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Resin composite color change by spatula manipulation, effects on surface and color stability

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ABSTRACT

Aim: *To evaluate the influence of spatulas on resin composite color stability, and characterize the surface of both the spatulas and the resin composites before and after manipulation.* ***Materials and Method:*** *Discs (ø6 mm x 2 mm) of suprananofilled resin composite (Palfique LX5/Tokuyama) and nanohybrid resin (Empress Direct/Ivoclar Vivadent) were fabricated. They were divided into groups (n=10), and manipulated with different spatulas: non-manipulated (control), metal spatulas (Almore Millennium/ Golgran, Almore #3/ Quinelato, LM Arte Modella/ Quinelato), or plastic spatulas (Jon). Manipulation involved lightly pressing the spatula 50 times against the resin composite on waterproof paper. Color was analyzed at three time points: immediately, after finishing and polishing, and after 24-hour immersion in distilled water, with CIELab* parameters, ΔE_{ab}, ΔE_{00}, and ΔWI_D. Spatulas and resins were submitted to micromorphological and energy-dispersive X-ray spectroscopic (EDS) analysis. Generalized linear models or Kruskal-Wallis and Dunn tests (α=5%) were applied.* ***Results:*** *Resin color changed after manipulation with a metal spatula, but there was no significant difference in ΔE_{ab} and ΔE_{00} for the resin composites according to the spatula ($p>0.05$). The suprananofilled resin varied more than the nanohybrid resin over time ($p<0.05$). Abrasive wear was observed on the spatulas after manipulating the resin composites, with greater wear for the spatulas used with suprananofilled resin. EDS showed different spatula and resin composite compositions.* ***Conclusions:*** *Metal spatulas influenced resin composite color stability, with greater color change for suprananofilled resin. The spatulas exhibited abrasive wear, attributable to the difference in hardness between the spatulas and the resin composites.*

Keywords: *color - composite resins - scanning electron microscopy*

Alteração de cor de resina composta após manipulação com espátula, efeitos na superfície e estabilidade de cor

RESUMO

Este estudo avaliou a influência das espátulas na estabilidade da cor da resina composta e caracterizou a superfície das espátulas e das resinas compostas antes e após a manipulação. Foram confeccionados discos (ø6mm x 2mm) de resina composta suprananoparticulada (Palfique LX5/Tokuyama) e nanohíbrida (Empress Direct/Ivoclar Vivadent). Cada grupo foi manipulado com diferentes espátulas (n=10): não manipuladas (controle), metálicas (Almore Millennium/ Golgran, Almore #3/ Quinelato, LM Arte Modella/ Quinelato) e espátula plástica (Jon). A manipulação foi realizada pressionando levemente a espátula 50 vezes contra a resina composta sobre papel impermeável. A análise de cor foi avaliada em três momentos: imediato, após acabamento e polimento e após 24 horas de imersão em água destilada, com parâmetros CIELab, ΔE_{ab}, ΔE_{00} e ΔWI_D. Espátulas e resinas foram submetidas a análises micromorfológicas e espectroscópicas por raios X por energia dispersiva (EDS). Foram aplicados modelos lineares generalizados ou testes de Kruskal-Wallis e Dunn (α=5%). Houve alteração de cor após a manipulação das resinas com as espátulas metálicas, mas ΔE_{ab} e ΔE_{00} não apresentaram diferenças significativas para as resinas compostas em relação ao tipo de espátula ($p>0,05$). As variações foram maiores para a resina suprananoparticulada do que para a resina nanohíbrida durante todo o período de tempo ($p<0,05$). Foi observado desgaste abrasivo nas espátulas após manipulação das resinas compostas, notando maior desgaste para a resina suprananoparticulada. A EDS mostrou diferentes composições de espátula e resina composta. As espátulas metálicas influenciaram a estabilidade da cor da resina composta, destacando maior alteração de cor para a resina suprananoparticulada. As espátulas apresentaram desgaste abrasivo atribuído à diferença de dureza entre as espátulas e as resinas compostas.*

Palavras-chave: *cor - resinas compostas - microscopia eletrônica de varredura*

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INTRODUCTION

Mimicry and longevity are desirable features in restorative treatments[1]. Resin composites replicate the optical characteristics of dental tissue and have satisfactory physical properties, making them suitable for the purpose[2-6]. Resin composites consist of an organic matrix made primarily of Bis-GMA (bisphenol glycidyl methacrylate) monomers, which are highly viscous. This can be corrected by incorporating other monomers such as TEGDMA (triethylene glycol dimethacrylate) and UDMA (urethane dimethacrylate) as diluents[7,8]. The material is strengthened by adding fillers, particularly in the form of nanoparticles and nanohybrids, with excellent outcomes for resistance, handling, polishing, and long-term gloss retention[3,9-12]. Over time, fillers have undergone modifications to address issues of low mechanical strength, especially when used for posterior teeth[6,13-15]. These alterations have involved changes in the shape, composition, size and concentration of particles[16], particularly in nanofilled and nanohybrid resins[17]. Some manufacturers add particles such as zirconia (also known as zirconium dioxide) and barium glass to the resin composite to enhance its mechanical properties[18].

Resin composites for clinical applications require the use of specific instruments, such as spatulas, which are available in various shapes, thicknesses, lengths and compositions. Spatulas are used to remove the resin composite from its packaging, and place it in the cavity[19]. When the material is used, particularly for aesthetic procedures involving the vestibular surfaces of anterior teeth, it is smoothed with spatulas and brushes to achieve the proper contour and dental anatomy[20,21].

When the resin composite is manipulated with a spatula, the friction between the two surfaces in relative motion creates abrasion. The result of this process is determined by the roughness, geometry, coefficient of friction, velocity, load, distance, and hardness of the surfaces in contact. The difference in hardness between the materials can result in a phenomenon generally referred to as wear, based on the principles of tribology, the science used to describe the phenomena of friction, wear and lubrication[22]. Consequently, the interaction of the tribological pair of "resin composite and spatula" during manipulation could alter the surface of both the resin composite and the spatula, according to their hardness, leading to abrasive wear between them[23]. In resin composites, this wear-related interaction could interfere with color stability, leading to aesthetic problems, especially for treatments with high aesthetic demands in anterior teeth.

The aim of this study was therefore to evaluate the influence of spatula types on the color stability of resin composites, and to characterize the surface of the spatula and resin composite before and after manipulation. The following null hypotheses were tested: H01) The spatulas used do not alter the color of the resin composites; H02) There is no difference between the surfaces of the spatulas before and after manipulation of the resin composites; H03) There is no difference between the surfaces of the resin composites before and after manipulation by the spatulas.

MATERIALS AND METHOD

Preparation of specimens and manipulation of resins with spatulas

The resin composites and spatulas evaluated are presented in Table 1. A total of 100 Teflon molds (2 mm high and 6 mm internal diameter) were used to prepare 50 specimens with suprananofilled resin (LX5 Palfique/Tokuyama/WE enamel color), and 50 specimens with nanohybrid resin (Empress Direct/Ivoclar/BL-L enamel color), yielding 10 specimens of each resin allocated to each type of spatula. The resin composites were manipulated with the different metal spatulas tested (Almore Millennium/Golgran, Almore number 3/Quinelato, LM Arte Modella/Quinelato,) or plastic spatula (Jon), and then placed in the Teflon mold. Fifty manipulations were standardized with the respective spatula designated for each specimen, with movements similar to those used to flatten the resin on a cavity when performing a direct veneer. This number was determined after observations in collaboration with clinical professionals. The specimens were manipulated on a disposable impermeable paper block. The same face of a previously unused spatula was always employed for each specimen, thus eliminating the possibility of any potential manipulation-related influence between different types of resin composites. A plastic spatula (plastic spatula/Jon) was used for the specimens in the control group (non-manipulated), just to remove the resin from its packaging, and place it directly into the mold (without any manipulation).

Table 1. Specification of the materials.

Commercial name/ manufacturer	Description	Composition	Manufacturer's recommended use
Palfique LX5/ Tokuyama Shade WE	Suprananofilled resin composite	Organic phase: BisGMA (Bisphenol A-glycidyl methacrylate); TEGDMA (Triethylene glycol dimethacrylate) Inorganic phase: Silica-zirconia Particles: Spherical Average size: 0.2 μm Monodisperse = all particles have uniform dimensions Weight concentration: 82% Volume concentration: 71%	Photocuring for 20 seconds
Empress Direct/ Ivoclar Shade BL/L	Nanohybrid resin composite	Organic phase: Dimethacrylates (20-21% by weight, opalescent 17% by weight); Copolymer (77.5 - 79%; opalescent 83% by weight); Additives, catalysts, stabilizers, and pigments (<1.0% by weight) Inorganic phase: Barium glass; ytterbium trifluoride; mixed oxides; silicon dioxides Particle type: Nanohybrid Average size: 550 nm Weight concentration: 75-79% Volume concentration: 52-59%	Photocuring for 20 seconds
Golgran	Metal spatula/ Almore (ALM) Millennium - code 98-14	Metal	Manipulation and application of the material in the cavity/tooth.
Quinelato	Metal spatula/ #3 Almore - code QD.325.03	Metal	
Quinelato	Metal spatula/ LM-Arte Modella - code 442-443 XSi	Metal	
Jon	Plastic spatula/ Jon - code 7165 - color: green	Thermoplastic polymer	

* Information provided on the company websites

Two strips of polyester were used to eliminate any bubbles and achieve a smooth surface: one strip was placed between the glass plate and the mold, and the other, over the mold after resin insertion. A glass slide was then placed over the resin, and a weight of 500 grams was applied for 30 seconds. Subsequently, the glass slide was removed for resin composite photoactivation using a LED light device (Valo, Ultradent, South Jordan, UT, USA) for 20 seconds with irradiance 1000 mW/cm².

The specimens were identified according to the groups (n=10), and to their upper surface (facing the light from the photoactivator). Their surface was evaluated for color (initial), after applying the finishing/polishing protocol, and after storage in humidity for 24 hours in a bacteriological incubator at 37 °C.

Finishing and polishing procedures and storage

Specimen surfaces were finished and polished with fine and extra-fine polishing disks (Sof-Lex Pop-On/3M). The disks were attached to a dedicated mandrel for use at low speed, and gentle pressure was applied to each disk for 15 seconds. The polishing procedure was carried out by fixing the specimens on an acrylic plate with sticky wax, keeping the surface parallel to the horizontal plane. After applying each type of sanding disk, the surface was rinsed with water and air-sprayed for 15 seconds. The sanding disks were replaced after every three applications. Subsequently, the specimens were submitted to surface color analysis, stored in distilled water for 24 hours, and reevaluated.

Color evaluations

Specimen surfaces were dried briefly with absorbent paper, and color analyzed by placing them a box with a white background to standardize the lighting. The spectrophotometer (VITA Easyshade® Advance, Vita, Germany) was previously calibrated automatically, by using the handpiece of the device positioned on the calibration block holder, with the measuring tip supported and pressing the calibration block at a 90° angle. Color was then evaluated with the measuring tip supported and fully seated, perpendicular to the surface of the specimens, always in the same position, in the central region. This measurement process was performed in duplicate to support the data used in the analysis. Specimen color was evaluated at three different times: immediately after fabrication, after finishing and polishing, and after 24 hours of immersion in distilled water.

The color of the resin composites was evaluated using the CIELab* parameters. The ΔL*, Δa* and Δb* values were measured for each group at each time, and used to assess color change (ΔE_{ab}) with the following formula[24]: $\Delta E_{ab} = \sqrt{((\Delta L^*)^2 + (\Delta a^*)^2 + (\Delta b^*)^2)}$. The perceptibility and acceptability thresholds considered for ΔE_{ab} were 1.2 and 2.7, respectively[24,25]. The color change was also evaluated using CIEDE2000 (ΔE_{00}), which uses h (hue) and C (chroma) values[26]. ΔE_{00} values of 0.8 and 1.8 were adopted as the perceptibility and acceptability limits, respectively[25]. Dental staining was monitored using the Whiteness Index for Dentistry (WI_D), where the L*, a*, and b* parameters were used in the following equation[27]: $WI_D = 0.511L^* - 2.324a^* - 1.100b^*$. Differences in WI_D (ΔWI_D) were also assessed between the evaluations, using threshold values of 0.72 for perceptibility and 2.60 for acceptability[27].

Scanning electron microscopy (SEM) and relative chemical composition analyses of the spatulas and resin composites

The surfaces of the spatulas (n=2) and resin composites (n=2) were evaluated for micromorphology before and after manipulation by SEM (Thermo Fisher Scientific, Model Quattro S, Thermo Scientific UltraDry, Brno, Czech Republic), and for relative chemical composition by energy-dispersive X-ray spectroscopy (EDS) (6070, LEO Electron Microscopy/Oxford, Cambridge, England). All resin surfaces were gold sputtered (gold layer thickness estimated at 200 Å) using a sputter coater (Sputter Coater Emitech K450, Kent, United Kingdom).

Images of surface micromorphology were obtained for the spatulas at magnifications of 13 x and 500 x in high resolution, with a voltage of 20.00 kV and a spot size of 100 pA. The resin composite surfaces were assessed using a magnification of 5000 x in high resolution, with a voltage of 20.00 kV and a spot size of 100 pA. Qualitative surface analyses were conducted to assess the presence of scratches and/or irregularities on the surfaces of the spatulas, as well as the presence and morphology of the filler particles in the resin composites. The relative chemical composition was expressed as the percentage of chemical elements present in the central region of the surface of both the spatula and the resin composite specimen.

Statistical analyses

After descriptive and exploratory analysis of all the data, generalized linear mixed-effects models for repeated measures over time were applied to L* and WI_D. Generalized linear models were also fitted to analyze ΔE_{ab} and ΔE_{00}, considering the study factors of resin and spatula, and the interaction between them. Other variables that did not fit a known distribution were analyzed using non-parametric tests, such as Kruskal-Wallis and Dunn for spatula comparisons, Friedman and for time comparisons, and Mann-Whitney for resin comparisons. The analyses were conducted using the R software (R Core Team, 2023) with a significance level of 5%.

RESULTS

For the L* parameter (Table 2), neither resin changed significantly over time ($p>0.05$). The suprananofilled resin composite showed a difference in L* depending on the spatula used ($p<0.05$), with higher luminosity when a plastic spatula was used, and lower luminosity when the #3 Almore or LM-Arte Modella spatulas were used ($p<0.05$). The nanohybrid resin composite showed a significant decrease in WI_D over time ($p<0.05$), with no significant difference between the types of manipulation ($p>0.05$). The suprananofilled resin showed differences in WI_D depending on the spatula used ($p<0.05$), with higher values for the non-manipulated groups and those manipulated with a plastic spatula, at all time points ($p<0.05$),

Table 2. Mean (standard deviation) L* and WI_D according to spatula type, time, and resin

Parameter	Resin composite	Spatula	Time		
			Immediate	After polishing	After 24-hour storage in water
			Mean (standard deviation)	Mean (standard deviation)	Mean (standard deviation)
L*	Suprananofilled	Non-manipulated	91.07 (1.49) Ab	91.18 (1.30) Ab	91.07 (1.99) Ab
		Almore Millennium	90.81 (2.53) Ab	90.89 (2.68) Ab	90.49 (2.86) Ab
		#3 Almore	87.07 (2.30) Ac	87.46 (2.47) Ac	87.95 (2.48) Ac
		LM-Arte Modella	86.30 (1.99) Ac	86.10 (1.95) Ac	86.39 (1.68) Ac
		Plastic	93.60 (2.02) Aa	93.51 (2.03) Aa	93.94 (2.28) Aa
	p-value		p(spatula)<0.0001; p(time)=0.3967; p(interaction)=0.3626		
	Nanohybrid	Non-manipulated	86.00 (0.54) Aa	85.77 (0.68) Aa	85.69 (0.63) Aa
		Almore Millennium	85.47 (2.23) Aa	86.07 (1.82) Aa	86.16 (2.06) Aa
		#3 Almore	84.68 (1.01) Aa	84.77 (1.69) Aa	84.78 (1.10) Aa
		LM-Arte Modella	85.36 (1.11) Aa	85.41 (1.32) Aa	85.46 (1.54) Aa
		Plastic	85.67 (0.47) Aa	85.41 (0.68) Aa	85.46 (0.83) Aa
	p-value		p(spatula)=0.1678; p(time)=0.7942; p(interaction)=0.5271		
WI_D	Suprananofilled	Non-manipulated	44.62 (0.84) Aa	44.02 (1.11) Ba	42.64 (0.86) Ca
		Almore Millennium	39.19 (3.98) Abc	38.37 (3.95) Bbc	36.66 (4.19) Cc
		#3 Almore	40.83 (1.39) Ab	40.59 (1.73) Ab	39.60 (1.31) Bb
		LM-Arte Modella	37.23 (2.13) Ac	37.16 (1.95) Ac	36.35 (1.50) Ac
		Plastic	44.17 (0.65) Aa	43.33 (0.73) Ba	41.97 (0.76) Ca
	p-value		p(spatula)<0.0001; p(time)<0.0001; p(interaction)=0.1830		
	Nanohybrid	Non-manipulated	34.24 (0.45) Aa	32.72 (1.01) Ba	31.36 (0.75) Ca
		Almore Millennium	33.27 (1.05) Aa	32.64 (1.37) Ba	30.83 (1.41) Ca
		#3 Almore	33.55 (0.62) Aa	32.44 (1.09) Ba	30.94 (0.88) Ca
		LM-Arte Modella	34.04 (2.26) Aa	32.52 (1.52) Ba	30.78 (1.44) Ca
		Plastic	33.45 (0.59) Aa	32.60 (0.82) Ba	31.28 (1.20) Ca
	p-value		p(spatula)=0.4937; p(time)<0.0001; p(interaction)=0.6817		

The same letters (uppercase horizontally and lowercase vertically, comparing the spatula types in each resin), indicate that there is no statistically significant difference within each variable (p > 0.05).

and lower values for Almore Millennium and LM-Arte Modella ($p<0.05$). There was no significant difference for ΔE_{ab} and ΔE_{00} regarding the spatula used ($p>0.05$) (Table 3). The variations were greater for the suprananofilled resin than for the nanohybrid resin over the entire period ($p<0.05$).

Regarding the suprananofilled resin composite, ΔWI_D differed according to the spatula used ($p<0.05$) (Table 4), with the variation in ΔWI_D being more negative (decrease) with the Almore Millennium than with the #3 Almore spatula ($p<0.05$). ΔWI_D was more negative in the nanohybrid resin than in the suprananofilled resin when non-manipulated or manipulated with #3 Almore and LM-Arte Modella spatulas ($p<0.05$).

The images of the surface micromorphology of the spatulas (Figs. 1 to 4) show that the Almore Millennium, #3 Almore, and plastic spatulas appeared unchanged with non-manipulation, while the LM-Arte Modella spatula already showed some scratches on its surface. After manipulation, all the spatulas showed surface wear, with scratches appearing on the Almore Millennium spatula, and overall surface wear on the other spatulas. The

Table 3. Mean (standard deviation) ΔE_{ab} and ΔE_{00} according to spatula type and resin at each time interval

Parameter	Resin composite	Spatula	Time point	
			After polishing - Immediate	After 24 hours - Immediate
			Mean (standard deviation)	Mean (standard deviation)
ΔE_{ab}	Suprananofilled	Non-manipulated	1.51 (0.58) a	3.02 (0.82) a
		Almore Millennium	0.99 (0.57) a	2.87 (0.74) a
		#3 Almore	1.30 (0.42) a	2.69 (1.00) a
		LM-Arte Modella	1.42 (0.92) a	2.35 (0.93) a
		Plastic	1.11 (0.51) a	2.91 (0.72) a
	Nanohybrid	Non-manipulated	1.32 (0.68) a	*1.90 (0.77) a
		Almore Millennium	1.46 (0.71) a	*2.17 (0.48) a
		#3 Almore	1.43 (0.45) a	*1.67 (0.64) a
		LM-Arte Modella	1.20 (0.75) a	*2.05 (0.83) a
		Plastic	1.13 (0.51)a	*1.58 (0.82) a
	p-value		p(resin)=0.6481; p(spatula)=0.5894; p(interaction)=0.4288	p(resin)<0.0001; p(spatula)=0.4886; p(interaction)=0.2751
ΔE_{00}	Suprananofilled	Non-manipulated	1.03 (0.36) a	2.14 (0.58) a
		Almore Millennium	0.69 (0.35) a	1.89 (0.48) a
		#3 Almore	0.89 (0.26) a	1.89 (0.66) a
		LM-Arte Modella	1.10 (0.97) a	1.72 (0.84) a
		Plastic	0.75 (0.32) a	1.97 (0.47) a
	Nanohybrid	Non-manipulated	0.95 (0.45) a	*1.41 (0.46) a
		Almore Millennium	1.00 (0.47) a	*1.55 (0.32) a
		#3 Almore	0.98 (0.30) a	*1.28 (0.41) a
		LM-Arte Modella	0.96 (0.82) a	*1.61 (0.90) a
		Plastic	0.77 (0.35) a	*1.12 (0.53) a
	p-value		p(resin)=0.5025; p(spatula)=0.3046; p(interaction)=0.5337	p(resin)<0.0001; p(spatula)=0.5047; p(interaction)=0.1821

*Differs from the supranano filled resin under the same spatula and time point conditions ($p \leq 0.05$). Different letters in the vertical direction, comparing the spatula types with each resin, indicate statistically significant differences ($p \leq 0.05$) in each evaluated parameter.

surfaces of the spatulas used to manipulate the nanohybrid resin showed more scratches, while those used to manipulate the supranano filled resin showed smoother surfaces.

The evaluations by EDS (Figs. 1 to 4) showed that the composition of the Almore Millennium spatula was predominantly 85% iron (Fe) and 11% chromium (Cr), with 1% carbon, 1% oxygen (O), 1% silicon, and 1% manganese (Mn), while the #3 Almore spatula was composed of approximately 54% nickel (Ni), 44% titanium (Ti), and 2% carbon (C). The LM-Arte Modella spatula was made up of about 77% iron (Fe) and 16% chromium (Cr), with 1% aluminum (Al), 1% carbon (C), 1% nickel (Ni), 1% oxygen (O), 1% manganese (Mn), 1% silicon, and 1% molybdenum (Mo), and the plastic spatula had 67% carbon (C) and 33% oxygen (O) in its composition.

There was no difference between the images of the surfaces of the resin composites (Fig. 5) without manipulation and after manipulation. Regarding the composition of the resins, the EDS analysis showed that the supranano filled resin was composed of approximately 42% oxygen (O), 29% silicon (Si), 15% carbon (C), 12% zirconium (Zr), 1% sodium (Na), and 1% chlorine (Cl), while the nanohybrid resin consisted of approximately 38% oxygen (O), 25% silicon (Si), 14% carbon (C), 13% barium (Ba),

Table 4. Median (minimum; maximum) ΔWI_D according to spatula type and resin at each time point

Resin composite	Spatula	Time point	
		After polishing - Immediate	After 24 hours - Immediate
		Median (minimum; maximum)	Median (minimum; maximum)
Suprananofilled	Non-manipulated	-0.52 (-1.77; 0.31) a	-1.99 (-2.84; -0.84) ab
	Almore Millennium	-1.03 (-1.97; 0.34) a	-2.85 (-3.15; -1.39) b
	#3 Almore	-0.22 (-2.04; 1.47) a	-1.47 (-2.23; -0.20) a
	LM-Arte Modella	-0.94 (-2.67; 8.27) a	-1.38 (-3.44; 6.05) ab
	Plastic	-1.05 (-1.75; 0.76) a	-2.01 (-4.02; -0.86) ab
p-value		0.7171	0.0172
Nanohybrid	Non-manipulated	*-1.72 (-2.56; 0.62) a	*-3.13 (-4.26; -1.26) a
	Almore Millennium	-0.94 (-2.52; 1.43) a	-2.21 (-4.95; -1.09) a
	#3 Almore	-1.12 (-2.41; 0.24) a	*-2.78 (-3.76; -1.37) a
	LM-Arte Modella	-1.18 (-6.78; 1.03) a	*-2.69 (-9.28; -1.40) a
	Plastic	-0.85 (-2.28; 1.24) a	-2.20 (-4.41; -0.44) a
p-value		0.3997	0.5754

*Differ from the suprananofilled resin under the same spatula and time point conditions (p≤0.05). Different letters in the columns, comparing the spatula types in each resin, indicate that there is a statistically significant difference (p≤0.05).

5% zirconium (Zr), 3% aluminum (Al), 1% sodium (Na) and 1% calcium (Ca).

In addition to determining the composition of each spatula and resin composite, the EDS analysis enabled the Mohs hardness of each component (Table 5) to be related to the Mohs hardness of the spatula constituents and the filler particles of the suprananofilled and nanohybrid resin composites.

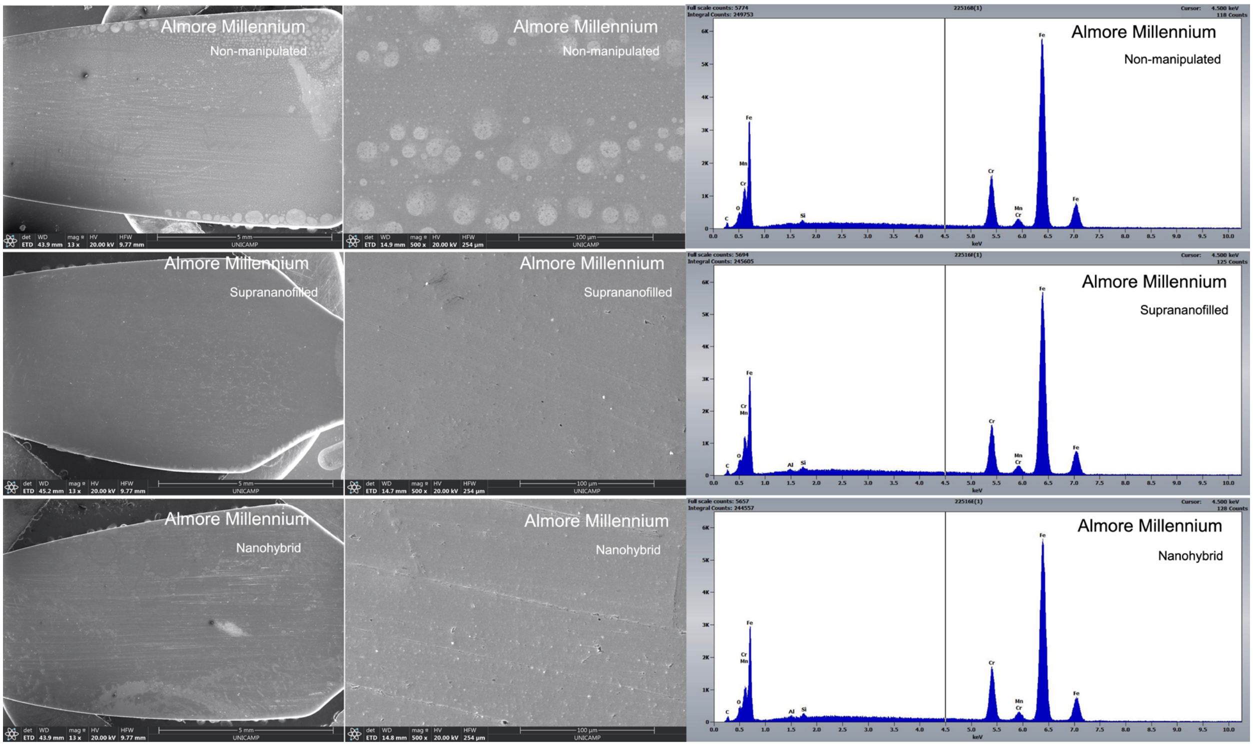

Fig. 1. Images of the surface micromorphology of Almore Millennium spatulas (13 and 500 x) according to different manipulation conditions and EDS analysis

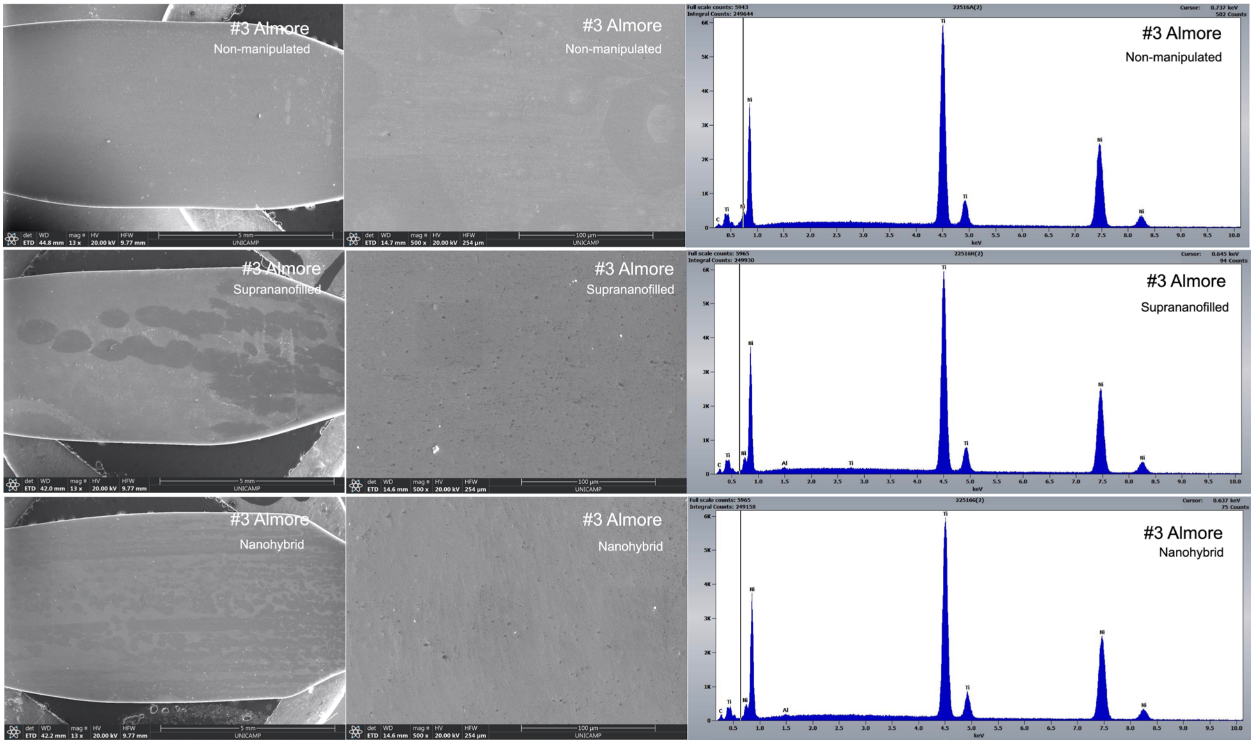

Fig. 2. Images of the surface micromorphology of #3 Almore spatulas (13 and 500 x) according to different manipulation conditions and EDS analysis

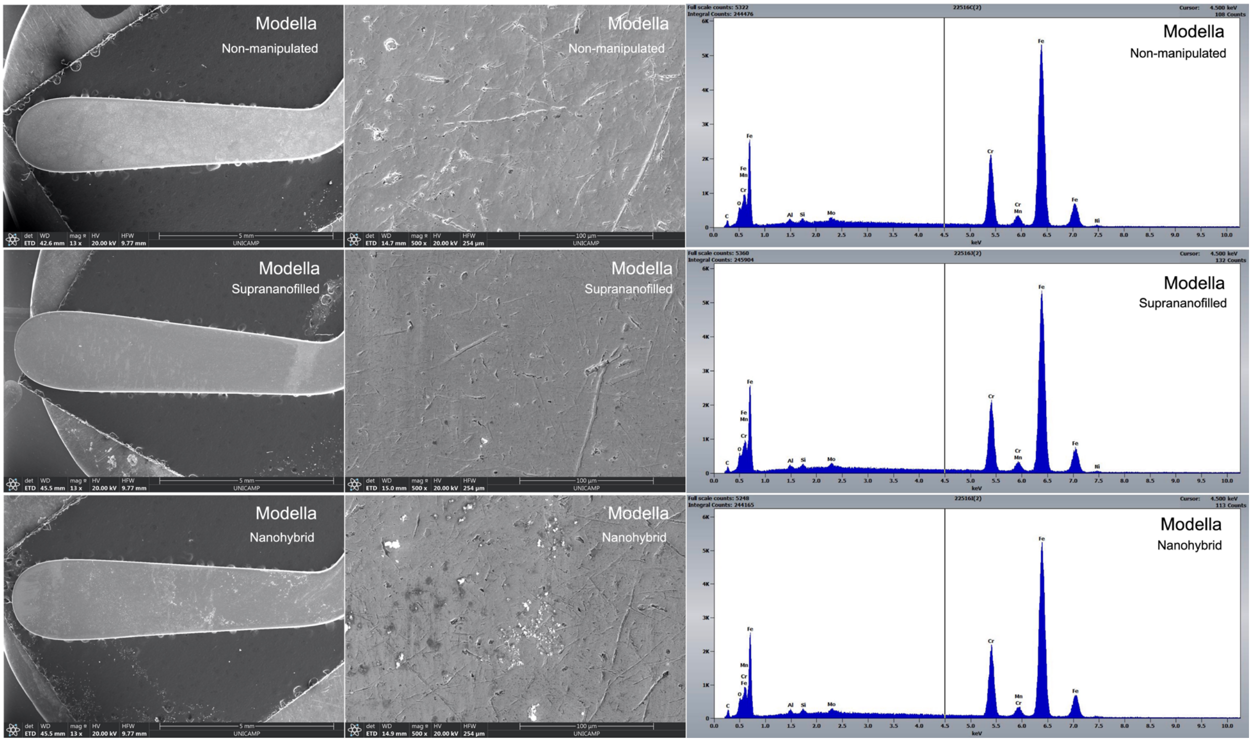

Fig. 3. Images of the surface micromorphology of LM-Arte Modella spatulas (13 and 500 x) according to different manipulation conditions and EDS analysis

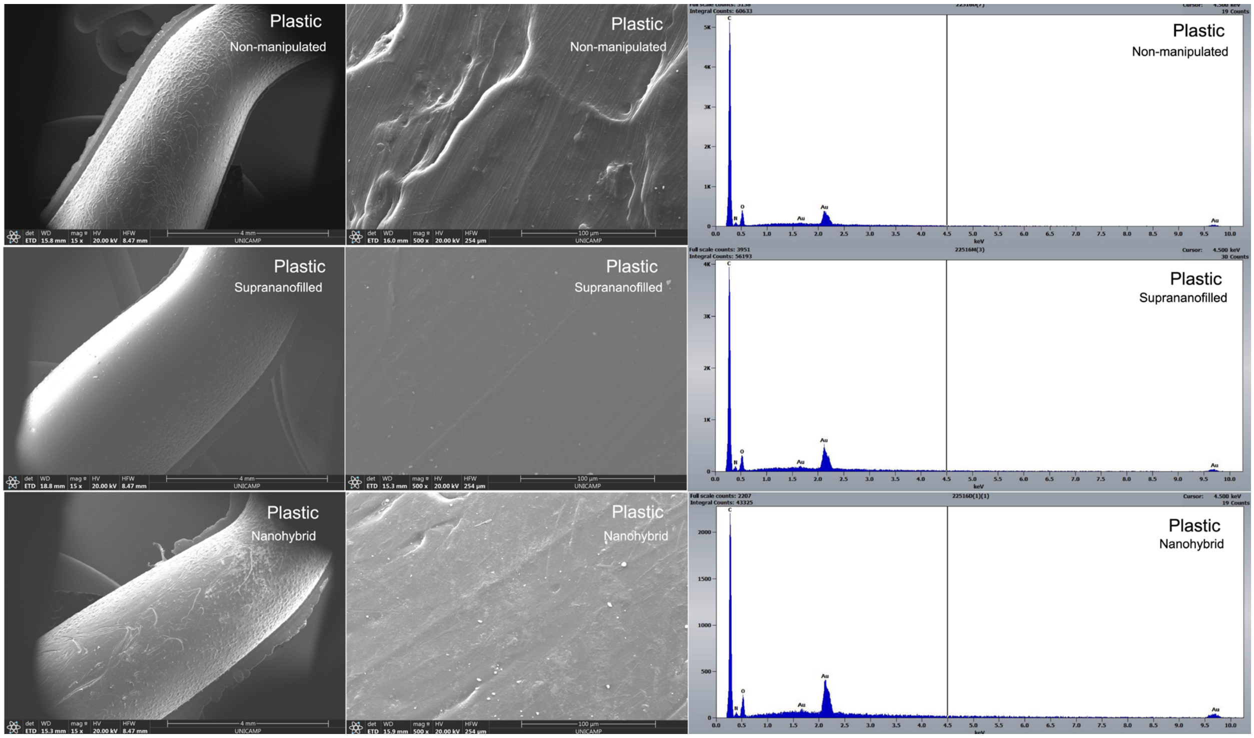

Fig. 4. Images of the surface micromorphology of plastic spatulas (13 and 500 x) according to different manipulation conditions and EDS analysis

Table 5: Percentage of constituents found in spatulas, and Mohs hardness value relationship

Chemical element	Mohs hardness	Constituents of the spatulas (%)			
		Almore Millennium	#3 Almore	LM-Arte Modella	Plastic
Iron	4[28]	85	-	77	-
Chromium	8.5[28]	11	-	16	-
Carbon	-	1	2	1	67
Oxygen	-	1	-	1	33
Silicon	6[28]	1	-	1	-
Manganese	2[28]	1	-	1	-
Molybdenum	5.5[28]	-	-	1	-
Aluminum	2.75[28]	-	-	1	-
Nickel	4[28]	-	54	1	-
Titanium	6 [29]	-	44	-	-

DISCUSSION

When resin composite is manipulated with spatulas, it is important that it should preserve its physical characteristics, especially those related to color stability. The null hypotheses tested in the present study were rejected because they stated that the spatulas used do not influence the color change of the resin composites (H01), and that there is no difference between the surfaces of the spatulas before and after resin manipulation (H02).

This study was able to correlate the color changes found in the resin composites (CIELab*) with the spatula surface alterations (SEM), and to explain these changes according to the constituents of the spatulas and the resin composites (EDS). The friction generated between the tribological “spatula vs. resin” pair resulted in the abrasion of both, due to the difference in hardness between the filler particles of the resin composites and the components of the spatulas. It could explain the color changes, especially of the suprananofilled resin, in which case the components of the spatula alloy had lower Mohs hardness than the filler particles. When two surfaces have significantly different hardness levels, the micro-coarseness of the harder surface grates against the softer surface like a micro-plowing mechanism. Microscopically, a burr is formed in front of the abrasive particle, and the material is continuously displaced laterally, forming ridges adjacent to the grooves produced. The cyclic movement causes many grooves to be formed parallel to the direction of movement of the abrasive coarse grain, and the proximity of these grooves can weaken the more ductile material, which becomes deformed and is removed by a microfracture mechanism[22,28,29].

Evaluation of the L* and WI_D parameters found no difference between the nanohybrid resin composite manipulated with different types of spatulas and the non-manipulated resin. Nanohybrid resins contain barium, an element with low hardness (Mohs hardness 1.25)[28-30], which is responsible for color stability. The spatulas used to manipulate the nanohybrid resin showed microgrooves on their surface (Figs. 1 to 4), but they were not sufficient to promote significant color alteration in this resin composite.

In contrast, the suprananofilled resin composite contains harder filler particles[31], which promoted greater wear on the surface of the spatulas (Figs. 1 to 4). The suprananofilled resin filler particles contain more zirconium (Fig. 5), which has high hardness (Mohs hardness of 8)[31]. This difference in hardness between the filler particles of the suprananofilled resin and the components of the alloy of the spatulas studied (Table 5) explains the significant color alterations in this resin.

There were significant differences between the L* and WI_D parameters of the suprananofilled resin when it was manipulated with different spatulas, compared to the unmanipulated resin. The L* value is related to the brightness of the resin composite. Only the Almore Millennium spatula did not cause alteration with manipulation. The #3 Almore and LM-Arte Modella spatulas led to lower brightness, while the plastic spatula increased brightness. This suggests that the alterations L* result from the different levels of hardness between the filler particles in the suprananofilled resin and the components of the spatulas (Table 5).

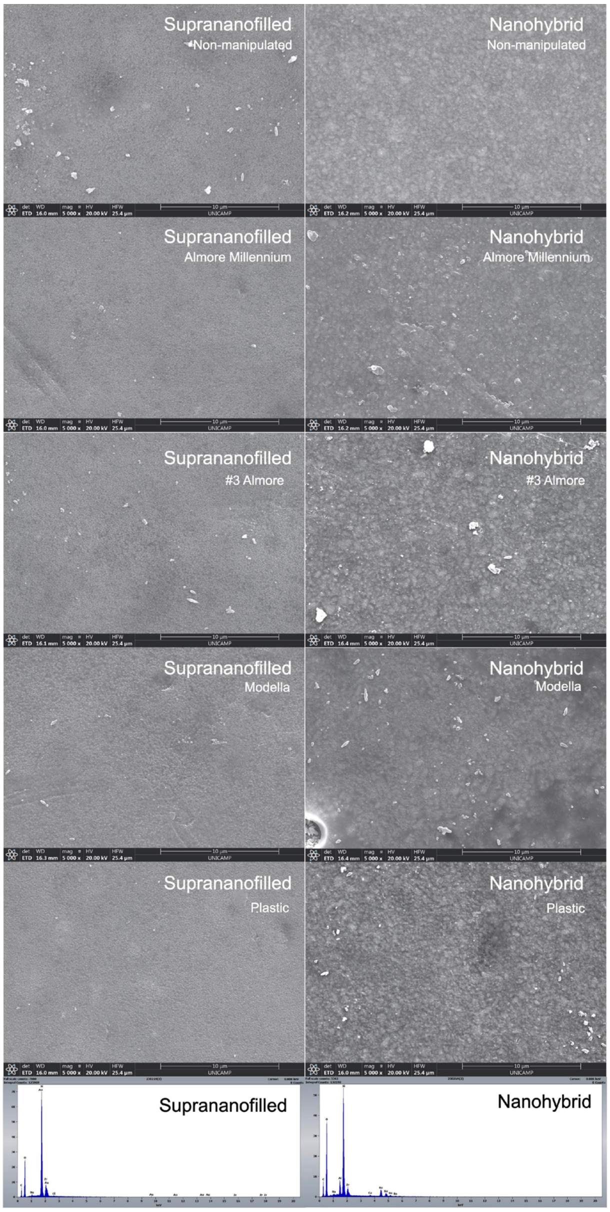

Fig. 5: Scanning electron microscopy images (5000 x magnification) of the surface of the composite resins manipulated by different spatulas according to EDS surface analysis

The WI_D parameter correlates better to visual perception than whitening evaluation indexes do. In this study, although the nanohybrid resin remained stable after manipulation using different spatulas, its color did change over time. Some studies show that resin composites may undergo color changes for up to 14 days after polymerization, after which they attain color stability. However, in this study, the suprananofilled resins underwent not only color change over time, but also significant alterations with manipulation using different spatulas.

Considering ΔE_{ab} and ΔE_{00}, there was no significant color difference for either resin composite, a result that cannot be attributed either to the manipulation by different spatulas or to the time point. However, even though the lower ΔE_{ab} and ΔE_{00} values for the nanohybrid resin were not significant at the last time point, they indicated better color stability after the time points evaluated. Staining was more pronounced at the immediate time point than at the final time (after 24 hours immersed in distilled water). Although color changes are expected over time for all resin composites, in this study, the color changes were greater in the nanohybrid than in the suprananofilled resin at the evaluated time points. It is important to highlight that the staining values of the suprananofilled composite were significant for the manipulation of different spatulas, but not for the time points.

Regarding the difference in staining values over time (ΔWI_D), the non-manipulated group showed significant staining differences for the suprananofilled resin. This was also the case in the groups manipulated with the different spatulas, except for the #3 Almore spatula. In contrast, the staining differences for the nanohybrid resin were not significant across the evaluated time points.

Concerning the limits of perceptibility and acceptability for ΔE_{ab} and ΔE_{00}, clinically perceptible color changes were found for the suprananofilled resin composite in the non-manipulated, #3 Almore, and LM-Arte Modella groups (at the immediate and after polishing time points), and clinically unacceptable changes were detected in the non-manipulated, Almore Millennium, and plastic groups (at the immediate and 24-hour immersion time points). In contrast, perceptible changes were found for the nanohybrid resin, but they were all clinically acceptable. Further research should be conducted on the color stability of suprananofilled resin, considering that even without manipulation, its color changed significantly between the immediate and the 24-hour immersion time points.

Regarding the analyzed color parameters, the most relevant parameters for color alteration were the L* and WI_D values. These showed a significant color change (lower values) for the suprananofilled resin, noting that the LM-Arte Modella spatula influenced the results more negatively. One of the metals contained in this spatula is aluminum, a ductile lightweight metal with a gray appearance[32-35]. These characteristics may explain the color changes in luminosity and staining. Furthermore, this spatula has the highest number of components, and the nature of these components may also have contributed to the color change in the L* and WI_D values.

The third null hypothesis tested (H03), stating that there is no difference between the resin surfaces before and after manipulation by the spatulas, was not rejected. Visual analysis of the results from SEM images, and EDS composition analysis of the resin composites showed no considerable alteration before or after manipulation. It was expected that the constituent metals of the spatulas would be incorporated into the restorative material; however, the EDS analysis of the resin composites showed no difference in composition before and after manipulation. It is worth noting that the nature of the organic matrix in resin composites may have influenced their color stability[12,16,36-39]. However, based on the results found in this study, the findings of the cited authors could not be corroborated.

This study has shown that the relationship between the composition of the inorganic particles of resin composites and the composition of the spatulas was relevant for the color stability of the restorative material, especially considering the different manipulations. Concerning clinical application, these findings can help understand the color changes caused by manipulation that may affect resin composite, which is particularly important on the vestibular surfaces of anterior teeth. In clinical practice, the authors suggest avoiding excessive manipulation with metal spatulas, especially of suprananofilled resin. Further research in this area would be welcome to enable more precise decision-making. Based on the findings of this research, it is also suggested that certain materials could reduce the friction between the spatulas and resin composite, such as the modeling liquids used to improve the sliding action at the interface between materials. Manufacturers could reconsider spatula composition, including the use of specific coatings for spatulas, such as Teflon films and cold-worked alloys, among other technologies.

In conclusion, the metal spatulas influenced the color stability of the resin composites, considering that the suprananofilled resin underwent greater color change as a result of the different manipulations. The surfaces of the spatulas showed that there was abrasive wear of two bodies, attributed to the difference in the hardness values of the spatulas and resin composites, considering that abrasion was greater in the spatulas used to manipulate the suprananofilled resin. The surfaces of the resin composites underwent no significant morphological change from the different manipulations.

CONFLICT INTERESTS

The authors declare no potential conflicts of interest regarding the research, authorship, and/or publication of this article.

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