ethanol on hardness, roughness, flexural strength, and color stability of a denture base material using a microwave-processed acrylic resin as a model system.
**Materials and Methods:** Sixty circular (14 × 4 mm) and 60 rectangular microwave-polymerized acrylic resin specimens (65 × 10 × 3 $mm^3$) were employed in this study. The sample was divided into six groups according to the ethanol concentrations used in the immersion solution, as follows: 0% (water), 4.5%, 10%, 19%, 42%, and 100%. The specimens remained immersed for 30 days at 37°C. The hardness test was performed by a hardness tester equipped with a Vickers diamond penetrator, and a surface roughness tester was used to measure the surface roughness of the specimens. Flexural strength testing was carried out on a universal testing machine. Color alterations (ΔE) were measured by a portable spectrophotometer after 12 and 30 days. Variables were analyzed by ANOVA/Tukey's test ($\alpha = 0.05$).
**Results:** For the range of ethanol-water solutions for immersion (water only, 4.5%, 10%, 19.5%, 42%, and 100%), the following results were obtained for hardness (13.9 ± 2.0, 12.1 ± 0.7, 12.9 ± 0.9, 11.2 ± 1.5, 5.7 ± 0.3, 2.7 ± 0.5 VHN), roughness (0.13 ± 0.01, 0.15 ± 0.07, 0.13 ± 0.05, 0.13 ± 0.02, 0.23 ± 0.05, 0.41 ± 0.19 $\mu$m), flexural strength (90 ± 12, 103 ± 18, 107 ± 16, 90 ± 25, 86 ± 22, 8 ± 2 MPa), and color (0.8 ± 0.6, 0.8 ± 0.3, 0.7 ± 0.4, 0.9 ± 0.3, 1.3 ± 0.3, 3.9 ± 1.5 ΔE) after 30 days.
**Conclusions:** The findings of this study showed that the ethanol concentrations of tested drinks affect the physical properties of the investigated acrylic resin. An obvious plasticizing effect was found, which could lead to a lower in vivo durability associated with alcohol consumption.

Acrylic resins are widely used for denture fabrication.[1] A drawback of these materials is that their esthetic, physical, and mechanical properties change rapidly over time in the oral environment. Typical changes are due to sorption, which depends upon liquid absorption and adsorption.[2] Furthermore, it is well known that foods and drinks contain solvents that can chemically soften polymeric dental materials.[3]

Ethanol and water are two solvents with deleterious effects for dental materials. Akova et al[4] stated that beverages can soften, degrade, and age dental composites for interim prostheses. The hardness of provisional materials is notably lowered after immersion in food-simulating liquids containing ethanol.[5] Ethanol also enhances the plasticization of the crack tip in poly(methyl methacrylate) (PMMA)[6] and causes irreversible dental composite degradation by penetrating the matrix and expanding the space between polymer chains.[7] Water, on the other hand, is a complex solvent because of its possible strong interaction with the polymer, due to its polarity and ability to form hydrogen bonds. Thus, there is a tendency for it to cluster and cause plasticization of the material matrix.[8]

A high percentage of elderly people consume ethanol-containing beverages.[9] Ethanol is present in several drinks and has been shown to soften the denture base PMMA.[10] The mechanical behavior of PMMA can be affected by the presence of ethanol according to the following mechanisms:[11] first, it presents a long-term dissolution on the surface of unloaded resin; second, a stress crazing effect is expected at points of high stress concentration.

In general, there are scarce reports of the effect of ethylic solutions simulating food contents on denture base materials, mostly restricted to mechanical properties. Regarding other properties, it has been described that alcoholic

beverages, like wine, are able to produce pronounced color changes in acrylic resin;[12] however, it is not known to what extent this effect is due to ethanol or to the presence of coloring substances.

The aim of this study was to evaluate the effect of different concentrations of ethanol in water on hardness, roughness, flexural strength, and color stability of a denture base material using a microwave-processed acrylic resin as a model system. The null hypotheses were that ethanol concentration has no effect on the color stability, hardness, roughness, and flexural strength of the tested acrylic resin.

## Materials and methods

Exposure to alcoholic beverages could potentially alter surface hardness during adsorption. Furthermore, it may cause some surface dissolution, affecting smoothness, and alcohol's absorption into the bulk of the PMMA could reduce its flexural strength and have chemical effects on the polymer, causing color changes. Different ethanol concentrations were selected to investigate the effects on those properties. Concentrations of 4.5%, 10%, 19%, and 42% (v/v) were used to simulate the alcoholic graduation of beer, wine, port wine, and distilled beverages, respectively.

The sample comprised 60 circular acrylic resin specimens for hardness and color stability assessments. Sixty other rectangular specimens were employed for flexural strength and roughness testing. The microwave-polymerized acrylic resin Onda-Cryl (Artigos Odontológicos Clássico Ltda, São Paulo, Brazil; lot number: 150107—monomer and 12507.0—polymer) was used in this study. Metal master patterns were individually invested in high-viscosity silicone (Zetalabor, Zhermack S.p.A, Badia Polesine, Rovigo, Italy) and supported by type III dental stone (Herodent, Vigodent SA Ind Com, Rio de Janeiro, Brazil) within flasks. Each flask contained six circular ($14.0 \times 4.0$ mm) or two rectangular patterns ($65.0 \times 10.0 \times 3.3$ mm$^3$). After the dental stone was set, the flasks were separated, and the master patterns were removed from the silicone mold.

The denture base resin was mixed according to the manufacturer's recommendations. A portion of monomer (7 mL) and polymer (21 mL) was mixed for each flask, thus a dough stage was reached, and then it was placed into the molds. A pneumatic press (PM-2000, Techno Máquinas Ltda, Vinhedo, Brazil) was used to pack the denture base resin first at 500 kgf and then at 1000 kgf for 60 minutes. The resin was polymerized in a microwave oven (ME28S, Electrolux SA, Manaus, Brazil) according to the manufacturer's recommendations—320 W for 3 minutes + 0 W for 4 minutes + 720 W for 3 minutes. The specimens were bench cooled overnight before deflasking. The excess resin was trimmed with a bur (Maxi-Cut, Malleifer SA, Ballaigues, Switzerland). Final dimensions and finishing were performed in a horizontal polisher (model APL-4, Arotec, São Paulo, Brazil) on all sides to eliminate any visible irregularities with a series of wet/dry sandpaper (180-, 220-, 360-, and 400-grit). Specimen dimensions were confirmed with a manual caliper (model 43175/301, Tramontina SA, Carlos Barbosa, Brazil).

Specimens were randomly divided into six groups of ten specimens for each format according to the water-ethanol solution for immersion. The first test group involved immersion in water alone and served as a negative control. Four other groups were immersed in 4.5%, 10%, 19%, and 42% alcoholic solutions. The last group was immersed in absolute ethanol (Labsynth Produtos para Laboratório, Diadema, Brazil; lot number: 98579). The groups were individually stored in covered glass bottles containing 250 mL of each liquid at $37.0 \pm 1.0^{\circ}$C for 30 days. The specimens were removed from the bottles only during the tests; when not used, they were kept immersed in the respective water-ethanol solution.

Surface microhardness was determined using a hardness tester (Shimadzu HMV-2) equipped with a Vickers diamond. Testing was conducted using a 25 g load and a 30-second contact. Eight indentations were made on each specimen. The individual recorded value was the average of the eight values obtained. The test was conducted on color stability of specimens immediately after the 30-day colorimetric assessment.

The Surface Roughness Tester SJ-201P (Mitutoyo Corp, Kawasaki, Japan) was used to measure the specimens' surface roughness after 30 days of immersion. The profiler was set to move a diamond stylus across the specimen surface under a constant load. The scanning duration for each line was 10 seconds with a constant force of 4 mN (0.4 gf) on the diamond stylus (5 $\mu$m radius). The surface morphology was measured with a linear variable differential transformer. The surface roughness was derived from computing the numerical values of the surface profile. The Ra value describes the overall roughness of a surface and is defined as the mean value of all absolute distances of the roughness profiles from the mean line within the measuring distance. Five measurements with a length of 4.8 mm and incremental distance of 1 mm between each scanning line were carried out for each specimen. The vertical resolution was 0.01 $\mu$m, which also represents the accuracy of Ra. The mean Ra was calculated from five lines as the mean roughness of the specimen.

Following roughness testing, rectangular specimens were immediately submitted to the flexural strength assessment. The flexural strength of each group was measured using a three-point bending test in a universal testing machine (EMIC, São José dos Pinhais, Brazil) at a crosshead speed of 1 mm/min. Stress was applied until fracture by a centrally located rod connected to a 50 kgf load cell. Flexural strength (S) was calculated using the following formula: $TS = 3WL/2bd^2$, where W is the maximum load before fracture, L is the distance between supports (50 mm), b is the specimen width, and d is the specimen thickness. Yield strength and modulus of elasticity for each specimen were also recorded. The crosshead movement of the machine was used to determine the modulus of elasticity for each specimen.

The color change ($\Delta$E) was measured according to Ma et al.[13] A portable spectrophotometer was employed (Color Guide 45/0, BYK-Gardner Latin America, Santo André, Brazil), and measurement was carried out in the center of each circular specimen. This instrument was used to quantify the tristimulus values and calculate $\Delta$E from data obtained before specimen immersion in the ethanol solutions, after 12 days and after 30 days. After initial color measurements, specimens were immersed in the solutions.

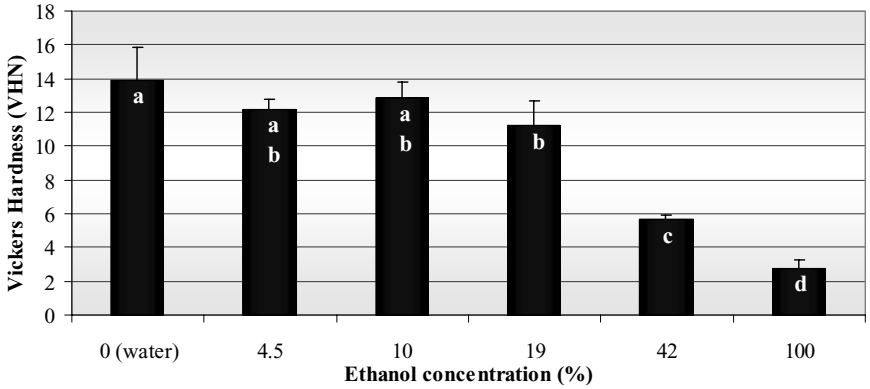


**Figure 1** Mean values for Vickers hardness according to different ethanol concentrations. Error bars illustrate standard deviations.

The magnitude of the total color difference is formulated by a single number, ΔE:

$$\Delta E = [(\Delta L^*)^2 + (\Delta a^*)^2 + (\Delta b^*)^2]^{1/2}$$

where L* stands for lightness, a* for redness-greenness, and b* for yellowness-blueness.

Two circular specimens were further studied for each group. These specimens were subsequently processed for SEM. One of the flat surfaces was coated with gold and observed in a scanning electron microscope (EVO 50, Carl Zeiss SMT, Inc., Thornwood, NY) in high-vacuum mode at 20 kV.

Data obtained for color stability were expressed as mean values, and differences among groups were tested by means of two-way ANOVA. The other quantitative variables were assessed by one-way ANOVA. Multiple comparisons were performed according to the Tukey HSD test. All analyses were performed at a 95% level of confidence. Data were analyzed with SPSS for Windows software (version 12.0.0, SPSS, Inc., Chicago, IL). A qualitative approach was used for the evaluation of photomicrographic results.

## Results

The Vickers hardness test showed that ethanol was able to cause superficial modifications in the specimens. Those modifications were significant (one-way ANOVA, F = 77.4, $p < 0.001$) and inversely proportional to ethanol concentration (Fig 1). Low concentrations, that is, 4.5% and 10%, did not result in significant changes when compared to the negative control.

Results for surface roughness were significantly influenced by the tested ethanol concentrations (one-way ANOVA, F = 15.3, $p < 0.001$). Nevertheless, the negative control and every concentration simulating alcoholic beverages were not significantly different (Fig 2). Mean Ra for the 100% concentration was higher than those found for the other five groups.

Table 1 presents results for modulus of elasticity, flexural strength, yield strength, and respective inferential analyses. The mean flexural strength and modulus of elasticity for the specimens immersed in 100% ethanol were significantly lower than those of the other groups. A trend for higher yield strength values was found for the 10% group. This variable showed lower values for each concentration above 10%. Negative control and

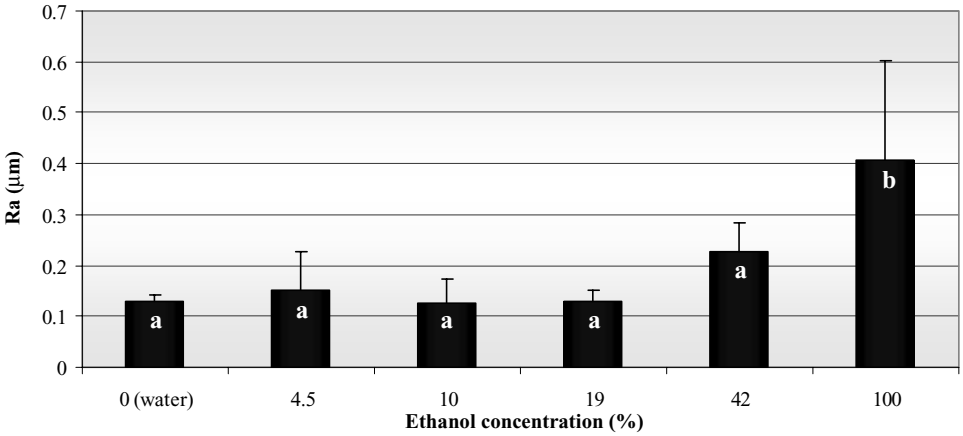


**Figure 2** Mean values for surface roughness (Ra) according to different ethanol concentrations. Error bars illustrate standard deviations.

**Table 1** Mean results (± standard deviation) for the flexural strength assessment according to different ethanol concentrations in volume (means with the same capital letter within the column are not significantly different)

| Ethanol concentration (%) | | Modulus of elasticity (MPa) | Flexural strength (MPa) | Yield strength (MPa) |
|---|---|---|---|---|
| 0 (water) | | 2773 (246) A | 90 (12) A | 69 (7) ABC |
| 4.5 | | 2925 (311) A | 103 (18) A | 72 (10) AB |
| 10 | | 2771 (615) A | 107 (16) A | 76 (10) A |
| 19 | | 2483 (630) A | 90 (25) A | 61 (18) BC |
| 42 | | 2329 (625) A | 86 (22) A | 55 (13) C |
| 100 | | 545 (165) B | 8 (2) B | 4 (1) D |
| One-way ANOVA | F | 35.1 | 43.2 | 60.2 |
| | *p* | <0.001* | <0.001* | <0.001* |

*Significant difference, $p < 0.05$.

the 4.5% group resulted in intermediate yield strength values between those of the 10% and 19% groups.

Two-way ANOVA revealed that both variation factors tested, that is, concentrations of ethanol and immersion times, significantly influenced the obtained values for ΔE (groups: $F = 50.0$, $p < 0.001$; Time: $F = 11.2$, $p = 0.001$). Furthermore, a significant interaction was found between the two factors ($F = 3.1$, $p = 0.012$). Figure 3 presents the mean ΔE for the six groups and results of the post hoc comparison. Measurements for the five concentrations from zero to 42% were similar, regardless of the time. Immersion time only influenced coloration of the specimens immersed in absolute ethanol, with more color changes observed during the 30-day assessment. This was the only treatment that resulted in visually perceptible alterations.

Figure 4 presents a micrograph illustrating surface characteristics after immersion in the 100% concentration. This image is representative of the tested specimens for that group, which showed similar results. SEM evaluation at 1000× magnification showed a smooth aspect for the specimens immersed in distilled water and 4.5% and 10% ethanol. A slightly more irregular surface was found for the 19% and 42% groups. Polishing defects were more evident for both groups. A distinct feature was evident for acrylic resin submitted to immersion in 100% ethanol. The pre-polymerized PMMA pearls became apparent on the surface.

## Discussion

From the color stability assay, the six groups demonstrated some degree of change. Since the colorimetric observation demonstrated that ΔE was similar for distilled water and ethanol concentrations from 4.5% to 42%, color changes were probably due to liquid sorption regardless of the dissolutive ethanol activity. Among the multiple factors associated with acrylic resin's color, water sorption and chemical reactivity are considered strongly relevant.[15] The effect shown by 100% ethanol was sound, and the mean ΔE was almost twice the values after a month of immersion as it was at 12 days. This fact indicates that ethanol exhibits a sorption degree much higher than that of water, and it was not stable before 30 days. On the other hand, water sorption of microwavable acrylic resin tends to be stable after approximately 7 days.[16]

Results for the 100% group were much above the threshold for visually perceived changes ($\Delta E > 1.0$),[13,14] and specimens presented a whitened aspect. The other group results were near

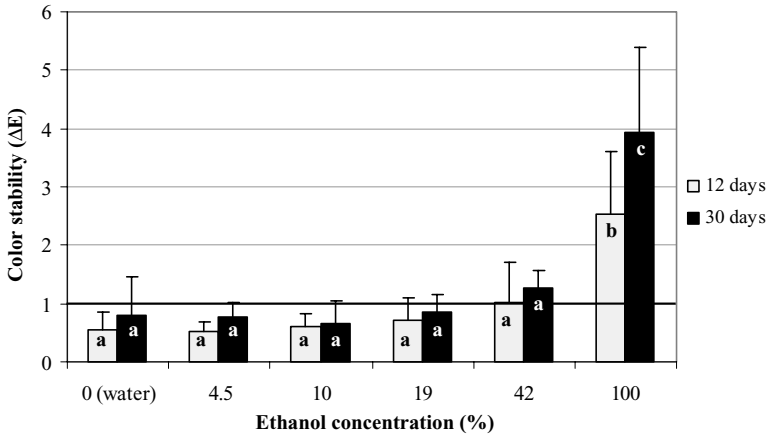


**Figure 3** Mean values for color stability according to the different ethanol concentrations and immersion times. Error bars illustrate standard deviations. The horizontal line represents the cutpoint for color change perception by human eyes ($\Delta E = 1.0$).[13,14] Mean values with the same letter are not significantly different (Tukey HSD test, $\alpha = 0.05$).

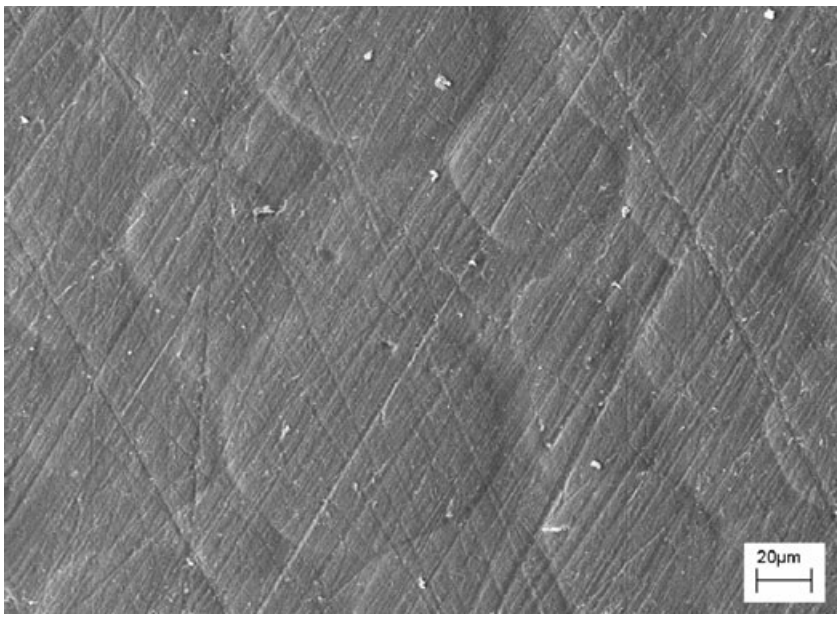


**Figure 4** Typical SEM image of specimen flat surfaces following immersion in the 100% ethanol solution.

that threshold, and some clinical extrapolations can be deduced. This study was undertaken to evaluate ethanol contents similar to the concentration of alcoholic beverages. Hypothetically, if beverage intake by regular drinkers is similar to that estimated by Guler et al[12] for coffee, 30 days of immersion simulates 2.5 years of regular consumption. Thus, the ethanol present in drinks cannot be considered a significant cause for color changes in denture bases. It is most likely that discoloration is a consequence of other substances present in other beverages, like coffee, juices, or wine.[17] In this study, the immersion time for coffee was used for other liquids so as to facilitate comparisons and due to the lack of a more adequate standard.

Another important finding is that the reduction in hardness was proportional to ethanol concentration. When compared to the control group, mean VHN values were significantly inferior for the 19% group and even lower for greater concentrations. This confirms the previously described softening effect of ethanol over acrylic resins.[11,18,19] It seems that ethanol, as well as water, helps to push the polymer chains apart[18] and allows them to slide (plastically deform) more easily.[19] This decrease in VHN values for PMMA is also caused by the plasticization effect enhanced by the ethanol,[6] which penetrates the matrix and expands the space between the chains.[7] This difference might be even greater, as long as one of the hardness test method limitations is associated with the accuracy of hardness indentations after the indenter is removed. These measurements can be affected by the material's elastic recovery,[20] which could have been important after plasticizing by solvents such as methanol and ethanol.[21]

The present results indicate that material surface roughness was not affected by 0% to 42% solutions; however, slight surface changes were observed by SEM images for the 19% and 42% groups, which implies that changes were approximately linear with concentration. Those irregularities were not considerable in terms of influencing mean Ra. According to the results of Quirynen et al,[22] an increase in bacterial colonization would be expected to occur on surfaces with Ra roughness values of 2.2 $\mu$m, and the median Ra for the 42% group was 0.22 $\mu$m. Nevertheless, ethanol's dissolutive characteristics could interact with the effects of other agents, like denture hygiene methods or diet. Denture base acrylic resin becomes rougher when subjected to brushing.[23] This roughening effect can be enhanced by ethanol consumption, since it altered the three surface variables assessed in this study, that is, hardness, roughness, and SEM aspect. Possible consequences of this interaction are discomfort[2] and higher microbial colonization rates.[24]

Another confirmation of the plasticizing effect of ethanol on acrylic resins[11,19] was the result for flexural strength testing. The dramatic change in the mechanical properties of acrylic resin occurs somewhere between 42% and 100% ethanol exposure. The 100% group showed notably lower values for flexural strength, modulus of elasticity, and yield strength than did the other groups. The first two variables presented a similar behavior after immersion in water and 4.5% to 42% ethanol; however, an interesting result was shown with yield strength. At lower concentrations, ethanol seems to increase the resilience of the acrylic resin tested, as the maximal stress reported (flexural strength) was stable, but maximal stress in elastic regimen (yield strength) increased. Concentrations at 19% or more, on the other hand, resulted in lower yield strength values. A decrease in flexural strength of denture base acrylic resin can result in greater fracture incidence by impact or occlusal forces.[25] Ethanol probably has no relevance when breakage of denture bases is concerned; however, higher degrees of plastic deformation might be a consequence of continuous consumption of distilled beverages.

Modulus of elasticity was calculated by means of the crosshead movement, and this is a relevant limitation. Variations in the cross-sectional area of specimens during the test would not be significant if associated with small strain;[26]

however, the specimen was subjected to plasticization by water and/or ethanol. High elongation values can result in dramatic changes in the cross-sectional area, as observed for tissue conditioners.[27] As real deformation was not assessed, modulus of elasticity for the 100% group should be interpreted with caution. Mean results for the other groups were relatively high and near to the observed modulus for dry PMMA.[28] As the differences among them were not significantly different, the results can be interpreted accordingly.

Another possible limitation is the loss of ethanol or water from the specimens during each test. This was minimized by means of the removal from the immersion media only during the test. A substantial loss of solvents would be only expected after a few hours at high temperatures such as 100°C,[29] so it is expected that this loss is negligible. The most important limitation of this study is the in vitro design employed. Patients do not continuously expose their dentures to ethanol solutions. Its in vivo action is intermittent, and some degree of adsorbed ethanol elution must occur between the doses. At this time, acrylic resin will adsorb water molecules, which act as a plasticizer agent[30,31] to a lesser extent than ethanol. This way, clinical effects of ethanol consumption on denture bases are probably less than those found in vitro. Further observational clinical studies could clarify this, but it can be inferred that concentrations found in beers and wines do not cause damage to PMMA-based denture base materials. Those odds are possibly associated with the consumption of distilled beverages. The main mechanism of likely clinical complications associated with ethanol consumption is based on the plasticizing effect.[11,19] The most significant consequence of this effect on the material is decreased hardness and yield strength, which may lead to lower clinical durability. Other studies should aim to evaluate the interaction between ethanol exposure and other factors, such as brushing.

## Conclusions

The present study showed that immersion in different ethanol concentrations affects several physical properties of a microwave-processed denture base resin. Ethanol showed a dissolutive and plasticizing effect over the tested material. Changes in surface properties were linear over most ethanol concentrations but showed a rapid change starting in the neighborhood of 42% for flexural strength and color stability.

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