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### ABSTRACT

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Liquid mediums used with opaque porcelain powders are crucial in the application of opaque slurries to oxidized metal surfaces. To augment the effective application of opaque porcelain, liquid mediums should wet oxidi() zed metal surfaces well (indicated by a low contact angle). The liquid-metal-surface interaction is a critical factor in the wetting of alloys by liquid suspensions. A multifactorial treatment design was used to evaluate the effects of various metals, liquids, surface preparation, and possible interactions on wetting. Five opaque liquid mediums ((Ceramco, Biobond, Ney, Vita, and Will-Ceram) were evaluated photographically by sessile drop contact angle measurements on five oxidized metal surfaces (A-33, Bake-On N/P, Option, Rexillium-III, and Triumph). Contact angle measurements were made on both smooth and sandblasted metal surfaces after an equilibrium period of one minute. The data were treated in a fully crossed design with eight replications per combination. Vita liquid demonstrated the lowest contact angles followed by Ney, Ceramco, Will-Ceram, and Biobond liquids. The statistical significance of this ranking varied with each of the five metals and two surfaces evaluated. Surface preparation had no significant effect on the contact angles of the noble metals (A-33 or Option) for any of the liquids evaluated. Differences in surface preparation were significant for Vita liquid on all three base metal alloys evaluated (Bake-On N/P, Rexillium-III, and Triumph), but Ney and Will-Ceram liquids demonstrated significant differences for surface preparation on Triumph metal only.

The Wetting Of Ceramic Opaque Suspensions On Oxidized Alloy Surfaces

Gerald D. Woolsey, DDS, MS Lewis Lorton, DDS, MSD William J. O'Brien, PhD



### ABSTRACT

Liquid mediums used with opaque porcelain powders are crucial in the application of opaque slurries to oxidized metal surfaces. To augment the effective application of opaque porcelain, liquid mediums should wet oxidiized metal surfaces well (indicated by a low contact angle). The liquid-metal-surface interaction is a critical factor in the wetting of alloys by liquid suspensions. A multifactorial treatment design was used to evaluate the effects of various metals, liquids, surface preparation, and possible interactions on wetting. Five opaque liquid mediums (Ceramco, Biobond, Ney, Vita, and Will-Ceram) were evaluated photographically by sessile drop contact angle measurements on five oxidized metal surfaces (A-33, Bake-On N/P, Option, Rexillium-III, and Triumph). Contact angle measurements were made on both smooth and sandblasted metal surfaces after an equilibrium period of one minute. The data were treated in a fully crossed design with eight replications per combination. Vita liquid demonstrated the lowest contact angles followed by Ney, Ceramco, Will-Ceram, and Biobond liquids. The statistical significance of this ranking varied with each of the five metals and two surfaces evaluated. Surface preparation had no significant effect on the contact angles of the noble metals (A-33 or Option) for any of the liquids evaluated. Differences in surface preparation were significant for Vita liquid on all three base metal alloys evaluated (Bake-On N/P, Rexillium-III, and Triumph), but Ney and Will-Ceram liquids demonstrated significant differences for surface preparation on Triumph metal only.

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### INTRODUCTION

The metal-opaque interface is the key to the bond of porcelain-metal restorations. Solutions of glycerin in water are generally used in dentistry to carry the porcelain opaque to the metal surface. The use of one opaque liquid with different combinations of porcelain, alloy, and alloy surface preparation have been observed to yield different wetting characteristics (GDW). The various liquid media used with porcelain opaque powders do not have the same apparent working characteristics, some of the liquids used with porcelain opaque powders appeared to wet metal surfaces more easily than others. This improved wetting can eliminate voids due to air entrapment in mechanical retentive areas of the metal surface at the opaque-metal interface. Improved wetting also eases the application of the opaque suspension to the oxidized metal surface. Liquids with lower surface tension have lower contact angles; this can allow trapped air to escape during mechanical agitation and vibration of the opaque suspension onto the metal surface. Two types of metal surface preparation are commonly used -- grinding with silicone oxide stones and air-abrading with 50 micron alumina (sandblasting). Both techniques are widely used, and are considered to be similar in effect.

The purpose of this study was to evaluate the effects of five different opaque porcelain liquids on five different metals. Each metal was prepared by two different techniques--half were finished smooth and the other surfaces were air-abraided. The liquid-metal-surface-preparation interaction with eight replications for each combination was evaluated. The evidence suggests that wetting can be enhanced by proper matching of liquid-metal-surface preparation.

## Review of the literature

The technique of forming ceramics from liquid, called "wet plastic forming,"<sup>1</sup> is used to form brick, whiteware, molds, and enamelware. The liquids used with ceramic powders bind the particles together during shaping of the mass while the ceramic is in a prefired "green" stage.

The quantity of fluid necessary to emulsify a ceramic powder is dependent upon the surface tension and density of the liquid, the density and particle size of the powder, and the presence of deflocculants, such as sodium silicate. The deflocculants reduce agglomeration of the particles and thereby reduce the quantity of fluid necessary to create a controllable mass of suspension.2

The fluid used to suspend a ceramic powder must have qualities which ensures that it will evaporate uniformly thus allowing the ceramic particles to become closely packed. The process of gradual evaporation must not seal moisture within the ceramic during

sintering. If moisture is sealed in the mass, the sintered ceramic will crack as the underlying moisture escapes.<sup>3</sup> Boiling temperature, surface tension of the liquid, plus density and particle size of the ceramic powder directly influence the rate of evaporation of the liquid from a ceramic suspension.<sup>2</sup> Surface tension of the suspension and the free surface energy of the metal substrate drastically affect the ability of the suspension to wet the substrate surface. Measuring the contact angle between the suspension and the substrate surface can provide considerable information about the ability of a liquid suspension to wet or spread on a surface. If the contact angle of the liquid suspension on the substrate surface is high (approaching 90 degrees), the tendency for the suspension to spread evenly over that surface is less<sup>4</sup>; by contrast, if the contact angle is low, the liquid spreads readily over the surface.

Although the effects of metal surface preparation on wetting by ceramic suspensions has yet to be studied, the effects of metal surface preparation on bond strength has been observed to some extent.<sup>5-7</sup> Lavine and Custer<sup>5</sup> found a slight increase in bond strength of gold-based alloys with roughened surfaces. Other investigators<sup>6-7</sup> have found no significant difference in roughened base metal alloys and alloys of low metal noble content in relation to surface roughness.

O'Brien and Ryge<sup>8</sup> emphasized the importance of wetting ceramic metal with porcelain during firing. The "cohesive plateau theory"<sup>9</sup> suggests the cohesive strength of porcelain is weaker than the

adhesive strength of porcelain-to-metal. This theory diminishes previous emphasis on porcelain-metal bond testing and stresses that bond tests previously reported<sup>6</sup> really measure cohesive bond strength rather than adhesive bond strength.

Materials and methods

Five brands of porcelain opaque liquid\* and one porcelain opaque powder¶ were evaluated on five oxidized ceramic metals<sup>§</sup> prepared with two different surface preparations.

A single opaque powder was used to minimize the effects of powder content and particle size on the surface tension of the opaque suspensions. The weight of the powder used with each liquid was determined by incremental additions of 10 mg of opaque powder to 0.05 cc of liquid. The optimum quantity of powder was determined when the next larger addition of powder to the slurry inhibited the formation of a distinguishable drop of slurry (Table 1).

- \* Liquid Brands: Ceramco Liquid Medium, Ceramco, Inc., East Windsor, NJ; Will-Ceram Poreclain Liquid, Willaims Gold Co., Buffalo, NY; Neydium Opaque Medium, J.M. Ney Co., Bloomfielf, CT; Biobond Opaquing Medium, Dentsply International, York, PA; Vita-P Modeling Fluid, Unitek, Monrovia, CA.
- V Opaque Powder: Ceramco-G Paint-O-Pake (orange), Ceramco Inc., East Windsor, NJ
- <sup>5</sup> Metals: Jelenko Triumph, J.F. Jelenko Gold Co., Armonk NY 10504; Rexillium III, Rx Jeneric Gold Co., Wallingford, CT 06493; Option, J.M. Ney Co., Bloomfield, CT 06002; Bake-On N/P, Ceramco Inc., East Windsor, NJ 08520; A-33, Howmedica Inc., Dental Division, Chicago, IL 60632

Metal discs (24 mm X 1.5 mm) were cast from each alloy by conventional lost-wax technique. The surfaces of the metal discs were ground flat and finished on 240 to 600 grit metallographic papers. Each metal blank was prepared by air-abrading with 50 micron alumina on one side, and the other side was left smooth. All metal blanks were oxidized as prescribed by the respective alloy manufacturer. The prepared metal blanks were placed on a flat surface and a small amount of porcelain suspension was picked up with a small cement spatula, held a standard distance of 10 mm from the metal surface. The spatula with the suspension was hand-vibrated with a Lecron carver until one uniform drop fell to the oxidized metal surface. Photographs of contact angles were made within the first 5 minutes of application. The contact angle for each sample was measured directly from photographic prints. Eight replications were made for each liquid-alloy-surface-finish combination and the data were treated in fully crossed design.

The results were analyzed in a completely crossed 3-way analysis of variance by using the BMDP2v program on a DATA General MV8000. Post hoc tests were a Bonferroni-type t test which limited the Type I error to 0.05 (Table 2).

### Results

<u>Noble Metals</u> (Howmedica A-33 and Ney Option) Surface preparation (sandblasting vs smooth) had no significant effect at the 0.05 level on the contact angles of the high gold ceramic alloy (Howmedica A-33) but was significant on the palladium alloy (Ney "Option") with Ceramco, Biobond, and Ney liquids. Vita, Ceramco, and Biobond liquids demonstrated significantly lower contact angles on Howmedica A-33 alloy than Ney or Will-Ceram liquids on the same alloy. Vita and Ceramco liquids also demonstrated lower contact angles on Ney Option alloy than Biobond, Ney or Will-Ceram liquids on the same alloy (Figures 1 and 2; Tables 3 and 4).

<u>Base Metals</u> (Bake-On N/P, Rexil<sup>1</sup> um III, and Jelenko Triumph) Differences in surface preparation were significant for Vita liquid on all three base alloy alloys. Ney and Will-Ceram liquids demonstrated significant differences for surface preparation on Jelenko Triumph alloy only. All other liquids and base alloy combinations failed to demonstrate any statistically significant effects on wetting by surface preparation (Figures 3 to 5; Tables 5 to 7).

### Conclusions

Using a liquid with opaque porcelain powder is essential in applying the opaque to a ceramo-metal surface. In this study, we found that metal surface preparation of a high gold content ceramo metal alloy was not a significant aid in the application of opaque porcelain.

Wetting of base metal alloys was enhanced significantly with Vita liquid on roughened surfaces of Rexillium III, Bake-On N/P, and Jelenko Triumph alloys; Ney and Will-Ceram liquids improved wetting on roughened surfaces of Jelenko Triumph alloy. These data suggest that the application of opaque porcelain to base metal alloys can be facilitated by the proper matching of a liquid and base metal system and, in selected combinations, surface roughness can also improve the wetting of base metal and palladium metal alloys.

### REFERENCES

- 1. Flinn, R.A., and Trojan, P.K. Engineering materials and their applications. Boston, Houghton Mifflin Co., 1981, p 332.
- 2. Kingery, W.D., Bowen, H.K., and Uhlman, D.R. Introduction to ceramics. New York, John Wiley and Sons, 1976, pp 11-15.
- 3. J.M. Ney Co. Neydium porcelain manual. Bloomfield, J.M. Ney Co., 1979, p 23.
- Craig, R.G. Restorative dental materials. St. Louis, C.V. Mosby Co., 1980, p 37.
- 5. Lavine, M.H., and Custer, R. Variables affecting the strength of bond between porcelain and gold. J Dent Res 45(1):32-36, 1966.
- Shell, J.S., and Nielson, J.P. A study of the bond between gold alloys and porcelain. J Dent Res 41(6):1424-1437, 1962.

- Carpenter, M.A., and Goodkind, R.J. The effect of varing surface texture on bond strength of one semiprecious and one non-precious ceramo-alloy. J Prosthet Dent 42(1):86-95, 1979.
- O"Brien, W.J., and Ryge, G. Contact angles of enamels on metals, J Prosthet Dent 15(6):1094-1100, 1965.
- 9. O'Brien, W.J. Cohesive plateau theory of porcelain-alloy bonding. In Yamada, H. and Grenoble, P., eds., Dental porcelain: state of the art-1977. Los Angeles, The University of Southern California, 1977, p 138.

## TABLE 1, Volumes of opaque powder

mixed with 0.05 cc of each liquid

Liquids	Powder mixed with liquids (mg)	Liquid lot numbers
Vita-P Modeling Fluid	140	03257
Neydium Opaque Medium	80	m2008
<b>Ceramco</b> Liquid Medium	140	18001
Will-Ceram Porcelain-	100	no lot number
nidara	100	NO TOC NUMBER
Biobond Opaquing Medium	140	B 80

# TABLE 2, Analysis of Variance

Metal	Source	DF	MS	F	Р
A-33					
	Liquid	4	3450	55 00	0000
	Surface	i	5	.08	.7781
	Interaction	4	81	1.24	. 3004
	Error	70	65		
Bake-On N/P					
	Liquid	4	249	3,14	.0196
	Surface	1	423	5.32	.0240
	Interaction	4	953	11.90	.0000
	Error	70	79		
Rexillium					
	Liquid	4	3894	63.00	.0000
	Surface	1	87	1.42	.2379
	Interaction	4	267	4.34	.0034
	Error	70	61		
Triumph					
_	Liquid	4	801	40.00	.0000
	Surface	1	4545	226.00	.0000
	Interaction	4	519	25.00	.0000
	Error	70	20		
Option					
	Liquid	4	1769	55.00	.0000
	Surface	1	470	14.00	.0000
	Interaction	4	492	15.00	.0000
	Error	70	32		

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TABLE 3, Contact angle values (in degrees) of liquid mediums on A-33 alloy.

Opaque liquid	Surface*	mean	
Vita	SM	28.2	
Vita	SB	33.3	
Ceramco	SM	37.7	
Ceramco	SB	40.1	
Biobond	SM	47.8	
Biobond	SB	45.3	
Ney	SM	64.4	
Ney	SB	57.9	
Will-Ceram	SM	67.7	
Will-Ceram	SB	66.1	

(means connected by brackets are not different at 0.05)

\* SB = Sandblasted surface SM = Smooth surface

#### Surface\* **Opaque** liquid mean Vita SM 39.9 Vita SB 40.0 SM 45.4 Ceramco SB 53.8 Ney Biobond 53.8 SB Ceramco SB 57.4 SB 63.6 Will-Ceram 66.5 Biobond SM 68.8 SM Ney 71.5 Will-Ceram SM

TABLE 4, Contact angle values (in degrees) of liquid mediums on Ney "Option" alloy.

(means connected by brackets are not different at 0.05)

\* SB = Sandblasted surface SM = Smooth surface

Dpaque liquid	Surface*	mean	
Vita	SB	33.4	
Ceramco	SB	39.9	
Ceramco	SM	47.6	
Ney	SB	48.0	
Biobond	SB	49.3	
Biobond	SM	53.8	
Will-Ceram	SB	55.8	
Ney	SM	62.8	
Vita	SM	67.3	
Will-Ceram	SM	70.3	

TABLE 5, Contact angle values (in degrees) of liquid mediums on Jelenko "Triumph" alloy.

(means connected by brackets are not different at 0.05)

\* SB = Sandblasted surface SM = Smooth surface

TABLE 6, Contact angle values (in degrees) of liquid mediums on "Bake-On-N/P" alloy.

Opa	que liquid	Surface*	mean
	 Vita	SB	42.6
	Ney	SM	47.6
	Ceramco	SB	54.4
	Will-Ceram	SM	56.6
	Biobond	SB	58.6
	Ney	SB	59.3
	Ceramco	SM	62.9
	Will-Ceram	SB	65.8
	Vita	SM	66.7
	Biobond	SM	67.8

(means connected by brackets are not different at 0.05)

\* SB = Sandblasted surface SM = Smooth surface

TABLE 7, Contact angle values (in degrees) of liquid mediums on "Rexillium III" alloy.

Opaque liquid	Surface*	mean	
Vita	SM	14.5	
Vita	SB	<b>30.</b> 9	
Ceramco	SB	42.3	
Ceramco	SM	45.1	
Biobond	SM	53.8	
Biobond	SB	54.4	
Will-Ceram	SB	57.3	
Ney	SB	60.8	
Will-Ceram	SM	60.9	
Ney	SM	61.0	

(means connected by brackets are not different at 0.05)

\* SB = Sandblasted surface SM = Smooth surface Fig 1. Contact angles of five opaque porcelain suspensions on "A-33" alloy (Howmedica Inc.), prepared with a smooth surface and a sandblasted surface.

Fig 2. Contact angles of five opaque porcelain suspensions on "Option" alloy (J.M. Ney Co.), prepared with a smooth surface and a sandblasted surface.

Fig 3. Contact angles of five opaque porcelain suspensions on "Bake-On N/P" alloy (Ceramco Inc.), prepared with a smooth surface and a sandblasted surface.

Fig 4. Contact angles of five opaque porcelain suspensions on "Rexillium-III" alloy (Jeneric Gold Co.), prepared with a smooth surface and a sandblasted surface.

Fig 5. Contact angles of five opaque porcelain suspensions on "Triumph" alloy (Jelenko Co.), prepared with a smooth surface and a sandblasted surface.





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Fig 5