The effect of chemical surface treatments of different denture base resins on the shear bond strength of denture repair

Y. Sinasi Sarac, DDS, PhD,a Duygu Sarac, DDS, PhD,b Tolga Kulunk, DDS,c and Safak Kulunk, DDSd
Faculty of Dentistry, Department of Prosthetic Dentistry, Ondokuz Mayis University, Samsun, Turkey

Statement of problem. Fracture of a repaired denture base often occurs at the junction of the base and repair materials rather than within these materials.

Purpose. The purpose of the study was to evaluate the shear bond strengths of 4 denture base acrylic resins following the use of 3 chemical solvents and to examine treated acrylic resin surfaces under a field emission scanning electron microscope (SEM).

Material and methods. Forty discs (15 mm in diameter and 3 mm thick) were fabricated for each denture base material (a conventionally molded, heat-polymerized [Meliodent, M], an injection-molded, heat-polymerized [SR-Ivocap, I], and a microwave-polymerized [Acron MC, A]) repaired with an autopolymerizing acrylic resin (Meliodent), for a total of 120 specimens, processed according to manufacturers’ instructions, embedded in acrylic resin blocks, and divided into 4 groups of 10. One of the groups served as control and had no surface treatment. In the 3 experimental groups, specimen surfaces were treated with chemical etchants by immersion in acetone (ac) for 30 seconds, in methylene chloride (mc) for 30 seconds, or in MMA (mo) for 180 seconds, respectively. Then autopolymerizing acrylic resin (Meliodent) was placed on the treated surfaces using a brass ring (6 mm in diameter and 2 mm in height) to confine the material to a standardized dimension. After 24 hours of storage at 37°C, the shear bond strength (MPa) of the specimens was measured in a universal testing machine. A 2-way analysis of variance and the Tukey HSD test were performed to identify significant differences (α=.05). The nature of the failure was noted as adhesive, cohesive, or mixed. The effect of the chemical treatments on the surface of base resins was examined under an SEM.

Results. Chemical treatments increased the bond strength of repair material significantly. Significant differences were found between the control and experimental groups (P<.001). In the control group, M showed the highest (16.7 MPa) bond strength, and A showed the lowest (9.4 MPa). No significant differences were detected between M (18.9 MPa) and A (19.9 MPa) with acetone treatment, or between M (19.3 MPa) and A (20.3 MPa) with methylene chloride treatment. The SEM observations showed that application of chemical etchants produced smoother surfaces than controls.

Conclusion. Chemical treatment prior to denture base repair showed significant improvement on the bond strength of the base materials. Although the microwave-polymerized acrylic resin, A, showed the lowest shear bond strength compared to the control groups, the highest percentage increase was obtained with A after chemical treatments. (J Prosthet Dent 2005;94:259-66.)

CLINICAL IMPLICATIONS

In this in vitro study, chemical treatments increased the bond strength of autopolymerized acrylic resin to base materials tested. The highest shear bond strength values were obtained with methylene chloride and acetone application, and the bond strengths obtained with methylene chloride were not significantly different from those obtained with acetone.

The fracture of acrylic resin denture base material is a common clinical occurrence. Denture fracture is generally related to faulty design, fabrication, and material choice.1 Fractures are more common in the midline of maxillary complete dentures.2,3 The fabrication of a new denture is an expensive and time-consuming procedure. Furthermore, being without a prosthesis may be an inconvenience for the patient. For this reason, the decision to repair a denture, whether as an interim or definitive measure, is a common one.4

Several materials have been used to repair fractured acrylic resin dentures, including autopolymerized,5-7 heat-polymerized,8 visible light-polymerized,5,7,9,10 and microwave-polymerized acrylic resins.11,12 Although various methods have been proposed for repairing fractured denture bases, the use of autopolymerizing acrylic resin, allowing for simple and quick repair, is the most popular.2,8 However, dentures repaired with autopolymerizing acrylic resin often refracture at

aAssistant Professor.
bAssistant Professor.
cResearch Assistant.
dResearch Assistant.
the repair site.\textsuperscript{13,14} The fracture of repaired dentures often occurs at the interface junction of the original base and repair materials, rather than within these materials.\textsuperscript{8} Mechanical and chemical surface modifications of denture base resin materials have been described to improve bond strength.\textsuperscript{15-18} Mechanical modifications to improve bond strength include grinding with burs,\textsuperscript{15} airborne-particle abrasion,\textsuperscript{16,17} and laser treatment\textsuperscript{18} to increase surface area and mechanical retention.

Successful denture repair relies on the phenomenon of adhesion. Strong bonding of the surfaces improves the strength of the repaired unit and reduces stress concentration.\textsuperscript{1,2} Adhesion between denture base and repair materials can be improved by first applying appropriate chemicals to the acrylic resin surfaces. These chemicals etch the surface by changing morphology and chemical properties of the materials.\textsuperscript{19} Normally this change is obtained by wetting the surfaces with methyl methacrylate (MMA).\textsuperscript{6} Organic solvents such as chloroform,\textsuperscript{8} acetone,\textsuperscript{12,19} and methylene chloride\textsuperscript{15,20,21} have also been used for this process. Some investigators have reported that these organic solvents increase the bond strength of a repair material to the denture base.\textsuperscript{8,12,13,19,20} Vallittu et al\textsuperscript{6} stated that wetting the repair surface with MMA dissolved the surface structure of polymethyl methacrylate (PMMA), and a duration of 180 seconds of wetting with MMA enhanced adhesion, compared with shorter durations of wetting. Although an effective application time of MMA for increasing the repair strength was reported by Vallittu,\textsuperscript{6} the authors did not identify guidelines in the literature concerning the most effective application time of methylene chloride and acetone. However, the application time for other chemical treatments was generally stated as 30 seconds.\textsuperscript{12,19}

Polymethyl methacrylate resins have been preferred as denture base resins because of their physical and esthetic properties as well as the material’s availability, reasonable cost, and ease of manipulation.\textsuperscript{14} For several years, PMMA resins were molded using simple compression techniques.\textsuperscript{22} Acrylic resins have been modified to improve not only their physical and chemical properties, but also their working properties to aid the processing of removable partial and complete dentures. One example is the introduction of injection molding, which allows directional control of the polymerization process through the sprue design. A constant flow of new material from the sprue compensates for the polymerization shrinkage.\textsuperscript{23}

Another technique is microwave polymerization of acrylic resins. These acrylic resins polymerize using microwave energy, which activates the decomposition of benzoyl peroxide, the initiator, and yields free radicals for the polymerization process.\textsuperscript{14} Microwave-polymerized acrylic resin has lower residual monomer levels relative to conventionally polymerized resins, and it has been suggested that residual monomer levels have an effect on the strength of repaired specimens.\textsuperscript{11}

The research hypothesis for this project was that the chemical and physical properties of denture base resins affect the bond strength of repair material as well as surface treatments. The purpose of this in vitro study was to evaluate the shear bond strength of a conventionally molded, heat-polymerized, an injection-molded, heat-polymerized, and a microwave-polymerized acrylic resin, repaired with an autopolymerizing acrylic resin, following the use of 3 chemical solvents, MMA monomer, acetone, and methylene chloride. The treated acrylic resin surfaces were examined under a field emission scanning electron microscope (SEM).

**MATERIAL AND METHODS**

The products used in this study are listed in Table I. Forty discs were fabricated for each denture base material (15 mm diameter and 3 mm thick), for a total of 120 specimens, processed according to manufacturers’ instructions. The conventionally molded, heat-polymerized denture resin (M) was placed under compression in 74°C water for 8 hours, and the microwave-polymerized resin (A) was processed using a 500-W power output for 3 minutes in a microwave oven (BK MD 1500; Beko, Istanbul, Turkey). Once processed, all the flasks were bench-cooled for 30 minutes. The injection molded, heat-polymerized acrylic resin (I) was flaked using the Ivocap system. Premixed capsules of resin and monomer were triturated for 5 minutes before injecting into the flask. Hydraulic pressure was maintained for 5 minutes before placing the assembly into boiling water (100°C) for 35 minutes. The assembly was then removed and placed in cold water for 20 minutes before deflasking the denture base acrylic resins.

All acrylic resin specimens were ground flat with 600-grit silicon carbide paper (CarbiMet; Buchler, Lake Bluff, Ill) to remove surface irregularities and excess material. The specimens were mounted with autopolymerizing acrylic resin (Meliodent; Heraeus Kulzer, Armonk, NY), with the flat surfaces facing up in the plastic holders (30 mm in diameter and 20 mm thick). The specimens were stored in water at 37°C for 7 days before the surface treatments.

After water storage, all specimens were ultrasonically cleaned with distilled water and dried with compressed air. The acrylic resin specimens were divided into 4 subgroups, each with 10 specimens. For each denture base resin, 1 subgroup served as a control and had no surface treatment. In the 3 experimental subgroups, the exposed specimen surfaces were treated with chemical etchants, either by immersion in acetone (ac) for 30 seconds,\textsuperscript{12} in methylene chloride (mc) for 30 seconds, or in MMA (mo) for 180 seconds,\textsuperscript{6} respectively. A brass ring
(6-mm inner diameter, 2-mm height) was used to add the autopolymerizing acrylic resin to the treated specimen surfaces. The autopolymerizing acrylic resin was mixed according to the manufacturer’s recommendation and placed into the brass ring (Fig. 1), and the excess material was removed from the top of the brass ring. The acrylic resin was polymerized under 2-bar pressure at 37°C for 10 minutes using a polymerization pressure vessel (Polyclav; Dentaurum, Ispringen, Germany), followed by storage in distilled water for 24 hours before testing.

The shear test was performed on the specimens in a universal testing machine (Lloyd LRX; Lloyd Instruments, Fareham, UK) at a 1 mm/min crosshead speed (Fig. 2). Data were analyzed by 2-way analysis of variance (ANOVA) (SPSS 10.0; SPSS Inc, Chicago, Ill), and mean values were compared by the Tukey HSD test ($\alpha=.05$). In addition, the type of failure was observed by use of a stereomicroscope (Stemi 2000-C; Carl Zeiss, Gottingen, Germany) at a magnification of ×10 to determine the amount of repair material left behind and to assess any damage to the denture base resin that might have occurred during the shear bond test. The nature of the failure was noted as adhesive, cohesive, or mixed. Cohesive failure was deemed to have occurred if more than 50% of the testing surface of the base material fractured.

To evaluate the effect of the chemical treatments on the surface of each denture base material, 4 specimens, $30 \times 30 \times 2$ mm, were prepared, and their surfaces were ground flat with 600-grit silicon carbide paper (CarbiMet; Buehler). These surfaces were treated with the same experimental protocol described previously.

### Table 1. Materials used in this study

<table>
<thead>
<tr>
<th>Material</th>
<th>Product</th>
<th>Code</th>
<th>Manufacturer</th>
</tr>
</thead>
<tbody>
<tr>
<td>Heat-polymerized (Conventional-molding) acrylic resin</td>
<td>Meliodent</td>
<td>M</td>
<td>Heraeus Kulzer, Armonk, NY</td>
</tr>
<tr>
<td>Heat-polymerized (Injection-molding) acrylic resin</td>
<td>SR-Ivocap</td>
<td>I</td>
<td>Ivoclar Vivadent, Schaan, Liechtenstein</td>
</tr>
<tr>
<td>Microwave-polymerized acrylic resin</td>
<td>Acron MC</td>
<td>A</td>
<td>GC Corp, Tokyo, Japan</td>
</tr>
<tr>
<td>Autopolymerized acrylic resin</td>
<td>Meliodent</td>
<td>–</td>
<td>Heraeus Kulzer</td>
</tr>
<tr>
<td>Acetone</td>
<td>–</td>
<td>ac</td>
<td>E. Merck, Darmstadt, Germany</td>
</tr>
<tr>
<td>Methylene chloride</td>
<td>–</td>
<td>mc</td>
<td>E. Merck</td>
</tr>
<tr>
<td>Methyl methacrylate</td>
<td>Meliodent</td>
<td>mo</td>
<td>Heraeus Kulzer</td>
</tr>
</tbody>
</table>

![Fig. 1. A, Brass ring placed on autopolymerizing acrylic resin block. B, Parts of brass ring.](image)

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![Fig. 2. Testing apparatus.](image)
All specimens were gold sputtered with a sputter coater (S150B; Edwards, Crawley, United Kingdom) and examined under a field emission SEM (JSM-6335F; Jeol, Tokyo, Japan) at 15.0 or 20.0 kV. The SEM photomicrographs were taken at 3200× magnification with a bar 10 μm.

Figure 3. SEM of conventionally molded, heat-polymerized denture base resin surfaces after treatment. A, Untreated. B, Treated with acetone. C, Treated with methylene chloride. D, Treated with MMA. Original magnification ×2000. Bar, 10 μm.

Table II. Mean shear bond strength values (SD) in MPa for repaired specimens

<table>
<thead>
<tr>
<th>Base material</th>
<th>Surface treatment</th>
<th>Percentage of increased strength</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Control</td>
<td>ac (0.5)</td>
</tr>
<tr>
<td>M</td>
<td>16.7 (0.5)</td>
<td>19.9 (0.9) a,c</td>
</tr>
<tr>
<td>I</td>
<td>12.7 (0.8)</td>
<td>19.9 (0.5) a,c</td>
</tr>
<tr>
<td>A</td>
<td>9.4 (0.9)</td>
<td>16.5 (0.5) a,c</td>
</tr>
</tbody>
</table>

Same letters indicate values that were not statistically different (P>.05).
% = Percentage of increased strength compared to control.

Table III. Results of ANOVA

<table>
<thead>
<tr>
<th>Source of variation</th>
<th>Sum of squares</th>
<th>df</th>
<th>Mean square</th>
<th>F</th>
<th>Significance</th>
</tr>
</thead>
<tbody>
<tr>
<td>Base material</td>
<td>223.496</td>
<td>2</td>
<td>111.748</td>
<td>211.662</td>
<td>.001</td>
</tr>
<tr>
<td>Surface treatment</td>
<td>700.551</td>
<td>3</td>
<td>233.517</td>
<td>442.306</td>
<td>.001</td>
</tr>
<tr>
<td>Base material × Surface treatment</td>
<td>272.377</td>
<td>6</td>
<td>45.396</td>
<td>85.985</td>
<td>.001</td>
</tr>
<tr>
<td>Error</td>
<td>57.019</td>
<td>108</td>
<td>.528</td>
<td></td>
<td>.001</td>
</tr>
<tr>
<td>Total</td>
<td>36209.390</td>
<td>120</td>
<td></td>
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<td></td>
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</tbody>
</table>

Table IV. Failure types

<table>
<thead>
<tr>
<th>Base material</th>
<th>Type of failure</th>
<th>Chemical etchant</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Untreated</td>
<td>Acetone</td>
</tr>
<tr>
<td>Meliodent</td>
<td>AD (%) 6 (60)</td>
<td>2 (20)</td>
</tr>
<tr>
<td></td>
<td>CO (%) –</td>
<td>–</td>
</tr>
<tr>
<td></td>
<td>MI (%) 4 (40)</td>
<td>8 (80)</td>
</tr>
<tr>
<td>SR-Ivocap</td>
<td>AD (%) 10 (100)</td>
<td>4 (40)</td>
</tr>
<tr>
<td></td>
<td>CO (%) –</td>
<td>–</td>
</tr>
<tr>
<td></td>
<td>MI (%) –</td>
<td>6 (60)</td>
</tr>
<tr>
<td>Acron MC</td>
<td>AD (%) 10 (100)</td>
<td>–</td>
</tr>
<tr>
<td></td>
<td>CO (%) –</td>
<td>5 (50)</td>
</tr>
<tr>
<td></td>
<td>MI (%) –</td>
<td>5 (50)</td>
</tr>
</tbody>
</table>

AD: Adhesive failure at interface; CO, cohesive fracture of base material; MI, mixed fracture at interface and base material.

RESULTS

The mean shear strength values and SDs of the groups are presented in Table II. A 2-way ANOVA was used to test for differences among the groups (Table III). When each chemical treatment was compared to the controls, the shear bond strength values increased significantly (P<.001). Significant differences were found between base materials for the control and experimental groups (P<.001). In the control groups, M showed the highest (16.7 MPa) bond strength, and A showed the lowest (9.4 MPa). No significant differences were detected between M (18.9 MPa) and A (19.9 MPa) specimens with acetone treatment or between M (19.3 MPa) and A (20.3 MPa) specimens with methylene chloride treatment. The experimental groups of I were not significantly different from each other.

The type and frequency of failures for specimens are presented in Table IV. The specimens showed 3 types of failures: adhesive (interface), cohesive (only at the base material), and mixed (interface and base material). Mixed and adhesive fractures were the most common types of failure for heat-polymerized denture base materials. However, microwave-polymerized denture material showed mixed and cohesive failures.

The SEM observations of all base material control groups revealed surfaces with scratches, valleys, and depressions. Application of acetone produced a smoother surface than the controls and also created a porous topography. Methylene chloride produced a smooth surface with hollows. Monomer application created a smoother surface texture than the control groups (Figs. 3 through 5).

DISCUSSION

The results of this study support the hypothesis that the chemical and physical properties of denture base resins, as well as surface treatments, affect the bond strength of repair material. The control groups of all denture base materials showed significantly lower bond strength values than the experimental groups (P<.001). For control groups, M showed the highest (16.7 MPa) and A showed the lowest (9.4 MPa) shear bond strength values. Rached et al detected repair strength values of 80.6% and 89.7% when comparing a microwave-polymerized acrylic resin (Acron MC) and a heat-polymerized acrylic resin (Luitconet 199) as the denture base material repaired with an autopolymerized acrylic resin (Acron MC/R), respectively. The authors further reported that the impact strength of a microwave-polymerized material was superior to that of a conventional heat-polymerized acrylic resin, but the repair strength of the microwaved material was lower than heat-polymerized acrylic resin.

In the control and experimental groups, I showed significantly lower bond strength than M (P<.001). Although the 2 base materials are polymerized with the same method, the bond strength of the repair material to them was different. Phoenix et al reported that injection-molded acrylic resins were more resilient than their compression-molded counterparts. Injection-molded acrylic resin generally requires a greater monomer content to improve flow characteristics and facilitate filling of the mold cavity. This often results in additional unreacted monomer within a polymerized acrylic resin. This is one possible explanation for the low bond strength values for the I control and experimental groups when compared to M.

To achieve optimum strength for repairs, it is essential that a good bond exist between base resin and repair material. Etching the repair surfaces with chemical etchants such as acetone, MMA, chloroform, and methylene chloride were suggested by some investigators as methods to dissolve the base resin and achieve a good bond. Chemical surface treatments cause superficial crack propagation, as well as the formation of numerous pits approximately 2 μm in diameter. This surface morphologic change may enhance the mechanical retention between fractured surface and repaired acrylic resin. According to Shen et al, chemical surface dissolution of acrylic resin could be affected by the degree of cross linking of the polymer chains. Nagai et al evaluated the strength and modulus of elasticity of repaired acrylic resin specimens reinforced with various processes, and reported that surface pretreatment with methylene chloride is effective in repairing heat-polymerized denture base resin. Vallittu et al reported that wetting the repair surface with MMA dissolved the surface structure of PMMA and that a duration of 180 seconds of wetting with MMA improved adhesion, compared with shorter durations of wetting. Also, the authors reported that a duration of 180 seconds of wetting with MMA created smoother surface texture than a shorter duration, when the treated surfaces were examined using SEM. Similar to these investigations, the result of this study showed that chemical etching increased the bond strength of the repair material to base material. When the shear bond values of control groups and test groups were compared, significant differences were found (P<.001). Only the percentage of increased strength was different. The increased bond strength demonstrated with chemical surface treatments may be attributed to superior adhesion as a result of monomer infiltration into the pits and cracks.

Nishigawa et al reported that the resin primer containing methylene chloride was effective on the shear strength of repair material. However, there was no significant difference between the control specimens.
and the specimens with primer. The authors applied a primer containing methylene chloride in a thin layer.\textsuperscript{21} In the present study, the highest bond strength obtained was with methylene chloride for each base material, but the bond strength value was not statistically different than acetone. Methylene chloride presumably reaches deeper into the polymer chains by displacing water and facilitates the penetration of the monomer.\textsuperscript{20} Although the most effective application time of MMA on the increase of the repair strength was reported by Vallittu,\textsuperscript{6} the authors could not identify the most effective application time of methylene chloride and acetone from the literature. Since an application time of the chemical treatment is generally stated as 30 seconds,\textsuperscript{12,19} this time period was used in the present study. The application of acetone and methylene chloride to repair surfaces for different time periods should be investigated in the future.

Chemical treatment primarily affected the microwave-polymerized acrylic resin. When the bond strength values were compared, A had the lowest bond strength between control groups. However, relative to the acetone and methylene chloride applications, A showed the highest significant values ($P<.001$). Also, the cohesive failure at the sites of fracture in the ac and mc subgroups of A indicate that surface treatment with chemical etchant created tight adhesion of the autopolymerized acrylic resin to microwave-polymerized base material.

To compare the repair strength of the base resins, most investigators used transverse strength tests.\textsuperscript{10,12,13,19} However, the fracture of repaired dentures often occurs at the junction of the previous and new material rather than within these materials.\textsuperscript{8} The debonding of a repair material from a base resin may be evaluated by measuring the shear bond strength, as was used in this study. However, this method did not simulate the clinical condition ideally, as repaired dentures are exposed to repetitive mechanical stress during mastication. Also, the specimens tested did not physically simulate an actual denture. Therefore, further investigations are necessary to evaluate the bonding under more closely simulated clinical conditions.

In this study, it was assumed that base material surfaces were affected by chemical etching. To test the hypothesis, the surfaces of the base materials were examined with SEM, and it was shown that the surfaces were dissolved by chemical etchants. When the effects of the etchants were compared, it was seen that methylene chloride provided smoother surfaces than the other etchants and also created holes on the base resin surface. Acetone application created a smoother surface but with more superficial pits than the control. Similar to this study, Rached and Del Bel Cury\textsuperscript{19} found that acetone application demonstrated a smoother and cleaner surface than monomer on PMMA surface texture. In the present study, a duration of 180 seconds of monomer wetting created smoother base resin surfaces than untreated specimens. Also, Vallittu et al\textsuperscript{6} reported that 180 seconds of monomer wetting provided smooth acrylic resin surfaces. The SEM micrographs showed that microwave-polymerized acrylic resin was the most affected denture base acrylic resin and methylene chloride was the most effective etching solution.

**CONCLUSIONS**

Within the limitations of this study, the following conclusions were drawn:

1. The denture base materials tested demonstrated significantly different ($P<.001$) shear bond strength values without chemical treatment.
2. The shear bond strength of the repair material to all denture base resin materials tested was increased with chemical treatments.
3. For all 3 acrylic resins tested, methylene chloride treatment produced statistically the same bond strength values as acetone treatment.

**REFERENCES**


Reprint requests to:
DR Y. SINASI SARAC
ONDOKUZ MAYIS UNIVERSITY
DIS HEKIMLIGI FAKULTESI
PROTETIK DIS TEDAVISI A.D.
55139 KURUPELIT
SAMUSUN, TURKEY
FAX: +90 362 457 60 32
E-MAIL: ssarac@omu.edu.tr

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Systematic review of survival rates for implants placed in the grafted maxillary sinus

Based on a systematic review of the literature from 1986 to 2002, this study sought to determine the survival rate of root-form dental implants placed in the grafted maxillary sinus. Secondary goals were to determine the effects of graft material, implant surface characteristics, and simultaneous versus delayed placement on survival rate. A search of the main electronic databases was performed in addition to a hand search of the most relevant journals. All relevant articles were screened according to specific inclusion criteria. Selected papers were reviewed for data extraction. The search yielded 252 articles applicable to sinus grafts associated with implant treatment. Of these, 39 met the inclusion criteria for qualitative data analysis. Only 3 of the articles were randomized controlled trials. The overall implant survival rate for the 39 included studies was 91.49%. The database included 6,913 implants placed in 2,046 subjects with loaded follow-up time ranging from 12 to 75 months. Implant survival was 87.70% with grafts of 100% autogenous bone, 94.88% when combining autogenous bone with various bone substitutes, and 95.98% with bone grafts consisting of bone substitutes alone. The survival rate for implants having smooth and rough surfaces was 85.64% and 95.98%, respectively. Simultaneous and delayed procedures displayed similar survival rates of 92.17% and 92.93%, respectively. When implants are placed in grafted maxillary sinuses, the performance of rough implants is superior to that of smooth implants. Bone-substitute materials are as effective as autogenous bone when used alone or in combination with autogenous bone. Studies using a split-mouth design with one variable are needed to further validate the findings.—Reprinted with permission of Quintessence Publishing.