Surface Properties of Dental Polymers: Measurements of Contact Angles, Roughness and Fluoride Release

Fátima Namena*, João Galan Jr.b, José Farias de Oliveirac, Rodrigo Derossi Cabreiraa,

Fernando Costa e Silva Filho^a, Alex Balduino Souza^b, Gustavo de Deus^b

^aInstituto de Biofísica Carlos Chagas Filho, CCS, Bloco G,
Universidade Federal do Rio de Janeiro – UFRJ,
Ilha do Fundão, 21949-900 Rio de Janeiro - RJ, Brazil

^bUniversidade Veiga de Almeida – UVA,
Rua Ibituruna, 108, Tijuca, 20271-901 Rio de Janeiro - RJ, Brazil

^cPrograma de Pós Graduação em Metalurgia e Ciência dos Materiais,
Centro de Tecnologia, COPPE, Universidade Federal do Rio de Janeiro – UFRJ,
Ilha do Fundão, CP 68501, 21945-970 Rio de Janeiro - RJ, Brazil

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Objective: Earlier studies on some dental materials measured roughness and/or contact angles or fluoride release separately. In the present study, five dental polymers were investigated to ascertain their contact angles, wettability, roughness, and fluoride release in dry or wet conditions. Methods: Samples for 5 materials were prepared and stored dry or wet in deionized water pH 6.8. Samples were submitted to finishing/polishing procedures, and the measurements in Goniometer, roughness (μ m) and fluoride analysis Results and conclusions: Except for the Ariston pHc, all the materials displayed high contact angles when measured with water, showing hydrophobic characteristics. Roughness changed the contact angles, especially those of Ariston (α < 0.05). Fluoride did not modify the contact angles, but increased the roughness of the finished material.

Keywords: dental polymers, surface roughness, surface hydrophobicity, release of fluoride

1. Introduction

Aesthetic restorative materials of composite resins regularly used in dentistry are composed of polymers to which inorganic particles are usually associated¹⁻⁵. These polymers, mainly BIS-GMA⁶, exhibit several particular characteristics such as the release of residual monomers, microporosity and cracking, etc⁷⁻⁹. All these characteristics seem to be related to surface finishing treatments, particularly those carried out in dental clinics¹⁰⁻¹². In addition, it has been pointed out that either ormocers (organically modified ceramics) or compomers (polyacid-modified resin composites) may be sources of released fluoride particles¹³⁻¹⁵.

Composite dental restorative materials can be modified mechanically by brushing, polishing, abrasion, erosion and microcracking processes, or chemically through an acid medium¹⁶⁻¹⁹. Hydrophobicity, an important characteristic of hard composites, affects the initial water absorption and the adhesion of oral bacteria. Changing the hydrophobicity of the dental polymer surfaces can also affect the adhesion of oral bacteria^{20-23,27}.

The breakdown of the marginal areas between enamel and restorative material can provide potential pathways for bacterial reinfection and recurrence of caries. Like other hard surfaces used in dental restorations, restorative materials are covered with an organic biofilm, which usually consists of host and bacterial elements. Different chemical properties and surface topographies of the various materials may play a role in biofilm formation and influence the differences in composition and general properties from one to another²⁴⁻²⁶.

Several studies have found that the fluoride added to materials can alter wettability and plaque formation, as well as the adherence of *Streptococcus mutans*²⁷⁻³⁰. However, other authors contend that the presence of fluoride on dental materials does not modify the materials' basic characteristics^{31,32}.

The measuring of contact angles at the solid-air-liquid meeting point is a widely known technique used to investigate the wettability of solid surfaces³³. The values obtained depend on the kind of surface topography, surface tension of the liquid and surface energy of the solid substrate^{33,34}. Thus, the relative wetting characteristics of a liquid-solid interface can be inferred by contact angle measurements. It has been shown that the contact angle on a solid surface decreases as the surface becomes rougher³⁸⁻³⁵. As mentioned earlier, finishing and polishing procedures may result in differences in the surface roughness of dental materials, thus possibly affecting the formation and adhesion of bacterial plaques³³. Another point that should be considered with regard to wettability is the presence of fluoride filler particles. According to previous reports³⁶, the release of fluoride affects the wettability of restorative materials.

The present study involved an investigation of the influence of finishing treatments on the basic surfaces properties of several polymers commonly used in dental restorations.

2. Materials and Methods

2.1. Dental polymers

2.1.1. Sample preparation

All the samples were prepared according to the manufacturers' instructions in a 10 mm diameter, 2 mm high circular matrix (Table 1). The materials were photoactivated using a curing unit (Gnatus®; Optilux 600, SP, Brazil) at 450 mW.cm⁻². Both sides of the samples of all the materials were photoactivated for 40 seconds. The materials inserted into the circular matrix were placed on glass slides in

three increments, using a Centrix syringe. The glass ionomer cement (ChemFlex®) was proportioned (50% powder/50% liquid) and handled according to the manufacturer's instructions before it was inserted into the matrix. The photocured resins were removed after polymerization and the ionomer removed after 15 minutes.

The samples were then subjected to the following surface treatments: Half samples were finished and polished and others half not. A group was stored in deionized water for 7 days (pH 6.8) while the other group were stored dry in a plastic container at 37 °C. Dental enamel from the extracted included molars was employed as the control. A total of 70 samples were tested (Group 1 – unpolished and unfinished samples and Group 2 – finished and polished samples. Both groups were stored wet and or dry).

The surfaces of the finished and polished samples were ground with 400, 500 and 800 grit silicon carbide sandpaper (Struers A/S, Copenhagen, Denmark), water cooled, and polished with fine-grained abrasive paste (Proxy; Ivoclar/Vivadent, Liechtenstein). Before tests all the samples were washed with deionized water; half were stored wet and other half stored dry for the experiments.

2.1.2. Measurement of contact angles

The contact angles were measured on one side of the samples. Briefly, the samples were placed in a Contact Angle Goniometer (Rame-Hart Inc., Mountain Lakes NJ, USA) attached to an Image Analyzer (Rame-Hart Inc., Mountain Lakes NJ, USA) and the measurements were done with either water or glycerol. Each sample was subjected to 10 measurements in each 4-angle position: vertical left, vertical right, horizontal left and horizontal right. A total of 1,760 measurements were taken.

2.1.3. Roughness determination

The surface roughness (μ m) was determined at 10 different points on all the samples, using a Surftest 211 profilometer (Mitutoyo, Japan).

2.1.4. Fluoride release

After storage for 7 days at pH 6.8 in 5 mL deionized water, the samples were analyzed in a potentiometer (Corning Model 125/2, USA) with a specific fluoride electrode, with the solution adjusted using 2.0 mL of TISAB II (Corning, USA).

2.1.5. Statistical analysis

The contact angle and surface roughness measurement data were analyzed by ANOVA and multiple comparisons were made using the Bonferroni test ($\alpha = 0.05$).

3. Results

3.1. Surface wettability

The contact angle (θ) of the dry or wet samples of dental material formed by drops of water or glycerol were measured, as indicated in Tables 2 and 3. The mean θ values differed between wet and dry samples. Unfinished samples showed similar water and glycerol drop contact angles (Table 2; left-hand side). However, the wettability of finished samples was very dissimilar. Water drops in contact with the surfaces of finished samples of Solitaire 2 or Tetric Ceram® showed higher θ values (81.2° and 79°, respectively) than the surfaces of ChemFlex® (73.5°), TPH (71.3°) and Ariston® (59.4°) samples. The latter material displayed the lowest water drop θ (Table 2; left-hand side). Glycerol drops, in contrast, showed similar θ values varying from 82.2° (Ariston®) to 89.3° (Tetric Ceram®).

The contact angles of the wet surfaces of samples of each of the aforementioned dental materials were also evaluated with water or glycerol drops. As indicated in Table 3, the wettability data before and after finishing treatments were different. Water drops in contact with unfinished surfaces produced contact angles varying from 69.7° (Ariston®) to 88.0° (Tetric Ceram®). Similarly to dry surfaces, the finishing treatment did not alter the wettability of wet surfaces.

Table 1. Manufacturers and basic composition of the materials.

Materials	Manufacturers	Basic composition	Batch number
Solitaire 2	Hereaus-Kulzer Germany	BaAlF polyglass and porous SiO ₂ monomers	DO154020 L9
Ariston® pHc	Ivoclar/Vivadent Liechtenstein	Dimethacrylate, Ba Fluorsilicate glass, SiO ₂ , ytterbium trifluoride	A09633
Tetric Ceram®	Ivoclar/Vivadent Liechtenstein	Bis-GMA, Urethane dimethacrylate tri ethylene glycol, Al, Ba Fluorsilicate, SiO ₂	E12544
ChemFlex®	Dentsply/DeTrey Milford, DE, USA	Glass ionomer cement, Fluoride Aluminum silicate, Polyacrylic acid	9805000607
TPH	Dentsply/DeTrey Milford, DE, USA	Bis-GMA, Modified Urethane, Bore Aluminum Silicate, SiO ₂	58790

Table 2. Contact angles (degrees °) and standard deviation at the air-liquid-solid interface of dry dental materials before and after surface finishing treatment.

Materials	Fini	shed	Unfinished		
	Water	Glycerol	Water	Glycerol	
Solitaire 2	81.2 (3.0) ^h	83.9 (3.5) ^a	75.3 (1.6)°	75.8 (2.0) ^e	
Ariston®	59.4 (3.7) ⁱ	82.1 (3.5) ^a	78.0 (2.6) ^d	74.7 (3.3) ^{e,f}	
Tetric Ceram®	$79.0 (1.7)^{j}$	89.3 (3.6) ^b	75.8 (3.9) ^{c,d}	79.3 (4.7) ^g	
Chemflex®	73.4 (2.3) ^k	87.8 (3.3) ^m	70.6 (3.9) ⁿ	79.3 (3.9) ^g	
TPH	$71.3 (2.9)^{1}$	87.5 (4.7) ^b	72.6 (2.8)°	72.5 (3.9) ^f	

A statistical analysis was carried out using ANOVA and the Bonferroni test. (Same letters in a column mean non-significant differences, while different letters indicate statistical differences).

Table 3. Contact angles (degrees °) and standard deviation at the air-liquid-solid interface of wet dental materials before and after surface finishing treatment

Materials	Fini	shed	Unfinished		
	Water	Glycerol	Water	Glycerol	
Solitaire 2	64.9 (5.9) ^a	72.8 (5.5) ^f	72.0 (3.3) ⁱ	75.1 (4.3) ⁿ	
Ariston®	57.8 (3.8) ^e	68.9 (5.6) ^c	69.6 (3.9) ^j	66.9 (4.1)°	
Tetric Ceram®	79.4 (6.0) ^b	85.4 (4.8) ^g	87.9 (2.7) ^k	82.2 (3.9) ^p	
Chemflex®	67.0 (6.6) ^a	68.2 (4.4) ^c	62.1 (6.0) ¹	72.0 (5.9) ^d	
TPH	79.1 (4.8) ^b	79.2 (9.3) ^h	81.8 (5.7) ^m	70.2 (4.3) ^d	

A statistical analysis was carried out using ANOVA and the Bonferroni test. (Same letters in a column mean non-significant differences, while different letters indicate statistical differences).

The data in Table 3 (right side) indicate that the finishing treatment further changed the wettability of the samples when measurements were taken using water drops. The resulting angles varied from 57.8° (Ariston®) to 79.4° (Tetric Ceram®) and 79.2° (TPH). Table 4 shows the contact angles (degrees) and standard deviation of the dental enamel surfaces in dry and wet conditions. As can be seen, there is no significant difference between water and glycerol in dry or wet conditions ($\alpha > 0.05$).

3.2. Surface roughness

Table 5 summarizes the surface roughness measurements (μm). The Ra values of the finished surfaces were higher than those of the unfinished ones.

3.3. Fluoride release

Figure 1 shows the data on fluoride released from finished and unfinished samples. The finishing procedure affected fluoride release from Ariston samples.

4. Discussion

The contact angle measurement method is probably the most definitive way to determine the hydrophobicity of material surfaces. The angle is very high for water if the substrate is hydrophobic. When the surface is hydrophilic, the droplet quickly dissipates and the measured angle is low. Like other methods, the contact angle method gives an average value for hydrophobicity³⁷. Contact angles change with surface topography, surface tension of the liquid, surface energy of the substrate, and level of interaction between the liquid and solid^{34,38-41}.

Our results show that the materials tested here presented different surface characteristics such as composition, fillers, presence of fluoride particles, and topography. It appears that treating surfaces with polishing materials can alter their wettability. All the materials analyzed in this study showed high contact angles in the dry condition, especially the finished and polished samples, regardless of the liquid used for measuring (Table 2). The only exception was Ariston® pHc, which showed low values when measured with water (59.41°). This material tested in the unfinished condition yielded contact angle values of 78.02°, indicating that the material should not be finished.

The materials tested in the dry condition with both measurement liquids showed statistical differences (p < 0.05) except for Solitaire and Ariston® (glycerol) and Tetric Ceram® and TPH (glycerol). It is possible that materials subjected to finishing procedures show similar superficial texture and surface energy. In similar conditions but without finishing, the materials showed more molecular interactions when tested with both water and glycerol. As Table 2 indicates, all the materials are hydrophobic in finished and unfinished conditions. The results of this study are partially compatible with previous find-

Table 4. Contact angles (degrees $^{\circ}$) and standard deviation of the dental enamel surfaces.

Conditions	Liquids			
	Water	Glycerol		
Dry	83.7 (8.68) ^a	79.9 (8.49) ^a		
Wet	67.3 (9.56) ^b	68.6 (6.70) ^b		

A statistical analysis was carried out using ANOVA and the Bonferroni test. (Same letters in a column mean non-significant differences, while different letters indicate statistical differences).

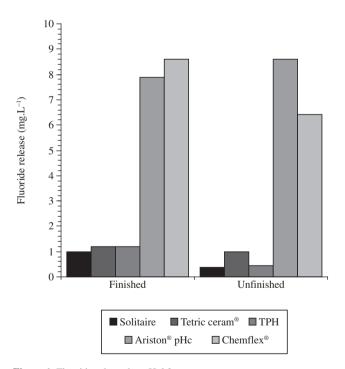


Figure 1. Fluoride released at pH 6.8.

ings⁴² which stated that contact angles on solid surfaces decrease as the surface becomes rougher.

The formation of dental biofilms depends on the characteristics of the hard surface²⁴. In the present study, we found that the dry condition and finishing procedures can also alter contact angles. The glass ionomer ChemFlex presented lower contact angles than the other materials (Table 1), indicating that this material has a tendency to absorb more saliva biofilms, which makes it more susceptible to the formation of dental plaque²⁴, regardless of its fluoride content. Roughness is prob-

Table 5. Mean values and standard deviation of surface roughness (µm).

Materials	Finished	Unfinished
Solitaire 2	0.493 (0.168) ^{d,e,f,g,h}	0.204 (0.105)a,b,c
Ariston®	$0.434 (0.083)^{g,k,n,q}$	0.184 (0.026) ^{b,m,p}
Tetric Ceram®	0.449 (0.087) ^{h,l,o,q}	0.140 (0.062) ^{c,p}
Chemflex®	0.912 (0.276) ^r	$0.645 (0.195)^{f,j}$
ТРН	$0.517 (0.123)^{d,i,j,k,l}$	0.369 (0.081) ^{a,e,i,m,n,o}

(Same letters in a column mean non-significant differences, while different letters indicate statistical differences).

ably a more significant factor than fluoride content, and the material has a tendency to be more hydrophilic than hydrophobic.

The mechanical and handling properties of dental composite resins depend on the concentration and particle size of the fillers. Proper finishing and polishing procedures are important to enhance the longevity of restored teeth. Previous studies have shown that finishing, surface roughness, surface integrity, and physicochemical properties of the restoration material can influence plaque retention^{13,15,26,48}.

The mean values of 83.77 to 79.94 measured on dry enamel using water as the liquid medium showed very similar surface hydrophobicity to that obtained with dental polymers such as Tetric Ceram® and Solitaire 2.

A comparison of the superficial roughness (μ m) of the finished and unfinished materials revealed statistically significant differences (p < 0.05) except between Ariston and Solitaire and between Ariston® and ChemFlex®. However, no difference in roughness was found between finished and unfinished TPH. The results of this study are congruous with an earlier investigation 11, which revealed significant differences in the roughness of dental materials subjected to different finishing procedures, with the lowest variability found in an ormocer material.

Several dental materials have fluoride incorporated into their matrices to improve the resistance of enamel and dentin to demineralization. However, the results have been conflicting insofar as clinical importance is concerned³⁹.

5. Conclusions

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

- 1. All the finished and polished materials showed lower roughness
- The Ariston pHc and Chemflex materials released larger amounts of fluoride than the other materials, and polishing altered their fluoride release; and
- The polishing procedure changed the contact angle of the materials tested here.

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