Article published online: 2021-05-26

Effect of Modeling Resins on Microhardness of Resin Composites

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Eur J Dent 2021;15:481–487

Abstract

Objectives This study was aimed to determine the effects of modeling resins on the surface microhardness of composites.

Materials and Methods Six resin-based composites (Charisma Smart, Estellite Asteria, CeramX-One SphereTEC, Admira Fusion, Filtek Ultimate, and Clearfil Majesty Es-2) and three wetting agents (Modeling Liquid, Composite Primer, and Modeling Resin) were investigated. In all, 240 specimens were prepared, and wetting agents were applied prior to light curing in the experimental groups. After 24 hours, specimens were polished and Vickers microhardness (VHN) values were measured.

Statistical Analysis Shapiro–Wilk and two-way analysis of variance (ANOVA) were used for analyses ($p < 0.05$).

Results Both modeling resin and composites were determined to be effective factors ($p < 0.001$). The control group showed the highest VHN (70.37 ± 7.94), followed by Modeling Liquid (64.68 ± 12.07), Composite Primer (59.84 ± 6.33), and Modeling Resin (58 ± 3.52[b]; $p < 0.001$). Filtek Ultimate showed the highest VHN (76.62 ± 9.78[c]), whereas Charisma Smart (58.87 ± 7.95), and Clearfil Majesty (67.27 ± 2.58) showed the lowest ($p < 0.001$). Clearfil Majesty–Modeling Liquid (46.62 ± 5.33) and Charisma Smart–Composite Primer (50.81 ± 0.39) combinations showed the lowest VHN, whereas Filtek Ultimate–control (87.15 ± 2.12) and Filtek Ultimate–Modeling Liquid (84.24 ± 3.11) showed the highest ($p < 0.001$).

Conclusion All tested modeling resins decreased VHN value, and the amount of reduction varied among composites and wetting agents. It might be safer not to use wetting agents unless they are necessary.

► **Keywords**:
► composite
► layering
► microhardness
► modeling resin
► wetting agent

Introduction

Since the first resin-based composite (RBC) was developed by Bowen in 1962, these materials have undergone many developments.[1] Improvements in filler technology, especially changes in related to filler size, shape, type, and silanization,[2] have enhanced the optical and mechanical properties of RBCs, as well as resistance to wear and discoloration.[3] Clinicians now have the ability to solve patients' esthetic complaints using RBCs via a minimally invasive procedure completed during a single appointment.[4] Emulating natural dental tissues with composites depends on the physical and optical properties of the composite material, restoration technique, and the clinician's experience among others.[5]

published online
May 26, 2021

DOI https://doi.org/10.1055/s-0041-1725577
ISSN 1305-7456.

Thieme Medical and Scientific Publishers Pvt. Ltd. A-12, 2nd Floor, Sector 2, Noida-201301 UP, India

Contemporary RBCs may have handling difficulties due to the stickiness of the material to dental instruments. Their viscous monomer content may be responsible for this.[6] Manufacturers have provided various restorative instruments to overcome the stickiness problem, including titanium/aluminum coated instruments, ultrasonic instruments, rubber tips, and composite brushes. In addition, some clinicians have used adhesive agents to avoid the stickiness of composite material. However, low-viscosity materials, such as resin adhesives, acetone, and isopropyl alcohol, are not intended for this purpose.[7-9] Nevertheless, no adverse effects of alcohol or adhesive system application on composite interfaces have been reported.[10] However, an adhesive agent used at the top restoration layer may have some risks as it will be exposed to the oral environment during drinking and eating. Leaving the adhesive agent as the final layer may negatively affect the optical properties of the composite material, as well as the color stability, depending on the type and composition of adhesive agent used. Hydrophilic materials show more unfavorable effects on color stability.[11]

Most recently, a few manufacturers have introduced wetting (modeling) agents, relatively friendly materials in terms of physical properties, for better manipulation. Some modeling agents are also resin-based materials that include few or no fillers.[2] For application, only a small drop of wetting resin is dropped on a pad, and a spatula or brush tip is slightly touched with the resin to moisturize the tip. Modeling agents should not be used to lower the viscosity of the composite material. Excessive resin on the tip should be removed using a paper tissue; otherwise, the over-wet instrument could change the physical and chemical properties of the composite material. The process may be repeated if moisturizing is needed again. These low-viscosity agents tend to improve the handling properties of composite resins by reducing the surface tension.[12] Modeling resins also fill and cover defects on the restoration by diffusing through any pores created during the layering procedure.[8,11,13] The use of wetting agents inhibits the stickiness of RBC materials and provides better manipulation. Modeling agents have been used in dental clinics, particularly for restorative treatments; however, there is a lack of scientific evidence regarding the effects on the properties of composite materials.[2,3,5,7]

For microhardness measurements, the Vickers microhardness (VHN) test is the most appropriate to evaluate the mechanical properties of resin composites in terms of reliability and accuracy.[14] Materials with lower hardness are susceptible to surface defects and fractures.[15] In contrast, those with high microhardness show increased wear resistance, that is, there is a relation between materials' mechanical properties and clinical longevity. To obtain long-lasting esthetic restorations, the physical characteristics of the RBCs, such as cross-link density or degree of conversion, should be maintained.[16] There is a positive correlation between the degree of conversion and surface hardness.[17] In terms of resisting masticatory forces, microhardness is one of the most important mechanical properties of the RBC material.[18]

This *in vitro* study evaluated the effects of modeling resin application on VHN values of RBC materials. The tested null (h_0) hypothesis was that modeling agents would have no effect on the surface microhardness of resin composites.

Materials and Methods

Enamel or shades of six different composite materials and three different modeling agents were evaluated in this *in vitro* study. The RBCs included two nanohybrid (Charisma Smart/CS, Kulzer, Hanau, Germany; Clearfil Majesty Es-2/CM, Kuraray, Noritake, Japan), one nanofilled (Filtek Ultimate/FU, 3M, St. Paul, Minnesota, United States), one supraspherical nanofilled (Estellite Asteria/EA, Tokuyama Dental, Tokyo, Japan), one spherical nanohybrid (CeramX One SphereTEC/CO, Dentsply Sirona, Konstanz, Germany), and one ormocer (Admira Fusion/AF (VOCO GmbH, Cuxhaven, Germany) materials. The wetting agents included Modeling Liquid (GC Corp., Tokyo, Japan), Composite Primer (GC Corp.), and Modeling Resin (KavoKerr, Orange, California, United States). The composition, type, and manufacturers of the tested resin-based materials are listed in ►**Table 1**. Ten samples for each modeling agent group and control group (thus, a total of 240 specimens) were prepared.

Preparation of Specimens

All specimens were prepared using silicone molds (10 mm in diameter and 2 mm in height), using Mylar matrix strips covered with glass slides on both sides. In the experimental groups, modeling agents were applied to the specimen's surface using a composite brush (Composite Brush, GC Corp., Tokyo, Japan). The brush was always slightly moisturized with wetting resin, and excessive material was removed using a clean paper tissue. Modeling agents were not used for the control group. Polymerization was performed using an LED curing unit (Elipar Deepcure S, 3M, St. Paul, Minnesota, United States) with exposure to a light intensity of 1,370 mW/cm^2 for 20 seconds from both sides. The polishing procedure was performed using abrasive discs embedded with aluminum oxide (Al_2O_3; Sof-Lex, 3M, St. Paul, Minnesota, United States). Coarse (100-µm Al_2O_3 particles), medium (40-µm Al_2O_3 particles), fine (24-µm Al_2O_3 particles), and super-fine (8-µm Al_2O_3 particles) discs were used. Polishing speed was set at approximately 20,000 rpm under continuous water cooling. The polishing process was performed according to the manufacturer's instructions (20 seconds application per disc, under slight hand pressure). The polishing discs were renewed for each specimen.

Microhardness (Vickers Microhardness) Test

Prior to the measurements, all specimens were immersed in distilled water for 24 hours. VHN was measured using a microhardness tester (Wilson Wolpert Micro-Vickers 401MVD, Wilson Wolpert Instruments, Aachen, Germany) with a dwell time of 10 seconds under a 200-g (1.96 Newton) load. Each specimen was placed on the VHN tester, and three diamond–pyramid-pattern indentations were formed. The VHN was measured using the length of the indentations and the formula $H = 1.854\ P/d^2$ (P: load and d: diagonal length). The mean value was calculated using the measurements of the three indentations and recorded as VHN value.

Intraobserver and interobserver correlation coefficient values were 0.951 to 0.982 and 0.950 to 0.982, respectively; both confidence intervals were 95% ($p < 0.001$).

Statistical Analysis

The data were analyzed using IBM SPSS, version 23 (SPSS, Chicago, Illinois, United States). The normality of the data distribution was examined using the Shapiro–Wilk test. A univariate method was used to examine the effects of modeling agent and composite interactions and effects on VHN values. Comparisons of the mean values were carried out using a two-way analysis of variance (ANOVA) test, and the significance level was set at $p < 0.05$.

Results

Wetting resin and composite both influenced surface microhardness ($p < 0.001$; ►**Table 2**). Composite factor (F: 216.188) was slightly more effective than wetting resin (F: 210.373), and the wetting resin + composite combination was less effective ($p < 0.001$; F: 30.949).

Regarding the modeling agents, a statistically significant difference in mean VHN measurements (kg/mm^2) was observed between the control group and experimental groups (►**Table 3**). The control group had the highest VHN value (70.37 ± 7.94^{a}); $p < 0.001$), followed by Modeling Liquid (64.68 ± 12.07^{d}). Modeling Resin had the lowest VHN value (58 ± 3.52^{b}; $p < 0.001$; ►**Fig. 1**).

Among the composites, the FU composite had the highest VHN value (76.62 ± 9.78^{c}) which was statistically significant ($p < 0.001$). There were no significant differences in VHN values between the EA, AF, and CO composites ($p \geq 0.05$). The CS and CM composites had the lowest VHN values ($p < 0.001$; ►**Table 3**; ►**Fig. 1**).

Regarding the interactions between composites and modeling agents, the CM–Modeling Liquid (46.62 ± 5.33^{A}) and CS–Composite Primer (50.81 ± 0.39^{AB}) combinations had the lowest VHN values ($p < 0.001$). The FU–control group (87.15 ± 2.12^{L}) and FU–Modeling Liquid (84.24 ± 3.11^{L})

Table 1 Compositions, type and manufacturers of the resin-based materials tested in this study

Code	Brand name	Filler type	Compositions	Manufacturer
CS	Charisma Smart	Nanohybrid	Matrix: Bis-EMA, HEDMA, TEGDMA	Kulzer, Hanau, Germany
			Filler: barium aluminum fluoride glass filler of 0.02-2 µm, 5 vol% pyrogenic silicon dioxide filler of 0.02–0.07 µm. 78 wt%, 65 vol%	
EA	Estellite Asteria	Supra nanofilled spherical	Matrix: Bis-GMA, Bis-MPEPP, TEGDMA, UDMA	Tokuyama Dental, Tokyo, Japan
			Filler: uniform supranano spherical silica and zirconia fillers (200 nm). 82 wt%, 71 vol%	
CO	Ceram-X One SphereTEC	Nanohybrid ceramic spherical	Matrix: polyurethanemethacrylate, Bis-EMA, TEGDMA	Dentsply Sirona, Konstanz, Germany
			Filler: prepolymerized spherical fillers (15 µm) and 0.6 µm barium glass fillers and 0.6 µm ytterbium fluoride, silicon dioxide nanofillers (10 nm). 77–79 wt% and 59–61 vol%	
AF	Admira Fusion	Ormocer	Matrix: resin ormocer	Voco, Cuxhaven, Germany
			Filler: glass ceramics filler, silicon oxide nano filler, (1 µm) 84 wt%, 69vol%	
FU	Filtek Ultimate	Nano-filled	Matrix: Bis-GMA, UDMA, TEGDMA, Bis-EMA, PEGDMA	3M, St. Paul, Minnesota, United States
			Filler: silica filler (20 nm), zirconia filler (4–11 nm), zirconia/silica cluster filler, 0.6–10 µm particle size. 78.5 wt%, 63.3 vol%	
CM	Clearfil Majesty ES-2	Nanohybrid	Matrix: Bis-GMA, hydrophobic aromatic DMA, and hydrophobic aliphatic DMA, dl-camphorquinone	Kuraray, Noritake, Japan
			Filler: silanated barium glass (particle size 0.37–1.5 µm) and prepolymerized organic filler. 78 wt%, 40 vol%	
	Modeling Liquid	–	UDMA, 2-hydroxyethyl methacrylate,3 dimethacryloxy propane, 2-hydroxy-1	GC Corp., Tokyo, Japan
	Composite Primer	–	HEMA, UDMA, tetrahydrofurfuryl methacrylate	GC Corp., Tokyo, Japan
	Modeling Resin	–	Poly(oxy-1,2-ethanediyl), α,α'-[(1- methylethylidene) di-4,1-phenylene]bis[ω-[(2- methyl-1-oxo-2-propen-1-yl) oxy]-, oxybenzone, 3-trimethoxysilylpropyl methacrylate 1,1'-azobis(1-cyclohexanecarbonitrile)	KavoKerr, Orange, California United States

Abbreviations: MDP: methacryloyloxydecyl dihydrogen phosphate; 4-MET: methacryloyloxyethyl trimellitic acid; MDTP: thiophosphate ester monomer; Bis-GMA: bisphenol A diglycidil ether dimethacrylate; UDMA: diurethane dimethacrylate; TEGDMA: triethylene glycol dimethacrylate; phA-m: phosphoric acid ester monomer.
Note: The data regarding the compositions of resin composites were obtained from the manufacturers of these composites.

Table 2 Two-way ANOVA comparison for mean VHN values in terms of wetting resin and composite factors

	Sum of values	SD	Mean values	F	*p*-Value
Wetting resin	5,518.857	3	1,839.619	210.373	<0.001
Composite	9,452.367	5	1,890.473	216.188	<0.001
Wetting resin × composite	4,059.545	15	270.636	30.949	<0.001

Abbreviations: ANOVA, analysis of variance; F, test statistics; SD, standard deviation; VHN, Vickers microhardness.

Table 3 Descriptive statistics in terms of mean VHN values according to the composite materials and wetting resins

Composite	Modelling resins				Total
	Control group	Modeling resin	Composite Primer	Modeling Liquid	
EA	66.77 ± 1.6^{IJK}	57.26 ± 1.66^{CDE}	56.66 ± 3.16^{CDE}	64.79 ± 5.09^{GHIJ}	61.37 ± 5.48^{b}
AF	65.24 ± 1.67^{HIJ}	59.05 ± 1.39^{CDE}	59.74 ± 1.07^{DEF}	66.71 ± 4.52^{IJK}	62.69 ± 4.18^{b}
FU	87.15 ± 2.12^{L}	64.05 ± 1.43^{FGHI}	71.05 ± 0.75^{K}	84.24 ± 3.11^{L}	76.62 ± 9.78^{c}
CO	66.25 ± 2.99^{IJK}	57.71 ± 1.38^{CDE}	60.55 ± 1.91^{DEFGH}	64.79 ± 4.81^{GHIJ}	62.33 ± 4.52^{b}
CS	69.55 ± 0.87^{JK}	54.19 ± 2.41^{BC}	50.81 ± 0.39^{AB}	60.91 ± 6.24^{EFGH}	58.87 ± 7.95^{a}
CM	67.27 ± 2.58^{IJK}	55.73 ± 1.5^{CD}	60.21 ± 2.22^{DEFG}	46.62 ± 5.33^{A}	57.46 ± 8.2^{a}
Total	70.37 ± 7.94^{a}	58 ± 3.52^{b}	59.84 ± 6.33^{c}	64.68 ± 12.07^{d}	63.22 ± 9.36

Abbreviations: AF, Admira Fusion; CM, Clearfil Majesty Es-2; CO, CeramX One SphereTEC; CS, Charisma Smart; EA, Estellite Asteria; FU, Filtek Ultimate.
$^{a–d}$No significant difference between wetting resins or composites with same letter.
$^{A–L}$No significant difference between wetting resin × composite interactions with same letter.

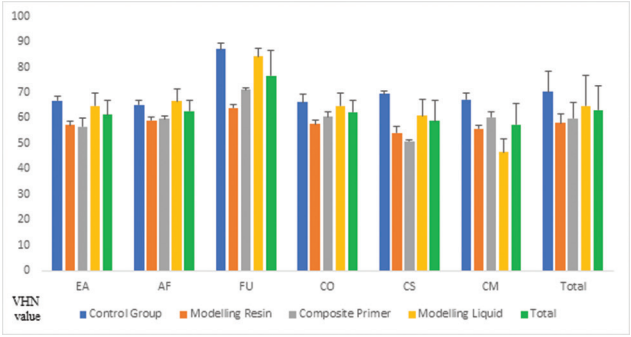

Fig. 1 Mean and standard deviation of VHN values, regarding modelling resins and composites. VHN, Vickers microhardness.

combinations had the highest values ($p < 0.001$; ►**Table 3**; ►**Fig. 1**).

Discussion

The null (h_0) hypothesis was rejected, as all of the evaluated wetting agents adversely affected the surface microhardness of the resin composites.

The increasing esthetic demands of patients have led clinicians to provide life-like restorations, emulating the natural tooth. Wetting agents may improve composite manipulation, particularly for the additive layering technique, involving the incremental addition of small composite pieces, instead the subtractive technique. The use of modeling resin with a composite brush may also help clinicians sculpt resin composite between incremental layers and support the construction of a smooth and structured final composite layer.[3] The present study is one of a few that have focused on the effects of modeling agents on the mechanical properties of composites, when used on the top layer.

The final layer of a restoration procedure has a determinative effect on the esthetic appearance, color efficacy, and surface roughness.[19] Well-polished and smooth restoration surfaces reduce plaque accumulation and accordingly decrease the risk for secondary caries and discoloration. Proper finishing and polishing procedures should be maintained to achieve long-lasting clinical restorations.[20] Contemporary RBCs have superior polishability when polished with compounds containing embedded Al_2O_3 or diamond particles.[21] In our study, specimens were polished using Al_2O_3-embedded Sof-Lex discs to obtain a standard and optimum surface smoothness. Following the polishing procedure, the specimens were immersed in distilled water for 24 hours to eliminate unreacted units of resin and to allow postpolymerization.[22]

The surface hardness of a material is a key parameter influencing its mechanical properties.[23] Surface hardness is directly related to wear[24] and is related to surface roughness, where softer materials tend to have rougher surfaces. This in turn may lead to susceptibility to discoloration, secondary caries, plaque accumulation, and gingival irritation.[25] The VHN test is often used for the quantitative measurement of microhardness.[26,27] In this study, the specimens were tested using 200-g force with a dwell time of 10 seconds, according to International Organization for Standardization (ISO) 6507/ASTM E 384 standards.[28] Microhardness may be affected by chemical characteristics, as well as filler type, shape, and size.[29] Filler content influences the optical and mechanical properties of a material, affecting the color stability, wear resistance, stiffness, compressive strength, and surface hardness.[30] A positive correlation has also been reported between filler content and surface hardness.[31]

Evaluation of the Modeling Agents

In this study, both modeling resin and composite influenced surface microhardness. Composite had the greatest effect, followed by modeling resin and the Modeling Resin + Composite combination (►**Table 2**).

All of the modeling agents caused a decrease in surface hardness, and the amount of reduction varied among the materials, in line with one previous study[2] but contradicting another.[7] The control group had the highest hardness, followed by Modeling Liquid, Composite Primer, and Modeling Resin, and the differences among the options were significant (►**Table 3**). The fact that the highest VHN value was recorded for the control group might be related to the filler content of the final composite layer.[2] As modeling agents make up a lower percentage of fillers, the application of the agents to the surface might have created a resin-rich surface layer. Consequently, the filler content of the final composite layer might be directly related to the VHN value.[26] In our study, all of the specimens were polished after applying modeling agent. Although the resin-rich surface layer was expected to be removed by the polishing procedure, it may be that the wetting agents diffused to deeper layers of the RBCs.[32] Tuncer et al[2] evaluated the effects of a modeling agent (Modeling Resin, Bisco, Illinois, United States) on surface microhardness of different RBCs, and the hardness decreased due to the use of modeling agent for GrandioSO (Voco, Cuxhaven, Germany) and Gradia Direct Posterior (GC, Corp.). This was associated with the high level of resin-rich surface layer. Our study supports this result, as all of the tested modeling agents lowered the surface hardness of the composites. However, regarding our results, Modeling Liquid might be considered the most reliable modeling agent, as significantly higher VHN values were observed compared to the other wetting agents ($p < 0.001$).

The Modeling Resin group had the lowest hardness (58 ± 3.52^{b}) among all groups. This might be related to the fact that it lacks the diurethane dimethacrylate (UDMA) molecule, which consists of two urethane links and a flexible aliphatic core, and forms double-hydrogen bonds.[33] UDMA-containing resins have been reported to have superior polymerization rates and a high degree of conversion.[34] Accordingly, the degree of conversion and polymerization rate might have affected the surface hardness of the specimens in the Modeling Resin group, resulting in the lowest values.[2,17] However, Tuncer et al[2] stated that differences in hardness among different composites might not be attributable to the degree of conversion at all.

The Composite Primer group had lower hardness than the Modeling Liquid and control groups (►**Fig. 1**). This might be explained by the 2-hydroxyethyl methacrylate content of the Composite Primer agent. HEMA is a hydrophilic monomer which causes water absorption due to a hydroxyl group and carbonyl group.[35] Hence, HEMA including Modeling Liquid might have reduced the surface hardness of the RBCs.[36] Kutuk et al[7] used Modeling Liquid and two universal adhesive agents (G-Premio Bond, GC Corp.; OptiBond XTR, KavoKerr, Orange, California, United States) as modeling agents in combination with a nanohybrid RBC (Essentia, DE shade, GC Corp.). The lowest microhardness was observed for OptiBond XTR. The Modeling Liquid group maintained the surface roughness better than universal adhesives and showed the lowest degree of color change. Both Modeling Liquid and universal adhesive agents were suggested useful for composite stratification; however, Modeling Liquid was mentioned as the most appropriate material due to high mechanical properties and stability.[7]

Evaluation of the Resin Composites

The hardness of RBCs is also affected by filler characteristics,[37] and there are strong interactions between polymers and nanoparticles.[38] Nanofilled resin composites show improved hardness, improved abrasion resistance, high gloss retention, and superior polishing ability.[39] In our study, the nanofilled resin composite FU had the highest VHN value (►**Fig. 1**). In addition, there were no significant differences in VHN values between the EA, AF, and CO composites. Al_2O_3, barium glass, and ZrO_2 filler particles have natural properties that increase the hardness of resin composites via intense ionic interatomic bonds.[40] Similar physical properties obtained with AF (ormocer) and CO (nanoceramic) which are ceramic-based resin composites, and EA (nanozirconia), a zirconium-based resin composite, might be related to the filler types of these RBCs. The lowest VHN values were obtained with CS and CM composites (►**Fig. 1**). The CM and CS composites contain prepolymerized fillers, which may cause weak cross-linking between the polymer matrix and the fillers.[30]

Regarding the interactions between resin composites and wetting resins, the CM–Modeling Liquid (46.62 ± 5.33^{A}) and CS–Composite Primer (50.81 ± 0.39^{AB}) combinations had the lowest VHN values. The FU–control group (87.15 ± 2.12^{L}) and FU–Modeling Liquid (84.24 ± 3.11^{L}) combinations had the highest VHN values (►**Fig. 1**). De Paula et al[3] evaluated the effects of wetting agents on the cross-link density and degree of conversion of resin-based nanocomposites (Filtek Z350 XT, 3M; IPS Empress Direct, Ivoclar Vivadent, Schaan, Liechtenstein). Two adhesive systems (Adper Single Bond 2, 3M; Scotchbond Multi-Purpose bonding agent, 3M) and ethanol (70% and absolute ethanol) were used as wetting agents. They reported that the degree of conversion decreased when using 70% ethanol and adhesive systems combined with IPS Empress Direct. Filtek Z350 XT showed similar results for degree of conversion regardless of the modeling agents used. They stated that the reduction in cross-linking density might lead to a decrease in a material's mechanical properties. The cross-link density of IPS Empress Direct decreased for both adhesive systems, while that of Filtek Z350 XT decreased for only Scotchbond Multi-Purpose bonding agent. Absolute ethanol may protect the surface structure more safely compared to 70% ethanol and an adhesive system.[9] Thus, as suggested by Kutuk et al,[7] it may be necessary to avoid using adhesive agents as modeling agents to maintain the mechanical properties and stability of resin composites. In our study, Modeling Liquid had significantly higher surface hardness

than controls. However, the difference was present only for the CS and CM composite interactions; these two composites also had the lowest surface hardness. Regarding the EA, AF, FU, and CO composite interactions, there were no significant differences between Modeling Liquid and the control group (►**Fig. 1**). Therefore, with regard to EA, AF, FU, and CO composite interactions, Modeling Liquid application might be safer than not using a wetting agent at all. In addition, FU composite might be the most reliable RBC, with the FU–Modeling Liquid combination being the most reliable composite–modeling agent combination, in terms of surface microhardness.

Conclusion

Within the limitations of this *in vitro* study, all of the evaluated modeling agents resulted in a reduction in surface microhardness of RBC materials, and the degree of reduction varied among the brands of modeling agents and resin composites. Modeling Liquid might be considered as a safer wetting agent in terms of surface microhardness; however, the safest approach is not to use wetting agents at all. Further longitudinal clinical trials should be undertaken to precisely understand the effects of modeling agents.

Funding
None.

Conflict of Interest
None declared.

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