# Effect of Disinfectants on the Hardness and Roughness of Reline Acrylic Resins

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***Purpose:*** **Potential effects on hardness and roughness of a necessary and effective disinfecting regimen (1% sodium hypocholorite and 4% chlorhexidine) were investigated for two hard chairside reline resins versus a heat-polymerizing denture base acrylic resin.**

***Materials and Methods:*** **Two standard hard chairside reliners (Kooliner and Duraliner II), one heat-treated chairside reliner (Duraliner II +10 minutes in water at 55°C), and one standard denture base material (Lucitone 550) were exposed to two disinfecting solutions (1% sodium hypochlorite; 4% chlorhexidine gluconate), and tested for two surface properties [Vickers hardness number (VHN, kg/mm$^2$); Roughness (Ra, $\mu$m)] for different times and conditions (1 hour after production, after 48 hours at 37 $\pm$ 2°C in water, after two disinfection cycles, after 7 days in disinfection solutions, after 7 days in water only). For each experimental condition, eight specimens were made from each material. Data were analyzed by analysis of variance followed by Tukey's test, and Student's *t*-test ($p$= 0.05).**

***Results:*** **For Kooliner (from 6.2 $\pm$ 0.3 to 6.5 $\pm$ 0.5 VHN) and Lucitone 550 (from 16.5 $\pm$ 0.4 to 18.4 $\pm$ 1.7 VHN), no significant changes in hardness were observed either after the disinfection or after 7 days of immersion, regardless of the disinfectant solution used. For Duraliner II (from 4.0 $\pm$ 0.1 to 4.2 $\pm$ 0.1 VHN), with and without heat treatment, a small but significant increase in hardness was observed for the specimens immersed in the disinfectant solutions for 7 days (from 4.3 $\pm$ 0.2 to 4.8 $\pm$ 0.5 VHN). All materials showed no significant change in roughness (Kooliner: from 0.13 $\pm$ 0.05 to 0.48 $\pm$ 0.24 $\mu$m; Duraliner II, with and without heat treatment: from 0.15 $\pm$ 0.04 to 0.29 $\pm$ 0.07 $\mu$m; Lucitone 550: from 0.44 $\pm$ 0.19 to 0.49 $\pm$ 0.15 $\mu$m) after disinfection and after storage in water for 7 days.**

***Conclusions:*** **The disinfectant solutions, 1% sodium hypochlorite and 4% chlorhexidine gluconate, caused no apparent damage on hardness and roughness of the materials evaluated.**

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**INDEX WORDS: denture base acrylic resins, hard chairside reline resins, hardness, roughness, disinfection, removable prostheses**

AS DENTURE wearers become older and lose much of their supporting alveolar bone, the need for continuing prosthodontic care such as relines becomes more evident. The methods used for relining dentures can be performed directly in the mouth, by autopolymerizing reline resins, or indirectly, using heat-activated materials.[1] A direct relining system is not only simple and practical to perform but also time- and cost-effective. Advantages of the hard chairside reline resins

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include ease of manipulation and low exothermic heat of reaction.[1,2]

After relining, adjustments and polishing procedures are often required. It has been demonstrated that the polishing portion of a normal adjustment procedure for a previously inserted denture was found to produce massive contamination of the pumice slurry.[3,4] Although the risk of infection depends upon the minimum infective dose of any given organism, counts of the magnitude reported by Witt and Hart[5] pose a significant risk. Furthermore, the presence of bacteria in high numbers in laboratory dental pumice as observed by Williams et al[6] is not in keeping with acceptable practices of hygienic control, sterilization, and disinfection in the dental operatory and laboratory. These studies demonstrate that the usual operating procedures of the prosthodontic laboratory are a possible source of cross-contamination between patients, technicians, and dental personnel. Therefore, precautionary measures, such as discarding the pumice after each use, using a liquid disinfectant instead of water as a mixing medium for pumice, autoclaving the pumice and rag wheels, and using separate instruments and pumice for new dentures and for dentures that have already been inserted in the mouth,[3,4,7,8] have been suggested to reduce cross-contamination. However, some of these measures, such as mixing the pumice with a chemical disinfectant, have been shown to be ineffective in prevention of cross-contamination.[7] Furthermore, considering that the other methods are inconvenient and time consuming, they are not commonly used in routine practice.

Another method to prevent cross-contamination is denture disinfection, which can be performed using ozone,[9] microwave irradiation,[10,11] or immersion in chemical solutions.[8,12,13] When the immersion procedure is used, the choice of the disinfectant should be made with regard to its effectiveness in inactivating microorganisms without any adverse effects on the denture materials. Therefore, the effects of the disinfectant solutions on the denture base materials have been investigated. Shen et al[14] observed that all the resins evaluated, when exposed to glutaraldehyde alkaline disinfectant solution with a phenolic buffer for 2 hours or more, had a soft surface. As the time of exposure increased, the matrix phase seemed to dissolve slowly and more polymer beads were exposed. Asad et al[15] found that the modulus of rupture for a homopolymer (non-cross-linked polymethylmethacrylate) was significantly affected when specimens were stored in an alcohol-based disinfectant. In a further study,[16] a highly significant change in the hardness was found when the specimens were immersed in glutaraldehyde and chlorhexidine for 7 days. Ma et al[17] observed that a phenolic-based disinfectant caused surface damage to five resins after 30 minutes of immersion. The roughness of the acrylic resin surfaces is of considerable importance, since the adhesion of microorganisms to a surface is a prerequisite for the colonization of that surface. According to Verran and Maryan,[18] the denture may function as a reservoir of infection, and surface irregularities would increase the likelihood of microorganisms remaining on the surface after the prosthesis has been cleaned.[19]

In a preliminary study, an infection-control protocol intended to prevent cross-contamination was evaluated.[20] The results demonstrated that 1% sodium hypochlorite and 4% chlorhexidine gluconate solutions proved to be effective in reducing the microbial growth on the dentures in 10 minutes. Thus, the aim of this study was to investigate the effect of 1% sodium hypochlorite and 4% chlorhexidine gluconate disinfectant solutions on the hardness and roughness of two hard chairside reline resins and one heat-polymerizing denture base acrylic resin. The hypothesis to be tested was that both disinfectant solutions could cause adverse effect on the hardness and roughness of the denture base and reline materials.

## Materials and Methods

Two autopolymerizing acrylic resins and one heat-polymerizing denture base resin were evaluated. The names of the resins, manufacturers, proportions of powder to liquid, composition, lot numbers, and the polymerization conditions recommended by the manufacturers are presented in Table 1.

According to the manufacturer, Duraliner II liquid contains butyl methacrylate and the cross-linking agent ethylene glycol dimethacrylate. Kooliner liquid contains isobutyl methacrylate without a cross-linking agent.[21] These materials were selected to evaluate the influence of disinfectant solutions on the hardness of reline resins having different compositions. The Lucitone 550 was selected as representative of the conventional poly (methyl methacrylate) heat-polymerizing acrylic resins, which are commonly used for the fabrication of denture bases.

**Table 1.** Materials Used

| *Product* | *Manufacturer* | *Powder/ Liquid Ratio* | *Composition* | | *Lot No.* | *Curing Cycle* |
|---|---|---|---|---|---|---|
| | | | *Powder (P)* | *Liquid (L)* | | |
| Duraliner II | Reliance Dental Mfg. Co., Worth, IL | 10 ml/7 ml | PEMA† | BMA† | P– 030993 L- 020394 | 12 min at 37 ± 2°C |
| Kooliner | Coe Laboratories, Inc., Chicago, IL | 10 ml/4 ml | PEMA‡ | IBMA‡ | P– 091093[A] L– 100493[A] | 10 min at 37 ± 2°C |
| Lucitone 550 | Dentsply International Inc., York, PA | 21 gm/10 ml | PMMA§ | MMA/ EDGMA§ | P– 200792 L– 021294 | 90 min at 73°C and then 100°C boiling water for 30 min |

PEMA = poly(ethyl methacrylate); PMMA = poly (methyl methacrylate); IBMA = isobutyl methacrylate; BMA = butyl methacrylate; MMA = methyl methacrylate; EDGMA = ethylene glycol dimethacrylate.
† Personal communication with Mr. Bob Faxel, Reliance Dental, September 1997.
‡ From Arima et al[21]
§ Dentsply, Acrylic Resin Material Safety Data Sheet, September 2000.

Test specimens were produced in molds prepared by the investment of stainless steel dies (12 × 12 × 8 mm) in silicone rubber, further supported by dental stone within the flask. The square shape of the specimens was chosen to ensure that the roughness measurements were made at the same locations on the specimen surface. The liquid/powder ratio of the polymer dough for all materials was mixed according to the manufacturers' instructions (Table 1), inserted into the molds, and packed. For the hard chairside reline resins, the flasks were pre-heated at 37 ± 2°C in an incubator (Olidef CZ, Ribeirão Preto, São Paulo, Brazil) and maintained at this temperature during the polymerization time recommended by the manufacturers (Table 1). Lucitone 550 specimens were polymerized under pressure in a thermostatically controlled water-bath (Termotron P100, São Paulo, Brazil), using the short polymerization cycle (Table 1).

After polymerization, the specimens were observed by visual inspection, and presented a smooth surface and did not show voids or porosity. Sixteen specimens were made for each material. For Duraliner II material, an additional 16 specimens were obtained and subjected to heat treatment (10 minutes in water at 55°C) as suggested by the manufacturer to reduce the residual monomer taste. Immediately after polymerization, any flash and excess were removed by polishing using progressively finer grades of silicon carbide paper (600-1200), to obtain a smooth, flat surface.

Microhardness measurements were obtained for all specimens with a Vickers Hardness Tester (Otto Wolpert, Germany). Although less sensitive than other methods (e.g. Wallace or Knoop hardness tests), the Vickers hardness test is a valid tool for evaluating the hardness, viscoelastic properties, and other responses of rigid polymers,[22] and some studies have used the Vickers hardness test to detect changes in hardness of denture base acrylic resin[23-26] and acrylic resin denture teeth.[27] The test involves the use of a diamond indenter point in the shape of a square-based pyramid. For Lucitone 550 and Kooliner specimens, a 30-g load and a 30-second time were used. When Duraliner II was tested, the load was 10 g, so the indentation could be properly measured. The diagonals of the pyramid impressed on the specimen by the Vickers indenter were measured and noted. The operator of the test machine read the lengths of the diagonals immediately after each indentation, with a minimal (as short as 10 seconds) period of time elapsed between making and reading the indentations. It was assumed that due to the short time elapsed between making and reading the indentation, the viscoelastic recovery of the diagonals after indentation was minimal.[22] The smooth flat surface of the specimens facilitated the visualization of the diagonals during the measurements. Eight indentations were made at different points on each specimen and the mean value was calculated for that specimen. Vickers hardness number (VHN) was then calculated for each specimen and the means of individual specimens were averaged, thus providing a mean value representative of the materials in a particular experimental condition.

The surface roughness (Ra, $\mu$m) was analyzed with a surface roughness profilometer (Prazis, RUG-03, Casilla de Correo, Rep. Argentina) with a diamond stylus (tip radius 2 $\mu$m). Ra is the arithmetic average of the absolute values of the measured profile height of surface irregularities, measured from a mean line within a preset length of the specimen.[17] A reading was obtained by the needle passing across 0.8 mm length at 0.5 mm/s to the nearest 0.01 $\mu$m. This procedure was repeated two more times at the same position for a total of three readings. So, three readings, each consisting of three lengths of 0.8 mm were taken at each position, resulting in 2.4 mm of reading. As surface roughness was measured at four positions, radially across each specimen, a final Ra average was calculated and the means of individual specimens were averaged. An orientation jig was fabricated to position the stylus of the profilometer instrument to the same location on the specimen for repeated measurements.

The hardness and roughness readings were performed within the first hour in dry conditions at room temperature (23 ± 2°C). These values were used as controls, as the patients will be wearing the relined denture bases soon after polymerization, without any effect of water sorption and disinfection. The specimens were then stored in distilled water at 37 ± 2°C for 48 hours, and hardness and roughness were measured. This time was chosen as being representative of the first recall appointment for adjustments after relining. Thereafter, specimens were disinfected twice, simulating when dentures come from the patient and before being returned to the patient. Each specimen was scrubbed with 4% chlorhexidine gluconate for 1 minute,[20,28] rinsed in water, and then immersed for 10 minutes in one of the two disinfectant solutions (n = 8): 4% chlorhexidine gluconate (FGM; Produtos Odontológicos, Joinvile, SC, Brazil) and 1% sodium hypochlorite (Miyako do Brazil Ind. e Com. LTDA., São Paulo, Brazil). The solutions were obtained by dissolving the disinfectants chlorhexidine gluconate and sodium hypochlorite in distilled water at room temperature (23 ± 2°C). The treatment times and temperature used in the present investigation were based on a previous clinical study in which the protocol proved to be effective in disinfecting removable dental prostheses.[20] After disinfection, hardness and roughness were again evaluated. In order to determine whether an extended exposure time to the disinfectant would adversely affect the acrylic resins, measurements were also taken after the specimens were immersed in the disinfectant solutions for 7 days. Eight specimens of each material were also manufactured to evaluate the effect of water immersion only on both hardness and roughness. These specimens were not submitted to the disinfection procedures, but kept in water at 37 ± 2°C for 7 days. Measurements of hardness and roughness were made on these specimens after polymerization and after 7 days of immersion in water.

For specimens subjected to the disinfectant solutions, the results obtained were analyzed by repeated measures analysis of variance (ANOVA), followed by Tukey's test. Analyses of data obtained for the specimens immersed in water only were conducted using the Student's *t*-test. Statistical analyses were conducted at 95% level of confidence.

## Results

It can be seen from Table 2 that the hardness of all materials remained unaffected after being submitted to two disinfection cycles. After 7 days of immersion in both disinfectant solutions, only the Duraliner II specimens, tested with or without heat treatment, exhibited a small but significant ($p < 0.05$) increase in hardness mean values. In general, 4% chlorhexidine gluconate and 1% sodium hypochlorite disinfectant solutions caused no significant effect on the roughness of all materials evaluated (Table 3).

The effect of immersion in water on the hardness and roughness of all the materials is shown in Tables 2 and 3, respectively. A small but significant ($p < 0.05$) increase in hardness was observed for materials Kooliner and Duraliner II (non-heat-treated specimens) after immersing in water at 37 ± 2°C for 7 days. Duraliner II heat-treated specimens and Lucitone 550 specimens showed no significant change. For all materials, the statistical

**Table 2.** Effect of Disinfection and Water Immersion on Hardness of Materials (VHN-kg/mm$^2$)

| | | *Conditions:* | | | | |
|---|---|---|---|---|---|---|
| | *Time=* | *1 Hour After Polymerization* | *48 Hours* | *2 Cycles of Disinfection* | *7 Days* | *7 Days* |
| *Material* | *Temperature=* | *23 ± 2°C* | *37 ± 2°C* | *23 ± 2°C* | *23 ± 2°C* | *37 ± 2°C* |
| | *Solution=* | *None* | *Water* | *Disinfectant* | *Disinfectant* | *Water* |
| Kooliner | 1% Sodium hypochlorite | 6.2 (0.3) a | 7.1 (0.6) bc | 6.5 (0.3) a | 6.7 (0.3) ac | — |
| | 4% Chlorhexidine gluconate | 6.5 (0.5) a | 6.4 (0.4) a | 6.4 (0.3) a | 6.9 (0.3) a | — |
| | Water alone | 5.8 (0.1) a | — | — | — | 5.9 (0.1) b |
| Duraliner II | 1% Sodium hypochlorite | 4.0 (0.1) a | 4.1 (0.1) ab | 4.1 (0.1) ab | 4.5 (0.3) b | — |
| | 4% Chlorhexidine gluconate | 4.0 (0.1) a | 4.1 (0.1) a | 4.3 (0.3) b | 4.3 (0.2) b | — |
| | Water alone | 4.0 (0.3) a | — | — | — | 4.5 (0.6) b |
| Duraliner II (heat-treated) | 1% Sodium hypochlorite | 4.2 (0.1) a | 4.4 (0.2) a | 4.4 (0.3) a | 4.7 (0.3) b | — |
| | 4% Chlorhexidine gluconate | 4.0 (0.2) a | 4.2 (0.2) a | 4.0 (0.1) a | 4.8 (0.5) b | — |
| | Water alone | 3.9 (0.1) a | — | — | — | 4.0 (0.2) a |
| Lucitone 550 | 1% Sodium hypochlorite | 18.4 (1.7) a | 17.2 (1.5) a | 17.9 (2.1) a | 17.6 (1.0) a | — |
| | 4% Chlorhexidine gluconate | 16.5 (0.4) a | 16.9 (0.8) a | 16.8 (1.8) a | 17.7 (1.2) a | — |
| | Water alone | 15.7 (0.7) a | — | — | — | 15.2 (0.7) a |

Values are means; standard deviations are given in parentheses. Horizontally, means with the same letter were not significantly different from each other at the $p = 0.05$ level. No comparisons were made among materials.

**Table 3.** Effect of Disinfection and Water Immersion on Roughness of Materials (Ra-$\mu$m)

| Material | Conditions: Time= / Temperature= / Solution= | 1 Hour After Polymerization / 23 ± 2°C / None | 48 Hours / 37 ± 2°C / Water | 2 Cycles of Disinfection / 23 ± 2°C / Disinfectant | 7 Days / 23 ± 2°C / Disinfectant | 7 Days / 37 ± 2°C / Water |
|---|---|---|---|---|---|---|
| Kooliner | 1% Sodium hypochlorite | 0.13 (0.05) a | 0.25 (0.03) b | 0.26 (0.03) b | 0.26 (0.03) b | — |
| | 4% Chlorhexidine gluconate | 0.48 (0.24) a | 0.55 (0.20) a | 0.60 (0.18) a | 0.56 (0.36) a | — |
| | Water alone | 0.27 (0.12) a | — | — | — | 0.23 (0.08) a |
| Duraliner II | 1% Sodium hypochlorite | 0.21 (0.06) a | 0.27 (0.05) a | 0.26 (0.04) a | 0.25 (0.03) a | — |
| | 4% Chlorhexidine gluconate | 0.15 (0.04) a | 0.21 (0.03) b | 0.17 (0.03) ab | 0.19 (0.03) b | — |
| | Water alone | 0.14 (0.06) a | — | — | — | 0.16 (0.06) a |
| Duraliner II (heat-treated) | 1% Sodium hypochlorite | 0.22 (0.04) a | 0.23 (0.04) a | 0.23 (0.03) a | 0.25 (0.04) a | — |
| | 4% Chlorhexidine gluconate | 0.29 (0.07) a | 0.33 (0.14) ab | 0.30 (0.08) a | 0.37 (0.16) b | — |
| | Water alone | 0.25 (0.07) a | — | — | — | 0.24 (0.07) a |
| Lucitone 550 | 1% Sodium hypochlorite | 0.44 (0.19) a | 0.40 (0.19) a | 0.43 (0.21) a | 0.42 (0.19) a | — |
| | 4% Chlorhexidine gluconate | 0.49 (0.15) a | 0.47 (0.25) a | 0.57 (0.25) a | 0.70 (0.51) a | — |
| | Water alone | 0.44 (0.15) a | — | — | — | 0.47 (0.11) a |

Values are means; standard deviations are given in parentheses. Horizontally, means with the same letter were not significantly different from each other at the $p = 0.05$ level. No comparisons were made among materials.

analyses of the roughness data revealed that there were no significant changes after immersion in water.

## Discussion

In this study, two hard chairside reline resins and one heat-polymerized acrylic resin were investigated with respect to the effect of disinfectant solutions on hardness property. The Vickers hardness tests were not capable of detecting any softening effect of the solutions evaluated (1% sodium hypochlorite and 4% chlorhexidine gluconate) on the materials, even after the specimens had been immersed in the solutions for 7 days. In fact, Duraliner II specimens showed a small but significant increase in hardness after this period of immersion. Similarly, it was observed that the hardness of the Kooliner and Duraliner II specimens (non-heat-treated) showed a small but significant increase in hardness after they were immersed in water for 7 days. During the polymerization reaction, varying amounts of methyl methacrylate monomer still remain in the acrylic resin[29-31] and may act as a plasticizer, thus decreasing the mechanical properties of the polymerized resins.[32-34] This residual monomer might continue to be consumed by a further polymerization at the site of polymer-free radicals in the matrix.[35,36] In addition, residual monomer can be slowly leached into the storage solutions, increasing the hardness of acrylic resins.[34] At the same time, however, acrylic resins absorb water molecules,[32,37,38] which also act as plasticizers, thus compromising the mechanical strength of the denture base and reline resins.[39,40] Von Fraunhofer and Suchatlampong[41] studied the indentation resistance of denture base polymers and observed that storage in water could result in softening of the surface in heat-polymerized acrylic resins. Although not examined in this study, it may be possible that the increase in hardness observed for the autopolymerizing reline resins Kooliner and Duraliner II was related to further polymerization and residual monomer release mechanisms, which probably overcame the plasticizing effect of the water uptake. It should be emphasized that, although statistically significant, the increase in hardness observed in the present study was extremely small (less than 1 VHN unit) and not of any practical importance. Similar results were found by Haywood et al,[42] who observed that the Wallace hardness of three hard reline materials was slightly increased after immersion in hypochlorite cleanser for up to 6 months. They considered that the changes were likely to be of no clinical significance.

The hardness values observed for Lucitone 550 are in agreement with those reported for poly (methyl methacrylate) denture teeth (17.0 VHN)[27] and heat-polymerizing denture base acrylic resins (18.6 VHN).[33] Similar to Buudai et al's investigation,[43] the hard chairside reline resins exhibited lower hardness values than the heat-polymerized denture base resin Lucitone 550.

The results of this study also indicated that the roughness of the materials was not affected by immersion in any of the disinfectant solutions and water for 7 days. Shen et al[14] studied the effect of glutaraldehyde base disinfectants (alkaline, phenol buffered) on surface morphology of denture base resins and reported that no apparent surface change was observed with the regular alkaline formulation. However, the disinfectant with phenolic buffer caused surface pitting of the material after 10 minutes of immersion, and softening and swelling of the surface after 2 hours of immersion. Similar results were obtained by Ma et al,[17] who observed that the phenolic-based disinfectant was the only solution that caused surface damage to five resins after 30 minutes of immersion. Therefore, the favorable results obtained in this study could be attributable to the fact that the immersion solutions used do not contain chemicals, such as phenol, that may cause surface damage.

The mean surface roughness values of all materials were lower than those reported by Zissis et al,[44] who found mean values ranging from 0.7 $\mu$m to 4.4 $\mu$m for hard reline resins and from 3.4 $\mu$m to 7.6 $\mu$m for denture base acrylic resins. In addition, the results of this study revealed that the reline materials generally showed mean surface roughness values near the threshold surface roughness for bacterial retention (Ra $= 0.2\ \mu$m) below which no further reduction in bacterial accumulation could be expected.[45] This finding suggests that the reline resins exhibited favorable surface characteristics, with mean roughness values of such magnitude that plaque accumulation could be minimized, thus facilitating the denture hygiene. Conversely, the surface roughness of the denture base material Lucitone 550 was higher (from 0.40 $\mu$m to 0.70 $\mu$m) than the cited threshold. Nevertheless, according to the results from Quirynen et al,[46] an increase in bacterial colonization would be expected to occur on surface with Ra roughness values of 2.2 $\mu$m.

In previous investigations, the disinfectant solutions used in this study proved to be effective in reducing the microbial growth,[20] with no adverse effect on the flexural strength of denture base acrylic resins[13] and the hardness of acrylic resin denture teeth.[27] In the present study, no change in roughness of the materials was observed, and a small but statistically significant increase in hardness was detected. It is important to mention that these properties are only a very limited view of the materials, and that as far as roughness and hardness were concerned, clinically important differences could not be detected.

## Conclusions

Within the limitations of the current experiments, the following were concluded:

There were no apparent important differences in hardness and roughness that arose as a result of disinfection.

1. For materials Kooliner and Lucitone 550, no significant changes in hardness were found either after the disinfection or after 7 days of immersion in both disinfectant solutions evaluated ($p > 0.05$).
2. Duraliner II specimens showed a small but significant increase ($p < 0.05$) in hardness after immersion in the disinfectant solutions for 7 days.
3. After immersion in water for 7 days, a small but significant increase ($p < 0.05$) in hardness was observed for Kooliner and Duraliner II specimens (non-heat-treated).
4. For all materials, immersion in the disinfectant solutions and water for 7 days produced no significant effect in roughness ($p > 0.05$).

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