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EVALUATION OF RESIN DIE MATERIALS, (U)  
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Evaluation of resin die materials

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Products produced from the mineral gypsum are used extensively for the construction of models and dies. In general, the performance of these materials in the indirect fabrication of cast fixed restoration is adequate.<sup>1,2</sup> However, breakage and abrasion of fragile die margins are not uncommon occurrences.

Certain mechanical features of resin-based die materials have focused increased attention upon these substances as potential substitutes for the commonly used dentite products. Previously reported data, however, are insufficient to either confirm or to refute the efficacy of the use of resin-based models and dies in fixed prosthodontic procedures.

Commercial materials and equipment are identified in this report to adequately specify the experimental procedure. Such identification does not imply recommendation or endorsement nor does it imply that the equipment and materials are necessarily the best available for the purpose.

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The present investigation was conducted to assess the potential clinical performance capabilities of three resin-based die materials.<sup>+</sup>

#### MATERIALS AND METHODS

The resins were obtained from proprietary sources. Each product was proportioned and mixed in accordance with its manufacturer's instructions. Instructional information supplied with Epoxydent did not include a recommended setting time. Therefore, this material was allowed to set for the minimum period of time that permitted manipulation of specimens without producing distortion.

Determination of mechanical properties. Specimens for the determination of compressive strength were 6 mm X 12 mm cylinders. Tensile specimens were 6 mm X 0.5 mm discs. The ends of the test pieces were surfaced plane and perpendicular to their axes with aqueous slurries of 240-grit abrasive powder.

Compressive strength and tensile (diametral compressive) strength were determined on a constant strain rate testing machine<sup>#</sup> at a cross-head speed of 0.02 in per minute. Initial strength values for Epoxydent and Epoxy Die Material were obtained at the time of setting of the specimens. Subsequent measurements were made on specimens aged 2 hours, 24 hours and 7 days. Data for Pri-Die were obtained at 18 hours, 24 hours and 7 days after mixing the resin.

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<sup>+</sup> Epoxydent, Oxydental Products, Inc., Irvington, NY; Epoxy Die Material, Dentsply International, Inc., York, PA; and Pri-Die, J. F. Jelenko and Co., Inc., New Rochelle, NY.

<sup>#</sup> Instron Universal Testing Machine, Instron Engineering Corp., Canton, MA.

Hardness specimens were made in 20 X 25 mm cylindrical molds. One end of each resin cylinder was kept in contact with a glass plate throughout the setting process to obtain a smooth test surface. Superficial hardness (Rockwell 15N) was measured at times corresponding to those at which strength measurements were made.

Quantitative determination of inorganic filler content. Three hardened discs (6 mm X 0.5 mm) of each product weighing approximately 0.07 gram each were heated individually in porcelain crucibles for 3 hours at 650° C. Nonvolatile materials remaining in the crucibles were dried over anhydrous calcium chloride for 24 hours and weighed.

Determination of compatibility with impression materials. The compatibility of the test materials with a polysulfide,<sup>§</sup> a polyether<sup>¶</sup> and three silicone<sup>Ω</sup> impression materials was assessed by the method described in American Dental Association Specification No. 19.<sup>5</sup>

Determination of dimensional accuracy and detail reproduction. The determination of dimensional accuracy under simulated clinical

§ Permlastic Light Bodied Impression Material, Kerr Sybron Corp., Romulus, MI.

¶ Polyjel, L. D. Caulk Co., Milford, DE.

Ω Xantropren, Unitek Corp., Monrovia, CA, Citricon, Kerr Sybron Corp., Romulus, MI, and President Light Bodied Impression Material, Coltene Inc., Alstatten, Switzerland.

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conditions was accomplished using a brass model which represented a partially edentulous segment of a dental arch. Orientation pegs placed in the base of the model insured accurate seating of custom aluminum impression trays. The trays were machined to allow coverage of the model with a uniform thickness (2 mm) of the impression material.

Impressions of the brass model were made using polysulfide elastomers<sup>II</sup> in a simultaneous double mix syringe technique. All impressions were cured at 37° C. and 100 percent relative humidity. Twenty minutes after removal from the brass model, each impression was filled with a resin-based die material. The resultant models were allowed to harden under ambient conditions (~23° C. and 50 percent relative humidity) prior to further handling.

Reproducible measurement of the brass model's horizontal dimension was facilitated by reference lines. The mean value and standard error of ten measurements made at a magnification of X 32 with the use of a traveling microscope<sup>φ</sup> were 38.477 and 0.004 mm respectively. Resin replicas were measured similarly, but at times corresponding to those at which mechanical properties of the test materials were determined. Five replicas of each test material were measured in triplicate at each time interval.

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II Permlastic Light Bodied and Heavy Bodied Impression Material,  
Kerr Sybron Corp., Romulus, MI.

φ Gaertner Scientific Corp., Chicago, IL.

Further assessment of dimensional accuracy of the die materials was made using a plastic model of a maxillary molar.<sup>\*\*</sup> The root-portion of the model was mounted in an autopolymerizing resin and its crown prepared to receive a cast full coverage restoration. Custom trays were fabricated from a tray acrylic.<sup>++</sup> The design of the trays insured coverage of the preparation with a uniform thickness (2 mm) of impression material. Impressions and replicas of the preparation were made as described for the brass model. Dies poured from a type IV dental stone<sup>##</sup> served as controls.

Three dies of each test material and the control were stored for 24 hours at ambient temperature and humidity prior to further manipulation. An additional set of three dies for each test material and the control were stored under ambient conditions for 7 days.

Full crown wax patterns were constructed on the dies. The patterns were invested in a high heat investment<sup>§§</sup> and cast in a high-fusing precious alloy<sup>¶¶</sup> with the aid of an automatic induction casting machine<sup>ΩΩ</sup>. Each casting was seated in turn on its die and the prepared tooth. Subjective assessment of fit was made independently by two dentists.

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\*\* Columbia Dentoform Co., New York, NY.

++ Fastray, Harry J. Bosworth Co., Chicago, IL.

## Velmix, Kerr Sybron Corp., Emeryville, CA.

§§ Ceramigold, Whip Mix Corp., Louisville, KY.

¶¶ Cameo, J. F. Jelenko and Co., Inc., New Rochelle, NY.

ΩΩ Electromatic Casting Machine, Howmet Corp., Chicago, IL.

The ability of each test material to reproduce fine detail was assessed by the procedure described in American Dental Association Specification No. 25.<sup>5</sup>

#### RESULTS

A setting time of 4 hours appeared to be adequate for specimens made from Epoxydent. Recommended setting times of 5 hours for Epoxy Die Material and 10 hours for Pri-Die were sufficient for specimens made from these products.

Data on the apparent mechanical properties of the die resins are summarized in Table I. Initial compressive strengths of Epoxydent and Epoxy Die Material were 9,500 psi and 9,300 psi, respectively. Maximum compressive strengths for both materials were obtained at 7 days after setting. Pri-Die exhibited an initial compressive strength of 14,200 psi. Significant changes in the compressive strength of Pri-Die were not shown by specimens aged beyond 18 hours.

Severe plastic deformation of Epoxydent specimens precluded measurement of initial tensile strength and tensile strength at 2 hours after setting. Maximum tensile strength of Epoxydent (4,100 psi) was attained at 7 days, whereas that of Epoxy Die Material (3,600 psi) was reached at 2 hours. Pri-Die exhibited an initial tensile strength of 3,900 psi. Subsequent measurements did not demonstrate a significant increase in tensile strength.

Initial hardness values (Rockwell 15N) ranged from 26 for Epoxydent to 83 for Pri-Die. Hardness increased with advancing age of the specimens.



All of the test materials successfully counterreplicated a 0.025 mm line inscribed on a stainless steel block.

Subjection of the die resins to pyrolysis revealed that the nonvolatile inorganic filler contents of Pri-Die, Epoxydent and Epoxy Die Material were 49, 48 and 26 percent by weight, respectively.

Values for the linear dimensional changes of the test resins under simulated clinical conditions are shown in Table II. Shrinkage was a characteristic feature of Epoxy Die Material and Pri-Die at the time of initial measurement and of all of the materials at 24 hours. Initially, however, models produced from Epoxydent were larger than the master die.

The comparative assessment of the accuracy of full crowns fabricated on resin and stone dies is summarized in Table III. All castings demonstrated acceptable marginal fit on their respective dies. All castings made on conventional stone dies exhibited well fitting margins when placed on the prepared tooth. Castings fabricated on 24-hour-old Epoxydent dies were found to be acceptable when transferred to the prepared tooth. Epoxydent dies aged 7 days as well as all dies made from the other test materials yielded castings which failed to seat completely on the prepared model.

All test resins exhibited smooth nontacky surfaces when cured against Xantropren or Polyjel. However, contact surfaces of specimens poured in Citricon impressions were soft and tacky.

Surface improvement of Epoxy Die Material and Pri-Die was accomplished with the use of recommended die separators prior to

pouring the resins. Improvement of the surface features of Epoxydent was not observed.

Permlastic showed a tendency to adhere to the fine detail of the resin models. Resins poured against President impression material showed macroscopic surface and subsurface porosity. Application of die separators reduced the adherence of Permlastic to the die resins but did not reduce the porosity observed with President.

#### DISCUSSION

The apparent properties of the test resins are remarkably similar. The small, but measurable property differences are probably not significant clinically.

Observations on the setting characteristics of Epoxydent were not consistent with a two-hour setting time reported by other investigators.<sup>2</sup> Factors such as batch variation, storage conditions, age or changes in product formulation may account for the discrepancy.

Early compressive strengths of the test resins tend to be higher than those of improved dental stones.<sup>6</sup> At 24 hours, however, the compressive strengths of the resins and stones are comparable. On the other hand, hardness and tensile strength data would suggest that resin dies aged beyond 24 hours would be more durable than stone dies of similar age.

Abrasion resistance<sup>2,3</sup> and high tensile strength are the most noteworthy mechanical features of the resin die materials. However, adherence to meticulous techniques of waxing and metal finishing may

preclude the need for materials exhibiting these characteristics. Additionally the adverse effects of polymerization shrinkage appear to outweigh the attribute of durability.

#### SUMMARY

Some mechanical properties, physical characteristics and manipulative features of three resin-based die materials were assessed. Advantages afforded by the strength and hardness of the test materials were overshadowed by relatively long setting times, incompatibility with certain impression materials and polymerization shrinkage. All dies aged beyond 24 hours were found to be unsuitable for use in precision fixed prosthodontic procedures.

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Table I. Apparent mechanical properties of resin die materials

Material	Compressive Strength (psi)	Tensile Strength (psi)	Hardness (Rockwell 15N)
<b>Epoxydent*</b>			
0 hours	9,500±500	---	26±3
2 hours	9,600±400	---	71±3
24 hours	13,000±200	3,400±100	94±2
7 days	15,900±100	4,100±100	99±1
<b>Epoxy Die Material*</b>			
0 hours	9,300±300	2,700±200	64±2
2 hours	11,200±100	3,600±200	75±1
24 hours	12,000±100	3,200±200	91±1
7 days	13,000±200	3,500±200	94±1
<b>Pri-Die<sup>+</sup></b>			
18 hours	14,200±200	3,900±200	83±2
24 hours	13,300±200	3,700±300	85±3
7 days	14,700±100	4,000±200	95±1

\* Time lapse between setting of the resin and specimen testing

+ Time lapse between mixing of the resin and specimen testing

Table II. Linear dimensional changes of resin-based die materials under simulated clinical conditions

Epoxydent <sup>*</sup>	% Change	Epoxy Die Material <sup>*</sup>	% Change	Pri-Die <sup>+</sup>	% Change
0	+0.10±.02	0	-0.10±.05	18 hours	-0.03±.03
2 hours	0.00±.03	2 hours	-0.11±.04		
24 hours	-0.04±.05	24 hours	-0.11±.01	24 hours	-0.08±.03
7 days	-0.11±.06	7 days	-0.10±.03	7 days	-0.10±.02

\* Time lapse between setting of the resin and specimen measurement

+ Time lapse between mixing of the resin and specimen measurement

Table III. Marginal fit of full coverage castings

Die Material	Fit on Die		Fit on Prepared Tooth	
	24 hours	7 days	24 hours	7 days
Epoxydent	Acceptable	Acceptable	Acceptable	Not Acceptable
Epoxy Die Material	Acceptable	Acceptable	Not Acceptable	Not Acceptable
Pri-Die	Acceptable	Acceptable	Not Acceptable	Not Acceptable
Velmix*	Acceptable	Acceptable	Acceptable	Acceptable

\* Control