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STUDY REGARDING THE INFLUENCE OF VARIOUS MODELING AGENTS ON SURFACE MICROHARDNESS AND ON SURFACE ROUGHNESS OF NANOHYBRID COMPOSITE RESINS

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ABSTRACT

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Introduction The purpose of this study was to investigate the effects of different modeling agents on surface hardness and on surface roughness of some conventional nanohybrid composite resins.
Methodology Samples of two nanohybrid composite resins: Essentia - group I (n=40) and Neo Spectra ST HV - group II (n=40) were included in this study. Three modeling agents were applied on top of the last composite layer before light curing: Modeling Liquid - subgroup 2 (n=10), 7th generation of bonding system-G-Bond - subgroup 3 (n=10), and a universal bonding system G-Premio Bond - subgroup 4 (n=10). In subgroup 1 (n=10) no modeling agent was applied. Half of the samples in subgroups 1, 2, 3 and 4 from each group were subjected to surface hardness determination using a digital electronic hardness tester (Vickers Hardness Number (VHN) mean value was reported) and half of them to surface roughness evaluation by Atomic Force Microscopy (AFM) analysis (AFM analysis) (root mean square parameter (Rq) was reported).
Results In group I and II statistically significant results were obtained when comparing the surface microhardness in subgroups 2, 3 and 4 with subgroup 1, the microhardness in subgroups 2 and 3 and in subgroup 2 and 4 (Wilcoxon test, $p<0.05$).In both groups, no statistically significant differences were obtained when comparing the mean Rq values among all subgroups (ANOVA and post hoc Bonferroni tests,$p<0.05$).
Conclusion All evaluated modeling agents decreased the surface microhardness of the tested nanohybrid composite resins. None of the modeling agents influenced the surface roughness of the composites.

KEYWORDS

Atomic Force Microscopy (AFM), Composite Resins, Microhardness, Modeling Agents, Roughness.

1. INTRODUCTION

Due to technological progress in material science composite resins have become the most commonly used direct restorative materials both on anterior and posterior area of the arches [1].

The main advantages are represented by their use in minimally invasive techniques, esthetic aspect, good mechanical properties, good handling properties

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(some composite resins having easy transportation, insertion and modeling characteristics) [2]. Rebuilding tooth anatomy is a mandatory step when restoring a tooth and due to the viscosity of resin monomers sometimes it is difficult to shape the composite in order to fit the natural anatomical aspect of the tooth.To prevent the adhesiveness of the composite to the instruments used for transportation, insertion or modeling, different resin monomers or substances were used to lubricate the tools or the brushes. In time practitioners started to use alcohol, acetone and isopropyl acid to control the handling and modeling characteristics of the composites, but they were considered inappropriate for the purpose. Alcohol used as a modeling agent can have detrimental effects on the resin matrix and can decrease the mechanical properties of composites [3]. Some producers introduced wetting agents (modeling liquids) for better handling. The lubricants can be applied in the layering process of composite application to minimize adhesiveness by wiping the instrument with modeling agents [4,5]. This approach facilitated the improvement of handling and insertion, but also simplified the modeling process of composite resins and improved the surface characteristics by smoothening the surface [6]. Most of the modeling agents are resin-based materials that include little or no filler [7]. Modeling liquids generally contain methacrylates such as urethane dimethacrylate (UDMA), bisphenol A-glycidyl methacrylate (Bis-GMA), and triethylene glycol dimethacrylate (TEGDMA). They are also composed of hydrophobic non-solvated resins and they have low or no organic fillers [8]. Chemical, structural and mechanical alterations have been reported in composite materials after being modeled with the instrument lubricated with modeling agent even when the chemical composition of the agent was similar to that of the composite resin [9].

As an alternative to modeling liquids dental clinicians have used bonding systems to improve composite handling properties, even if this use is not included in the manufacturers' specifications.Some studies have pointed that these techniques can negatively affect the physical properties and surface characteristics of composite resins [10-12]. Significantly higher decrease of composite surface micro-hardness was reported when a non-solvated adhesive (the 3rd step of etch and rinse bonding system) [10] or the self-etch primer (the 1st liquid of the 2-step self-etch bonding system) [11] were used as lubricants. On the contrary, other studies concluded that some modeling agents can preserve the surface hardness [13]. Only a few articles reported data regarding the influence of modeling agents on composite surface roughness and these data are controversial. Some of the studies pointed that the modeling liquid, the universal bonding agent or the 2nd step of a self-etch bonding system have no effect on the surface roughness of the investigated composite resins [10, 11]. On the other hand, in other studies the application of a modeling liquid determined an increased surface roughness of the composite resins [13]. The purpose of this study was to investigate the effects of various agents (modeling substances or adhesive systems) on the surface hardness and surface roughness of some conventional nanohybrid composite resins. The null hypotheses were: 1. the use of different modeling agents has no effect on the surface microhardness of nanohybrid composite resins; 2. the use of different modeling agents has no effect on the surface roughness of nanohybrid composite resins.

2. MATERIALS AND METHODS

Study design is presented in Figure 1.

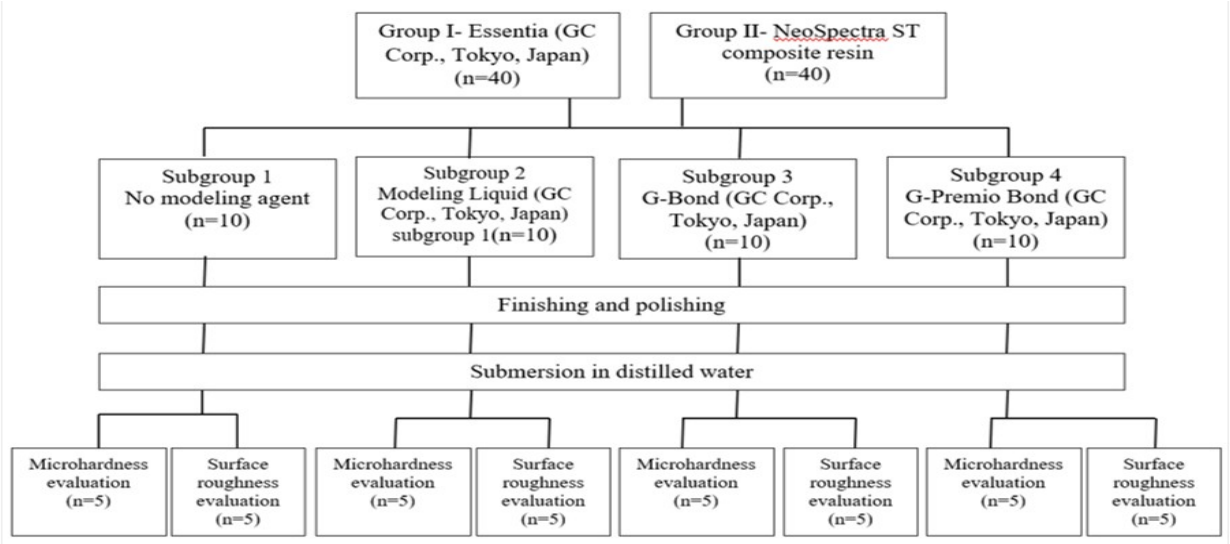

Figure 1. Study design

2.1. Sample preparation

Two nanohybrid composite resins were included in this study: Essentia - group I (GC Corp., Tokyo, Japan) (light enamel shade) and Neo Spectra ST HV- group II (Dentsly Sirona, Konstanz, Germany) (A1 shade).

Forty samples of each material were obtained by condensing the resin into the plastic cylinders 6 mm in diameter and 4 mm in height. The molds were placed on a glass plate in contact with a transparent matrix to ensure a smooth surface of the sample.

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Two layers of each material were inserted, each layer being individually cured for 40 seconds using a LED lamp (Woodpecker Med. Instrument, Guilin, China) with the intensity of 1.200 mW/cm2.

Before light-curing the last layer, three different modeling agents were applied on the surface of 30 samples from each group using a brush. Modeling Liquid (GC Corp., Tokyo, Japan) was applied on 10 samples from each group (subgroup 2), G-Bond bonding system (GC Corp., Tokyo, Japan) on 10 samples (subgroup 3) and universal bonding system G-Premio Bond (GC Corp., Tokyo, Japan) were applied in self-etch technique on 10 samples (subgroup 4). Different brushes were used for each modeling agent and for a specific type of agent the brush was replaced by a new one after 10 applications. The same quantity of the liquid (one drop) was placed in a plastic box, the brush was submersed one time in the liquid and the excess was removed by touching a paper towel with the brush. For the rest of 10 samples in each group no modeling agent or bonding system was applied before light-curing the last layer of composite resin (subgroup 1). Details related to the chemical composition of the two composite resins and modeling agents are presented in Table 1.

Table 1. Chemical composition of the tested materials

	Material type	Composition	The manufacturing company
Essentia	Nanohybrid composite resin	Matrix: UDMA, Bis-MEPP, Bis-EMA, Bis-GMA, TEGDMA Filler: prepolymerized silicon particles, barium glass (81% by weight and 65% by volume)	GC Corp., Tokyo, Japan
Neo Spectra ST HV	Nanohybrid composite resin	Matrix: Methacrylate modified polysiloxane (organically modified ceramic), Bis-EMA, TEGDMA Filler: prepolymerized spherical particles (15 µm) and 0.6 µm barium glass and 0.6 µm ytterbium fluoride particles, silicon dioxide nanoparticles (10 nm), (77–79% by weight and 59–61% by volume)	Dentsly Sirona, Konstanz, Germany
Modeling Liquid	Modeling agent	UDMA, 2-HEMA, TEGDME	GC Corp., Tokyo, Japan
G-Bond	adhesive system (7th generation)	Phosphoric acid ester monomers, 4-MET monomer, nanoparticles	GC Corp., Tokyo, Japan
G-Premio Bond	Universal adhesive system	4-MET, MDP and MDTP	GC Corp., Tokyo, Japan

UDMA- urethane dimethacrylate; Bis-MEPP- bisphenol-A ethoxylate dimethacrylate; Bis-EMA- ethoxylated bisphenol-A dimethacrylate; Bis-GMA-bisphenolglycidyl methacrylate; TEGDMA- triethylene glycol dimethacrylate; UDMA- urethane dimethacrylate; 2-HEMA- 2 hydroxyethyl methacrylate, TEGDME- trimethylene glycol dimethyl ether; 4-MET- 4-[2-(methacryloyloxy)ethoxycarbonyl] phathalicacid; MDP-10 Methacryloyloxydecyl dihydrogen phosphate; MDTP- 4,4′,4″- [(methanetriyltris (benzene-4,1-diyl)) tris (oxy)] triphthalonitrile

The samples were then removed from the plastic mold, the lower surfaces were marked and the upper surfaces of the samples were finished with medium, fine and extra fine abrasive discs (Sof-LexTM, 3M ESPE) under water cooling, for 20 seconds for each grit. The samples were then submersed for 24 hours in a container with distilled water. Half of the samples in subgroups 1, 2, 3 and 4 from each group were subsequently subjected to surface hardness determination and half of them to surface roughness evaluation.

2.2. Determination of surface microhardness

On the unmarked surfaces of the samples Vickers hardness was determined using a digital electronic hardness tester (Micro-Vickers Hardness System CV-400 DMTM, CV Instruments Namicon). CV-400 mico/macrohardness tester is a solid and accurate hardness tester used on an industrial and laboratory scale. It is equipped with an automatic Vickers indentation head and a special indentation measurement and evaluation software. In this study a load of 200 g with a 10-second dwell time was applied on Vickers hardness head, according to the International Organization for Standardization (ISO) 6507/ASTM E 384 standards. For each sample 2 indentations were made, the distance between the indentations being of 1 mm. The surface hardness was determined by measuring the indentation diagonal and was expressed as Vickers Hardness Number (VHN). The final surface hardness of a sample was calculated as the average value of the two determinations.

2.3. Determination of surface microroughness

Half of the samples in each group were analyzed for surface roughness using atomic force microscopy (SOLVER PRO-M scanning probe microscope, NTMDT, Russia). The measurements were performed in air environment and in static force operating mode. 2D and 3D images were obtained on sample area of 20 × 20 µm. On 3D images the surface roughness was reported as the root mean square roughness parameter (Rq). Two hundred fifty-six linear scans were performed on each section and the final Rq value of the sample was reported as the mean value of all scans.

2.4. Statistical analyses

The data were analyzed using IBM SPSS Statistics 28.0.1 program (SPSS Inc., Chicago, IL, USA). The effects of modeling agents on hardness were analyzed using the Wilcoxon test (at $p<0.05$ significance level) and the effects on surface roughness using ANOVA and post hoc Bonferroni tests (at $p<0.05$ significance level).

3. RESULTS

3.1. Surface hardness evaluation

The mean values and standard deviation of surface microhardness (VHN) in groups and subgroups are presented in Table 2.

Table 2. Mean VHN values and standard deviations of surface microhardness (VHN) in groups and subgroups

	Subgroup 1	Subgroup 2	Subgroup 3	Subgroup 4
Group I	68.04 ± 0.45	63.58 ± 0.77	54.25 ± 1.34*	56.02 ± 0.48*
Group II	66.05 ± 0.67	58.23 ± 0.70	44.93 ± 0.99*	46.05 ± 0.60*

* represent no statistical differences among the subgroups in group ($p>0.05$)

In group I and II statistically significant results were obtained when comparing the surface microhardness values of the samples in subgroups 2, 3 and 4 with subgroup 1 of the samples in subgroups 2 and subgroup 3 and of subgroup 2 with subgroup 4 (Table 2).

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3.2. Surface roughness evaluation

3D and 2D aspects of some samples in group I subgroups 1-4 and group II subgroups 1-4 are presented in figure 2 and figure 3, respectively.

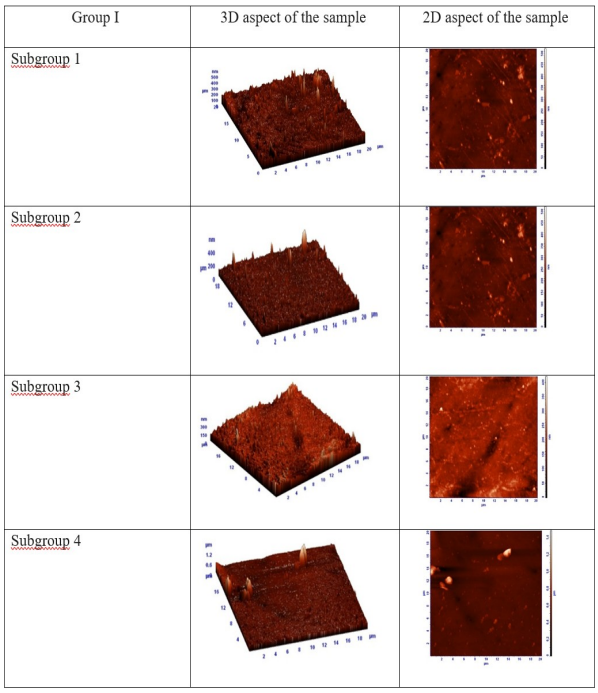

Figure 2. 3D and 2D aspect of Essentia samples when using no modeling agent (subgroup 1), Modeling Liquid (subgroup 2), G-Bond (subgroup 3), G-Premio Bond (subgroup 3).

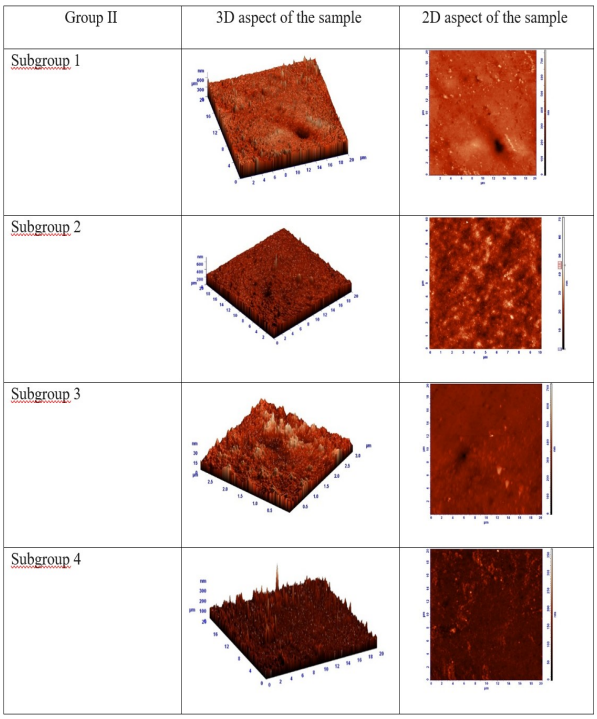

Figure 3. 3D and 2D aspect of NeoSpectra ST samples when using no modeling agent (subgroup 1), Modeling Liquid (subgroup 2), G-Bond (subgroup 3), G-Premio Bond (subgroup 3)

The mean Rq values and standard deviation in subgroups 1-4 of groups I and II are presented in table 3. In both groups, no statistically significant differences were obtained when comparing the surface roughness among subgroups 1, 2, 3 and 4.

Table 3. Mean Rq values and standard deviation in subgroups 1-4 of groups I and II

	Subgroup 1	Subgroup 2	Subgroup 3	Subgroup 4
Group I	0.051±0.003	0.067±0.002	0.055±0.003	0.064±0.003
Group II	0.052±0.002	0.064±0.003	0.057±0.004	0.065±0.002

4. DISCUSSION

The first null hypothesis of the study was rejected, all the agents used for modeling the composite resin decreasing the composite surface hardness. This might be determined by the filler content in the final composite layer after using modeling agents [7]. All modeling agents have a low filler percentage, so their application on the last composite layer lead to a resin-rich layer formation on the surface [14]. As a result, lower VHN values are obtained after modeling agent application. Generally, this extern layer having high resin content is removed by the finishing procedure. Although all the samples in this study were finished, VHN values were still lower in the groups where modeling agents were used when comparing to the control group (table 2). Even if the external resin-reach layer was removed by finishing, it seems that wetting agents can diffuse in the deeper layers of the material, changing their chemical composition and hardness [15]. Another explanation for decreasing the surface hardness as a result of modeling agents application is the presence of 2-HEMA molecule in the composition of the Modeling Liquid, a hydrophilic monomer which can cause water absorption due to a hydroxyl and carbonyl group [16]. Therefore, as it was reported even in previous studies, HEMA can reduce the hardness of composite resin [17]. The first null hypothesis of the study was rejected, all the agents used for modeling the composite resin decreasing the composite surface hardness. This might be determined by the filler content in the final composite layer after using modeling agents [7]. All modeling agents have a low filler percentage, so their application on the last composite layer lead to a resin-rich layer formation on the surface [14]. As a result, lower VHN values are obtained after modeling agent application. Generally, this extern layer having high resin content is removed by the finishing procedure. Although all the samples in this study were finished, VHN values were still lower in the groups where modeling agents were used when comparing to the control group (table 2). Even if the external resin-reach layer was removed by finishing, it seems that wetting agents can diffuse in the deeper layers of the material, changing their chemical composition and hardness [15]. Another explanation for decreasing the surface hardness as a result of modeling agents application is the presence of 2-HEMA molecule in the composition of the Modeling Liquid, a hydrophilic monomer which can cause water absorption due to a hydroxyl and carbonyl group [16]. Therefore, as it was reported even in previous studies, HEMA can reduce the hardness of composite resin [17]. In our study the group in which the Modeling Liquid was used recorded highest hardness value when comparing to the 7th generation of adhesive system and to the universal bonding system. That aspect might be correlated to the presence of UDMA molecule in the composition. This molecule consists of two urethane bonds and a flexible aliphatic core and forms double hydrogen bonds [18]. It has been reported that resins containing UDMA have superior polymerization rates and a high degree of conversion [18]. Consequently, the degree of conversion and polymerization rate can affect the surface hardness of the samples.

However, Tuncer et al. pointed that differences in microhardness between different composites may not be attributed to the degree of conversion [7]. Also Kutuk et al tested Modeling Liquid and two universal adhesive agents (G-Premio Bond, GC Corp.; OptiBond XTR, KavoKerr, Orange, CA, United States) as modeling agents in combination with nanohybrid composite resins [11]. The study found the lowest microhardness values when OptiBond XTR was used. Contradictory to the findings of this study, in our research the 7th generation of bonding agent determined lower microhardness of the tested materials when comparing to the Modeling Liquid group and control group and the same effect as the universal bonding resin. The composite resins hardness is also determined by the characteristics of filler particles and their interaction with the polymers [19,20]. It was reported that nanofilled composite resins exhibit improved hardness and abrasion resistance when comparing to other categories of composite resins [21]. That was the reason for including nanohybrid composites as testing materials in our study. The low-viscosity agents used to improve composites handling characteristics act by reducing the surface tension [22], but also by filling the defects in the material by diffusing through the pores resulted during layering procedure, making the material more resistant to degradation [5,23]. The final layer of restorative material has a decisive effect on aesthetics, color stability, and surface roughness [24]. Smooth and well-polished surfaces decrease plaque retention and consequently lower the risk of secondary caries and staining. In our study, the roughness values of both tested composite resins were lower than the plaque accumulation threshold of 20 µm [25]. Adequate finished and polished surfaces are mandatory to achieve long-lasting clinical restorations. Composites having nanoparticles present high polishability when using tools containing Al2O3 or diamond particles [26,27]. In our study, the specimens were polished using Sof-Lex aluminum oxide discs to achieve optimal surface smoothness. Following the polishing procedure, in our study the samples were submersed in distilled water for 24 hours to remove unreacted monomers and to allow post-polymerization process. It has been suggested that some liquid agents can be used to achieve smooth composite resin surface [28,29]. However, it has been proved to be very difficult to obtain a regular surface when using liquid resins [30]. All tested modeling agents in the present study had no effect on surface roughness of composite resins (table 3), so the second null hypotesis was accepted.].

5. CONCLUSIONS

Within the limitations of this study, all evaluated modeling agents decreased the surface microhardness of the tested nanohybrid composite resins. None of the modeling agents influenced the surface roughness of the composites. Further clinical studies should be performed for more accurate understanding of the effects of modeling agents on the mechanical properties and surface condition of composite resins.

AUTHOR CONTRIBUTIONS

Concept-SS, GP, GI; protocol-SS, IN, AM; data gathering and analysis-SS, IT, AG; data interpretation-SS, GP; revising the manuscript-SA, GI.

CONFLICT OF INTEREST

Authors declare that there is no conflict of interests.

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Simona Stoleriu is an educationist, researcher, and specialist in cariology and operative dentistry. Since 2018 she has been associate professor at the Faculty of Dental Medicine, "Grigore T. Popa" University of Medicine and Pharmacy, Iaşi, Romania. She received her PhD in Medical Dentistry in 2009 and she became senior specialist in General Dentistry in 2006. Her research activity focused on early diagnosis of dental caries, non-operative and restorative treatment of caries lesion, diagnosis and treatment of wear lesions, the behavior of restorative materials in oral environment, factors which can influence the surface condition and mechanical properties of direct restorative materials, and remineralization of dental hard tissues. She has also been invited as a speaker at many national and international congresses and received 10 awards for her scientific activity.

Questions

1. Different tools have been developed to improve the fit and the configuration of composite resins:
❑a. Titanium coated instruments;
❑b. Carbon coated instruments;
❑c. Resin knives;
❑d. Diamond disks;

2. Practitioners have used multiple lubricants in the layering process of composite application to minimize adhesiveness of the material to the instrument:
❑a. Acetone;
❑b. Glycerine;
❑c. Modeling liquid;
❑d. Flowable composite.

3. The following is true regarding the conclusions of the present study:
❑a. Modeling liquid had no effect on surface hardness;
❑b. The use of 7th generation of adhesive system increased surface roughness of composite resin;
❑c. The use of universal bonding system had no effect on surface roughness of composite resin;
❑d. Modeling liquid increased composite surface roughness.

4. Some studies have pointed that bonding agents used on the extern layer of the restoration:
❑a. Have no effect on composite color;
❑b. Have no effect on composite physical properties;
❑c. Can change the composite chemical properties;
❑d. Increase the viscosity of composite resin.