Resistance to Staining, Flexural Strength, and Chemical Solubility of Core Porcelains for All-Ceramic Crowns

Josephine F. Esquivel-Upshaw, DMD, MS[a]
John Chai, DMD, MS, MJ, DLaw[b]
Sandra Sansano, DDS[c]
David Shonberg, DDS[d]

Purpose: The increased demand for tooth-colored restorations has prompted the use of ceramics in areas that are subject to masticatory stresses. To maximize the strength of these restorations, manufacturers and clinicians advocate placement of core materials in lieu of veneering materials in areas that are more susceptible to fracture. The objectives of this study were to determine the: resistance to staining of three core porcelains used for all-ceramic restorations, Procera, IPS Empress, and In-Ceram, through the use of colorimetry and visual observation; flexural strength of these porcelains under a three-point bend test; and chemical solubility in a controlled environment. **Materials and Methods:** L*a*b* values were obtained for each specimen before and after immersion in a saturated solution of methylene blue in ethanol for 24 hours. Visual observation was also performed to ascertain color differences before and after staining. A three-point bend test was used to determine flexural strength. A reflux-condenser type, three-piece extraction apparatus was used with 4% acetic acid solution for 16 hours to determine solubility. Each sample was weighed before and after the reflux procedure to ascertain percentage weight loss. **Results:** The mean ΔE obtained for IPS Empress was 14.5; In-Ceram was 9.2, and Procera was 9.0. Flexural strength obtained for IPS Empress, In-Ceram, and Procera was 176.9 MPa, 323.4 MPa, and 464.3 MPa, respectively. Weight loss of IPS Empress, In-Ceram, and Procera was 0.056%, 0.734%, and 0.003%, respectively. **Conclusion:** IPS Empress showed the least resistance to staining. IPS Empress had the lowest flexural strength, while Procera had the highest. In-Ceram demonstrated the highest chemical solubility. *Int J Prosthodont 2001;14:284–288.*

The increased awareness of esthetics in dentistry has brought the advent of all-ceramic restorations into clinical practice. Several ceramic systems have inundated the market with promises of excellent color match, longevity, and compatibility. Most notable are commercial systems like IPS Empress (Ivoclar), In-Ceram (Vident/Vita), and Procera (Nobel Biocare).

IPS Empress is a heat-pressed ceramic system that uses the lost-wax technique. It is a combination of feldspathic porcelain (SiO_2 and Al_2O_3) with leucite crystals.[1] After waxing the crown to its proper shape and contour, it is invested in a special flask with a special type of investment material. The desired shade of a precerammed ceramic cylinder is plasticized at 1,100°C and pressed under vacuum and pressure into the mold of the investment.[2]

In-Ceram claims enhanced strength by using a glass-infiltration system over a porous core framework, thereby minimizing porosities.[3] The core framework is initially produced from slip-cast alumina that is partially sintered in a furnace. It is composed of a fine particle–sized aluminum oxide, which also contributes to its strength.[4–6]

Procera promotes the use of a computer-aided design/manufacturing (CAD/CAM) technology wherein the working die is digitized and enlarged by 20%.[7]

[a]Assistant Professor, Department of Prosthodontics, University of Florida College of Dentistry, Gainesville.
[b]Associate Professor, Associate Dean for Academic Affairs, and Director of Graduate Prosthodontic Program, Northwestern University, Chicago.
[c]Private Practice, Caracas, Venezuela.
[d]Private Practice, Chicago.

Reprint requests: *Dr Josephine F. Esquivel-Upshaw, University of Florida College of Dentistry, PO Box 100435, Gainesville, Florida 32610. Fax: + 352-846-0248. e-mail: jesquivel@dental.ufl.edu*

The enlarged die is then used for the dry-pressing technique, in which high-purity alumina powder is compacted to form an aluminous coping. Temperatures of up to 1,550°C are used to sinter this coping, which is then thermal etched prior to application of veneer porcelain. The whole system is known as the Procera technique.

In spite of advancing technology, strength is still a limitation with these restorations and has yet to equal the fracture resistance and durability of gold or porcelain-fused-to-metal crowns. To maximize the strength of these all-ceramic restorations, clinicians and manufacturers advocate the placement of core materials in areas, such as the occlusal surfaces, that are more susceptible to fracture in the hope of improving the physical properties. American Dental Association (ADA) specification No. 69[8] was formulated to examine the properties of all-ceramic restorations. Provisions for meeting the criteria under this specification are required for ADA acceptance.

The purposes of this study were to determine the: *(1)* resistance to staining of three core porcelains used for all-ceramic restorations, Procera, IPS Empress, and In-Ceram, through the use of colorimetry and visual observation; *(2)* flexural strength of these porcelains under a three-point bend test; and *(3)* chemical solubility in a controlled environment.

Materials and Methods

Resistance to Staining

Ten samples of each porcelain were fabricated from a 20 mm × 2 mm mold as recommended by ADA specification No. 69.[8] Each porcelain disk was ground to a final finish of 15 µm using 600-grit sandpaper.

A colorimeter was used to obtain objective measurements of color for each sample with the use of a Minolta Chroma Meter CR-300.[9,10] The colorimeter was calibrated according to the manufacturer's instructions. The CIELAB system was used to determine color differences within the samples through measurement of their L*a*b* values and computation of their ΔE, which is derived from the formula[10]:

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

L*a*b* values were made for each specimen, with three readings being averaged for each. All 30 disks were immersed in a saturated solution of methylene blue in ethanol for 24 hours. The samples were then cleaned with methylated spirit in an ultrasonic cleaner for 15 seconds. L*a*b* values were again recorded three times and averaged. Each disk was also observed visually to determine any change in color. Color deviation (ΔE) was computed for each sample.

Flexural Strength

Ten samples were requested from each manufacturer and ground to an approximate size of 5.0 ± 0.25 mm in width, 1.0 ± 0.2 mm in thickness, and 21 mm in length.[8] The dimensions were confirmed using a micrometer (Mitutoyo). All samples were brought to a uniform finish of 600 grit, which is equivalent to 15-µm particle size, using sandpaper (Buehler Scientific). The samples were cleaned using distilled water in an ultrasonic cleaner for 5 minutes. A universal testing machine (model 8501, Instron) was used with a three-point bending test to determine flexural strength. The span was fixed at 13.55 mm between the 1.6-mm-diameter supports. The rate of load application was adjusted to 0.5 N per second. The midpoint of the test beam was located, and the test beam was placed centrally between the supports. The load was applied to a 5-mm-wide face along a line perpendicular to the long axis of the beam. The load required to break the test piece was measured to the nearest 0.1 N. The cross-sectional dimensions (width and thickness) of each test piece were measured to the nearest 0.01 mm at the point where the fracture occurred.

The flexural strength, M, of each test piece was calculated using the formula:

$$M = 3Wl/2bd^2$$

where W = load at failure, in N; l= the test span (center-to-center distance between bearers), in mm; b = the width of the specimen or the dimension of the side at right angles to the direction of the applied load, in mm; and d = the thickness of the specimen or the dimension of the side parallel to the direction of the applied load, in mm.

Chemical Solubility

Ten samples were fabricated using a circular mold with a diameter of 16 mm and depth of 1.6 mm. Specimens were polished to a final finish of 15 µm using 600-grit sandpaper. A reflux-condenser type, three-piece extraction apparatus (Pyrex, Corning Scientific) was used. Each specimen was placed separately in glass-bottomed thimbles. Each thimble and specimen were conditioned to constant weight by storing at 150 ± 3°C in a furnace (Precision Scientific). The conditioning involved drying the samples repeatedly in a glass dessicator for 30 minutes prior to weighing them with an electronic balance (Mettler Instruments). Samples were repeatedly weighed within

Table 1 Materials Tested

	IPS Empress		In-Ceram		Procera	
	Mean	SD	Mean	SD	Mean	SD
Resistance to staining (ΔE)	14.5	3.6	9.2	4.1	9.0	2.8
Flexural strength (MPa)	176.9	13.0	323.4	51.9	464.3	41.3
Chemical solubility (% weight loss)	0.06	0.08	0.70	0.15	0.003	0.006

SD = standard deviation.

a 24-hour period until three readings that were within 0.1 mg were obtained. This value was recorded as W1. The thimbles were placed in the extraction apparatus, and the specimens were extracted using 4% acetic acid solution by refluxing for 16 hours with a reflux rate of 18-minute cycles. Each specimen was washed in the thimble with distilled water. The acetic acid and the wash were discarded. The thimble and the specimen were once again conditioned to constant weight (to the nearest 0.1 mg) at the abovementioned temperature within a 24-hour weighing cycle. The weighing procedure was repeated, and the value was noted as W2. The percentage loss in mass for each specimen was calculated using the formula:

$$(W1 - W2/W1) \times 100$$

Statistical Methods

All data were subjected to an analysis of variance (ANOVA) and Neuman-Keuls post hoc test to determine any significant difference among the groups at *P* = .05.

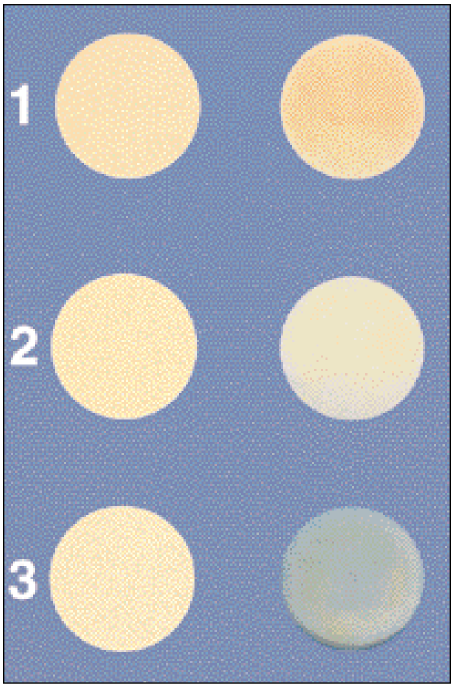

Fig 1 Procera *(1),* In-Ceram *(2),* and IPS Empress *(3)* disks before and after staining (*left* and *right* columns, respectively).

Results

The mean ΔE obtained for IPS Empress was higher than for In-Ceram and Procera (Table 1). One-way ANOVA followed by Newman-Keuls post hoc analysis indicated IPS Empress to show a significantly higher ΔE compared with the Procera and In-Ceram ($P < .05$). Visual observation also revealed all samples to be visibly stained, with the intensity of the staining of IPS Empress samples being greater than the other two (Fig 1).

The mean flexural strength obtained for IPS Empress, In-Ceram, and Procera was 177 MPa, 323 MPa, and 464 MPa, respectively (Table 1). A significant difference existed among all of the core porcelains, with Procera demonstrating the greatest flexural strength ($P < .05$).

The percentage weight loss of In-Ceram was greater than for the other two materials (Table 1). Statistical analysis revealed In-Ceram to differ significantly from IPS Empress and Procera ($P < .05$), and a difference was also detected between IPS Empress and Procera porcelains.

Discussion

Resistance to Staining

Color is a difficult quality to measure, and the use of a colorimeter permits some degree of quantitative analysis of color difference.[10] However, the clinical significance of the value of color differences (ΔE) has yet to be established. Goldstein and Schmitt[11] proposed that a ΔE greater than 0.4 can already be detected by the highly trained human eye. O'Brien et al,[12] on the other hand, classified the different values of ΔE, with 1 being excellent, 2 clinically acceptable, and 3.7 a poor match, based upon clinical observations. The ADA has itself established a ΔE value of 2 as the tolerance limit for shade guides.[13] Esquivel et al[14] performed a similar experiment involving low-fusing porcelains and established ΔE values of 0.34 to 0.88 to be the tolerance of the repeatability of measurement of the colorimeter with samples immersed in distilled water. The color deviation values for core porcelains immersed in methylene blue ranged from 9.02 to 14.47. Although the value of the clinical significance of ΔE has not been established, color deviation values in this range were clinically discernible. There seems to be a correlation between the value of ΔE and the visual assessment of staining. This was concluded in an earlier study,[15] wherein human observer responses were compared with colorimetric devices and both methods of color evaluation were found to be easily reconciled with the use of the CIELAB system. Further studies are required to determine the value at which color deviation becomes detectable to the human eye.

The significant difference noted with IPS Empress could be attributed to its larger particle size (10 to 15 µm) in the reinforcing phase versus Procera and In-Ceram (1 to 5 µm). Removal of the glaze exposes larger particles, producing a rougher surface that is more conducive to staining.

Although it can be argued that core porcelains could be glazed to prevent seepage of staining materials, ADA specification No. 69[8] calls for removal of the glaze prior to staining. The results of this protocol could therefore be likened clinically to porcelain surfaces, which are adjusted but not glazed. The fine-grit abrasive used in this experiment could be likened to a diamond bur commonly used to adjust porcelain clinically. It is therefore recommended that glazing should follow clinical adjustments of porcelain crowns to decrease the possibility of staining. The severity of the methylene blue stain can also be likened to everyday exposure to substances like coffee, tea, or cigarettes. This in vitro study shows an accelerated effect of what could happen through the course of several years of exposure to these substances.

Flexural Strength

A significant difference was noted in the flexural strengths of all core-ceramic groups. ADA specification No. 69[8] requires that the lower limit of flexural strength be at least 100 MPa. Although all core materials met this criterion, IPS Empress nevertheless exhibited the lowest flexural strength. The reason for this could be the composition of the material itself. IPS Empress was conceived with a lot of significant developments, namely heat pressing the ceramic, which significantly improved the strength.[16] It was noted[2] that leucite crystals in the ceramic ingots were initially clustered and that the softening and pressing process allowed them to disperse more evenly in the ceramic material. This homogenous distribution of crystals provides for greater strength of the ceramic. Subsequent firing enables a coefficient of thermal expansion difference to exist between the glass and the leucite crystals, which results in compressive stresses in the glassy phase of the ceramic. This provides for increased strength and resistance to fracture. However, there have been several recommendations to modify the manufactured IPS Empress ingots or the pressing procedures because of the problem of leucite crystals clustering.[17] As mentioned earlier, compressive stresses are generated by exposure of the material to elevated temperatures above 800°C. This promotes further crystalline growth and therefore increases the mechanical tension between the crystals and the glassy phase. The important factor here is that the crystals must be evenly distributed, that is, not clustered together; otherwise, the opposite effect will be seen.[2,18–20] A decrease in flexural strength can also be seen if larger-sized leucite particles are used.

Procera and In-Ceram have unique properties that contribute to their greater strength. As explained earlier, In-Ceram undergoes a process called glass infiltration that eliminates porosities, thereby increasing the strength of ceramics.[21,22] This allows for the creation of an interpenetrating network between the alumina and glass. Both substances act synergistically to increase the strength of the material. Alumina serves to deflect fractures and prevent or minimize crack propagation. Although the tendency would be for the fracture to proceed to the glassy phase, which is a weaker material, the interpenetrating network serves as a medium where alternating phases of alumina and glass are encountered. Therefore, there is no single path of least resistance in the material.[23,24] Procera has a very high alumina content, in the range of 99%, which explains the increased fracture resistance over the other two core ceramics.[25,26]

Chemical Solubility

Chemical solubility is an important property in that it directly affects strength as well as the esthetic value of the restoration. Surface irregularities can lead to eventual failure of the restoration because of crack propagation[22] and can also serve as niches for plaque retention. ADA specification No. 69[8] allows for a maximum of 0.5% weight loss for core porcelains. The International Standards Organization standard No. 6872 for dental ceramics was modified in 1995 to allow for a maximum of 2,000 µg/cm^2 for both type I (ceramic products that are provided as a powder) and type II (ceramics used for the fabrication of the supporting structure for crowns, veneers, inlays, and onlays; thus, materials that are intended to be layered with one or more type I materials) ceramics.[27] The procedural testing for both standards is essentially the same with slight modifications. IPS Empress and Procera were both within the limit allowed by ADA specification No. 69,[8] but In-Ceram exceeded this by 0.2%. The implication of this value is unclear in that this test does not truly reflect the effects of clinical saliva in the mouth. An earlier study[28] concluded that the effect of 4% acetic acid used for 1 week at 80°C can be likened to immersion in artificial saliva at 22°C for 22 years. Therefore, the chemical solubility of In-Ceram will probably occur over the course of several years and will prove to be negligible over time, although clinical studies will be needed to determine the validity of this. It is recommended that clinical studies be conducted to examine the effects of acidulated phosphate fluoride gel and citric acid on the solubility of these porcelains.

Conclusions

Three core porcelains, IPS Empress, In-Ceram, and Procera, were tested to determine their resistance to staining, flexural strength, and chemical solubility. The following conclusions were derived:

- IPS Empress demonstrated a significantly higher ΔE compared to Procera and In-Ceram. Visual observation confirmed intensified staining with this ceramic.
- All of the core porcelains exhibited a significant difference in flexural strength, with IPS Empress having the lowest and Procera the greatest.
- In-Ceram demonstrated a significantly higher chemical solubility than IPS Empress and Procera.

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