Influence of Curing Time, Overlay Material and Thickness on Three Light-curing Composites Used for Luting Indirect Composite Restorations

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Purpose: To assess the microhardness of three resin composites employed in the adhesive luting of indirect composite restorations and examine the influence of the overlay material and thickness as well as the curing time on polymerization rate.

Materials and Methods: Three commercially available resin composites were selected: Enamel Plus HRI (Micerium) (ENA), Saremco ELS (Saremco Dental) (SAR), Esthet-X HD (Dentsply/DeTrey) (EST-X). Post-polymerized cylinders of 6 different thicknesses were produced and used as overlays: 2 mm, 3 mm, 3.5 mm, 4 mm, 5 mm, and 6 mm. Two-mm-thick disks were produced and employed as underlays. A standardized amount of composite paste was placed between the underlay and the overlay surfaces which were maintained at a fixed distance of 0.5 mm. Light curing of the luting composite layer was performed through the overlays for 40, 80, or 120 s. For each specimen, the composite to be cured, the cured overlay, and the underlay were made out of the same batch of resin composite. All specimens were assigned to three experimental groups on the basis of the resin composite used, and to subgroups on the basis of the overlay thickness and the curing time, resulting in 54 experimental subgroups (n = 5). Forty-five additional specimens, 15 for each material under investigation, were produced and subjected to 40, 80, or 120 s of light curing using a microscope glass as an overlay; they were assigned to 9 control subgroups (n = 5). Three Vicker's hardness (VH) indentations were performed on each specimen. Means and standard deviations were calculated. Data were statistically analyzed using 3-way ANOVA. Within the same material, VH values lower than 55% of control were not considered acceptable.

Results: The used material, the overlay thickness, and the curing time significantly influenced VH values. In the ENA group, acceptable hardness values were achieved with 3.5-mm or thinner overlays after 120 or 80 s curing time (VH 41.75 and 39.32, respectively), and with 2-mm overlays after 40 s (VH 54.13). In the SAR group, acceptable hardness values were only achieved with 2-mm-thick overlays after 120 or 80 s curing time (VH 39.81 and 29.78, respectively). In the EST-X group, acceptable hardness values were only achieved with 3-mm or thinner overlays, after 120 or 80 s curing time (VH 36.20 and 36.03, respectively).

Conclusion: Curing time, restoration thickness, and overlay material significantly influenced the microhardness of the tested resin composites employed as luting agents. The clinician should carefully keep these factors under control.

Keywords: indirect composite restoration, luting, Vickers hardness.

J Adhes Dent 2012; 14: 377–384.
doi: 10.3290/j.jad.a22765

Submitted for publication: 26.06.10; accepted for publication: 10.07.11

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The ever-increasing demand for esthetic restorations has revolutionized modern dentistry and brought about the widespread use of resin composites. The achievement of a proper degree of conversion is of paramount clinical importance for these materials, as unreacted monomers and inadequate polymerization may compromise mechanical behavior under masticatory loads, leading to increased wear,[17] solubility, dimensional instability, color change, and reduced biocompatibility,[2] which may consequently affect the restoration's longevity and ultimate clinical success.[11,34] Inadequate polymerization occurs if the composite resin does not receive an adequate number of photons during the light-curing process.[23]

In accordance with respective clinical indications, resin composite materials are suitable for use for both indirect and direct restorations. Among the parameters that may influence the clinical success of indirect restorations, a proper degree of polymerization of the resin luting agent should be taken into account.[1] The adhesive luting of indirect restorations can be performed employing both dual-curing and light-curing cements.[7-15] An advantage of dual-curing materials is their self-curing component, which favors conversion even in the presence of scarce radiant energy; however, a disadvantage is that they have a relatively low viscosity and require a mixture of two elements, which promotes the formation of porosities or voids and the incorporation of bubbles. On the other hand, light-curing materials used as luting agents are easily handled and are characterized by controllable curing times that create high quality margins.[1] With no time restriction, it is easier to achieve a full seating of the inlay/onlay and to accurately remove all the excess cement, which improves the overall restoration quality. Only their light activation can constitute a disadvantage, since light polymerization of all portions of the cement may not always be possible, if the inlay is thick enough to significantly impair light penetration.[21]

Different methods exist to determine the degree of polymerization.[11] However, as hardness is considered one of the most important properties of a resin composite[6,26] and gives a good indication of the conversion of double bonds, hardness measurements are often used to determine the extent of polymerization of a resin composite.[9,16] Because higher hardness values are considered an indirect indication of greater material polymerization, microhardness assessment has become a widely accepted indirect method to effectively assess the degree of cure for resin composites.[11,32]

The aim of the present study was to assess the microhardness of three commercially available resin composites employed in the adhesive luting of indirect composite restorations, and evaluate the effect of the conversion rate of the overlay material, overlay thickness, and of curing time on the microhardness. The null hypothesis tested was that the microhardness values would not be affected by type of resin composite, overlay thickness, or curing time.

MATERIALS AND METHODS

Three commercially available resin composites were selected for this study: Enamel Plus HRI (shade UD2, Micerium, Avegno; Genova, Italy), Saremco ELS (shade A2, Saremco Dental; St.Gallen, Switzerland), and Esthet-X HD (shade A2, Dentsply DeTrey; Konstanz, Germany) (Table 1). Specimen preparation for hardness measurements was performed as proposed by Rueggeberg and Craig[28] and shown in Fig 1.

Manufacture of Resin Composite Overlays and Underlays

Composite pastes were placed into cylindrical molds with a 10-mm inner diameter. Layering was carried out in five increments of ca. 2 mm each, which were individually light polymerized for 40 s (T-LED, Anthos; Imola, Italy) with a 1380 mW/cm^2 output. After removing the molds, the 10-mm-high composite cylinders were subjected to an additional cycle of polymerization in a composite oven at 70°C for 10 min (Bulb PlusT; Micerium).[7,8] In order to obtain disks with perfectly flat and parallel circular bases, cured cylinders were secured on the arm of a Micromet M machine (Remet, Casalecchio di Reno; Bologna, Italy) and subjected to consecutive cuts perpendicular to their long axis. The distance between the consecutive cuts was regulated to produce cylinders with 6 different thicknesses: 2 mm, 3 mm, 3.5 mm, 4 mm, 5 mm, and 6 mm. The thickness of each cylinder was controlled using a digital caliper (series 500 caliper, Mitutoyo America; Aurora, IL, USA) with an accuracy of 0.01 mm.

For each of the 3 resin composites under investigation, 90 cylinders to be used as overlays were produced, 15 for each different thickness, resulting in a total of 270 cylinders. Moreover, further cylinders having a fixed thickness of 2 mm were similarly prepared and served as underlays (Fig 1).

Specimen Production for Experimental Groups

One surface of each overlay was subjected to airborne particle abrasion with 50-µm Al_2O_3 (Korox, Bego; Bremen, Germany) using an intraoral air-abrasion device (Dento-Prep, Micerium). The tip of the microetcher was kept 5 cm away from the surface and applied for 10 s at 2.0 bar pressure.[7] The surfaces were then water rinsed and thoroughly dried. A layer of bonding agent was applied with a microbrush to the sandblasted surfaces and gently dried to evaporate the solvent. The adhesive systems used were: EnaBond (Micerium) for Enamel Plus HRI specimens, James-2 (Saremco Dental AG) for Saremco ELS specimens, and XP Bond (Dentsply DeTrey) for Esthet-X HD specimens. The materials used are summarized in Table 1. A standardized amount of composite paste was placed between the underlay and the overlay bonding surface under low lighting conditions to inhibit photoactivation. A 0.5-mm-thick metal ring was kept between the underlay and the overlay in order to keep them at a fixed distance, thus ensuring a standard thickness of the composite

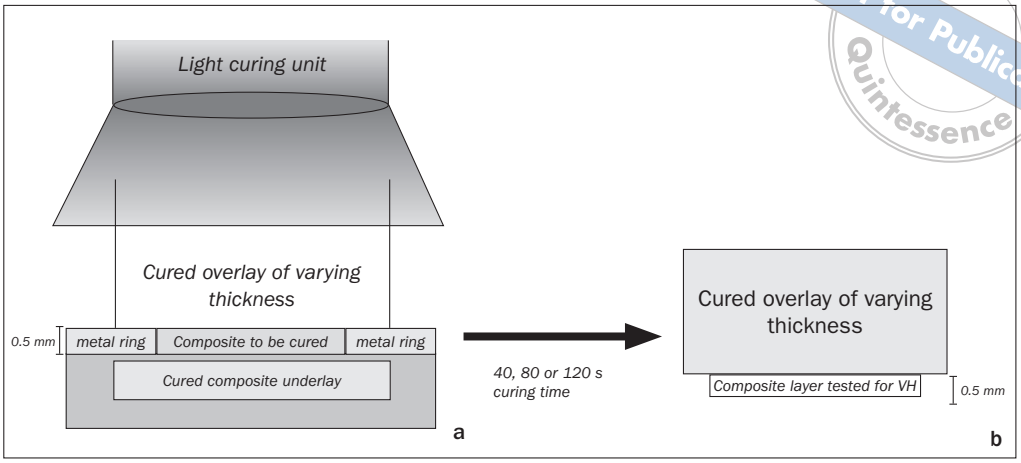

Fig 1 Cross section of specimen preparation (a) and final specimen shape (b) showing the 0.5-mm-thick resin layer to be subjected to Vickers Hardness test.

Table 1 Summary of the materials used in experimental groups

Groups	Bonding agent	Resin composite (Batch no.)	Resin matrix	Filler	Content % (w/v)	Manufacturer
ENA	EnaBond Light curing	Enamel Plus HRI (2009000112)	Bis-GMA, UDMA, TEG-DMA	Glass filler, SiO_2	75/53	Micerium, Avegno; Genova, Italy
SAR	James-2	Saremco ELS (06.2013-72)	Bis-GMA, bis-EMA, IBMA	Ba-Al-B-Si glass, Ba glass	77/60	Saremco Dental; St.Gallen, Switzerland
EST-X	XP Bond	Esthet-X HD (0410085)	Bis-GMA, bis-EMA, TEG-DMA	Ba-F-Al-B-Si glass, SiO_2	74/<50	Dentsply DeTrey; Konstanz, Germany

to be cured. A 0.05-mm-thick transparent polyethylene strip between the underlay and the uncured composite was used to avoid bonding at this interface. For each specimen, the 0.5-mm composite layer to be cured, the cured overlay, and the underlay were made out of the same batch of resin composite. The 270 specimens – assigned to three experimental groups on the basis of the resin composite used (Enamel Plus HRI: ENA group; Saremco ELS: SAR group; Esthet-X HD: EST-X group) and to subgroups on the basis of the overlay thickness (2, 3, 3.5, 4, 5, and 6 mm) – were divided again into further subgroups on the basis of the light-curing time, ie, 40, 80, or 120 s (T-LED, Anthos SRL), resulting in a total of 54 experimental subgroups composed of 5 specimens each (n = 5). Moreover, 9 control groups (n = 5), equally divided by the three materials and the three curing times, were added using microscopic slides to simulate completely transparent overlays. Polymerization was performed by placing the curing unit tip in direct contact with the overlay's upper surface. In this way, the underlays served as reflective material, while the cured overlays of varying thickness were used to control the amount of light reaching the composite to be cured and hence its degree of conversion. The obtained specimens, the shape of which is depicted in Fig 1, were stored at room temperature in black film canisters for at least 24 h before subsequent procedures.

Vicker's Hardness Measurement

Vicker's hardness (VH) readings were recorded on the free surface of the 0.5-mm-thick composite layer that was light-cured through the overlay (Fig 1). For each specimen, the mean value of three VH readings performed at approximately 2 mm distance from one another was used as the raw datum. Vicker's indentations were produced by applying a 10-N load for 10 s using a universal testing machine with a 500-N load cell (Lloyd LR 30K, Lloyd Instruments; Fareham, UK) provided with a standard 136-degree Vicker's diamond indenter (item #17, Affri, Induno Olona; Varese, Italy). Scanning electron microphotographs (EVO 50 XVP LaB6, Carl Zeiss; Cambridge, UK) were taken at different magnifications in order to measure the linear extent of the indentation diagonals (Figs 2 to 4). Subsequently, VH numbers were calculated according to the following formula:

$$VH = (1.854 \times F)/[(d1+d2)/2]^2,$$

where d1 and d2 are the measured diagonals (mm) and F is the predetermined applied load expressed in kilograms-force (1.0204 Kg).

Statistical Analysis

Data were arranged on the basis of the three factors under investigation (material, overlay thickness, and curing time). Means and standard deviations were calculated.

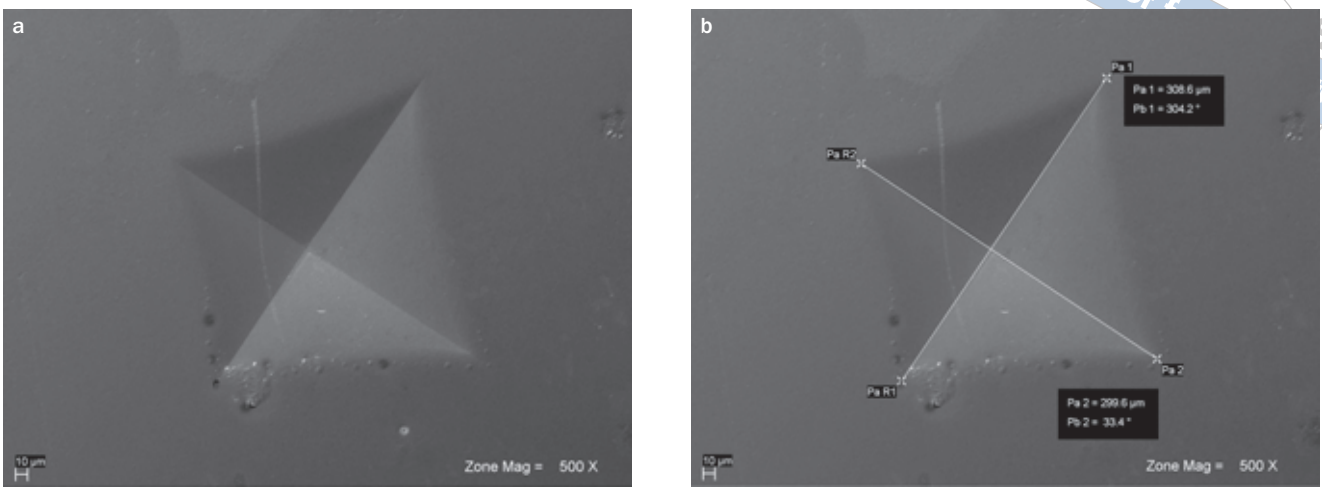

Fig 2 Scanning electron micrograph showing a VH indentation (a) and the measurement of its diagonals (b) on one specimen from the SAR group, 3 mm thick and cured for 80 s.

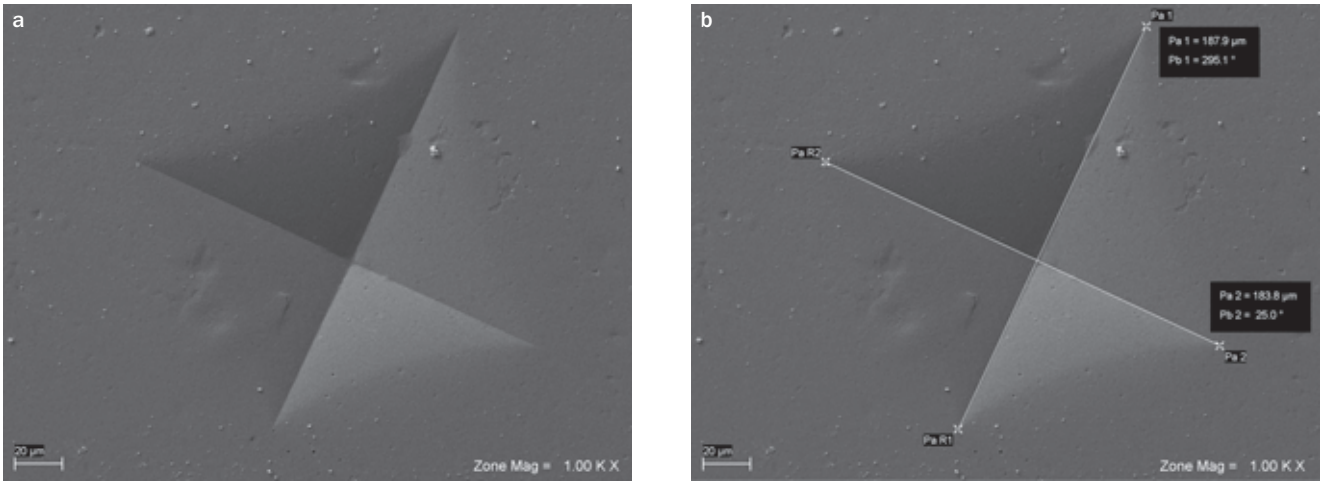

Fig 3 Scanning electron micrograph showing the VH indentation (a) and the measurement of its diagonals (b) on one specimen from the ENA group, 2 mm thick and cured for 80 s.

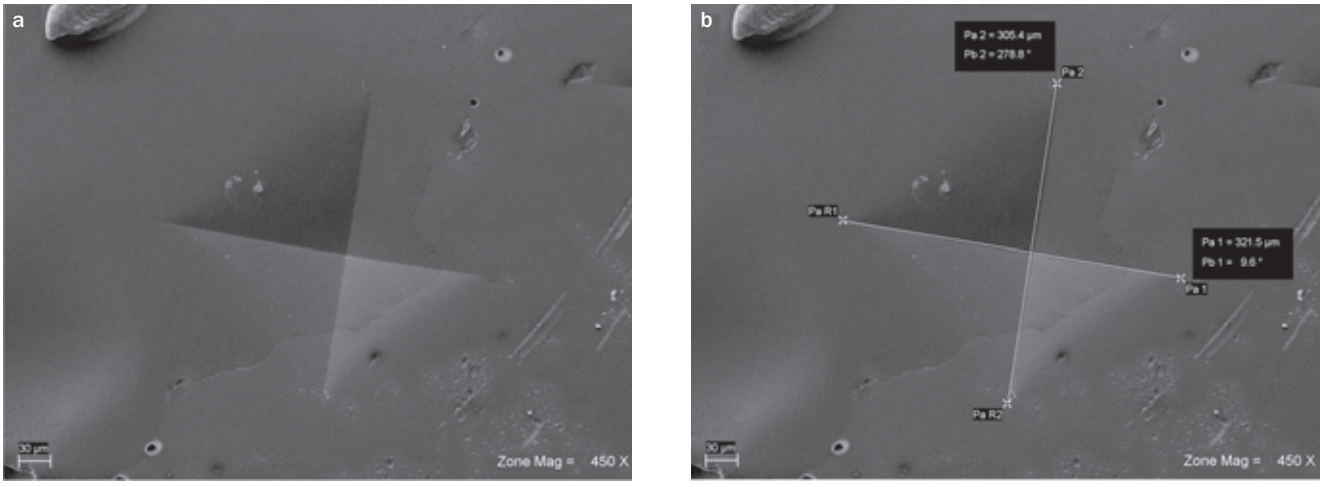

Fig 4 Scanning electron micrograph showing the VH indentation (a) and the measurement of its diagonals (b) on one specimen from the EST-X group, 3.5 mm thick and cured for 80 s.

Table 2 Mean VH numbers and standard deviations (SD) for experimental groups

ENA		Thickness							
		Control	2 mm	3 mm	3.5 mm	4 mm	5 mm	6 mm	
Curing Time	40 s	66.21^{a1}	**54.13**^{b2}	27.40^{c2}	24.32^{c2}	10.95^{d2}	1.84^{e3}	——	Mean
		(4.20)	(1.98)	(5.19)	(1.17)	(1.98)	(0.51)	——	(SD)
	80 s	66.32^{a1}	**59.47**^{b1}	**47.53**^{c1}	**39.32**^{d1}	22.35^{e1}	6.39^{f2}	——	Mean
		(2.07)	(0.49)	(2.98)	(0.46)	(3.06)	(3.22)	——	(SD)
	120 s	67.96^{a1}	**61.41**^{b1}	**48.23**^{c1}	**41.75**^{d1}	23.37^{e1}	13.77^{f1}	——	Mean
		(1.83)	(4.30)	(1.66)	(8.29)	(3.54)	(5.94)	——	(SD)
SAR		Thickness							
		Control	2 mm	3 mm	3.5 mm	4 mm	5 mm	6 mm	
Curing Time	40 s	42.88^{a1}	16.51^{b3}	9.62^{c2}	6.18^{c3}	1.02^{d3}	——	——	Mean
		(0.54)	(3.33)	(0.96)	(0.93)	(0.44)	——	——	(SD)
	80 s	43.90^{a1}	**29.78**^{b2}	19.59^{c1}	12.06^{d2}	9.64^{d2}	0.66^{e1}	——	Mean
		(1.32)	(5.66)	(2.44)	(1.97)	(1.14)	(0.12)	——	(SD)
	120 s	44.44^{a1}	**39.81**^{a1}	20.35^{b1}	16.91^{b,c1}	14.57^{c1}	1.38^{d1}	——	Mean
		(0.94)	(0.48)	(2.19)	(0.39)	(2.38)	(0.53)	——	(SD)
EST-X		Thickness							
		Control	2 mm	3 mm	3.5 mm	4 mm	5 mm	6 mm	
Curing Time	40 s	58.04^{a1}	17.66^{b3}	14.02^{b2}	5.20^{c2}	2.47^{c2}	——	——	Mean
		(4.16)	(9.15)	(0.53)	(0.26)	(0.09)	——	——	(SD)
	80 s	60.56^{a1}	**44.36**^{b2}	**36.03**^{c1}	16.73^{d1}	10.33^{e1}	0.57^{f1}	——	Mean
		(5.43)	(1.36)	(4.01)	(3.19)	(1.84)	(0.05)	——	(SD)
	120 s	61.32^{a1}	**49.93**^{b1}	**36.20**^{c1}	16.27^{d1}	11.52^{e1}	5.61^{f1}	——	Mean
		(1.50)	(4.56)	(5.16)	(0.86)	(1.27)	(0.31)	——	(SD)

Means and standard deviations for Vickers Hardness numbers obtained in the experimental groups and arranged on the basis of the material employed, the interposed composite disk thickness, and the curing time. Same superscript lower-case letters indicate no statistically significant differences among the levels of the factor thickness (reading horizontally). Different superscript numbers indicate significant differences among the levels of curing time (reading vertically). Mean values in bold were at least 55% of the respective control group.

The effect of the three factors on the mean VH values was analyzed using a three-way ANOVA. Multiple comparisons were performed according to the Holm-Sidak method. Values of $p < 0.05$ were considered to be statistically significant in all tests. Silikas et al[30] suggested that degree of conversion values below 55% might be contraindicated. As a consequence, for each different tested material and curing time, VH values below 55% of the respective control group were not considered clinically suitable.

RESULTS

Three-way ANOVA showed that mean VH values were statistically influenced by the material, the overlay thickness, and the curing time ($p < 0.05$). Moreover, all the interactions among the three factors were statistically significant ($p < 0.05$). Mean VH values, standard deviations, and the Holm-Sidak test results are summarized in Table 2 and in Fig 5.

In the ENA group, clinically acceptable hardness values (at least 55% of the control) were achieved after 120 s or 80 s curing time using 3.5-mm-thick (VH 41.75 and 39.32, respectively) or thinner overlays, and after 40 s using a 2-mm overlay (VH 54.13). In the SAR group, acceptable hardness values were only achieved after a 120-s or 80-s curing time using 2-mm-thick overlays (VH 39.81 and 29.78, respectively). In the EST-X group, acceptable results were only achieved using 3-mm or thinner overlays, after curing times of 120 s or 80 s.

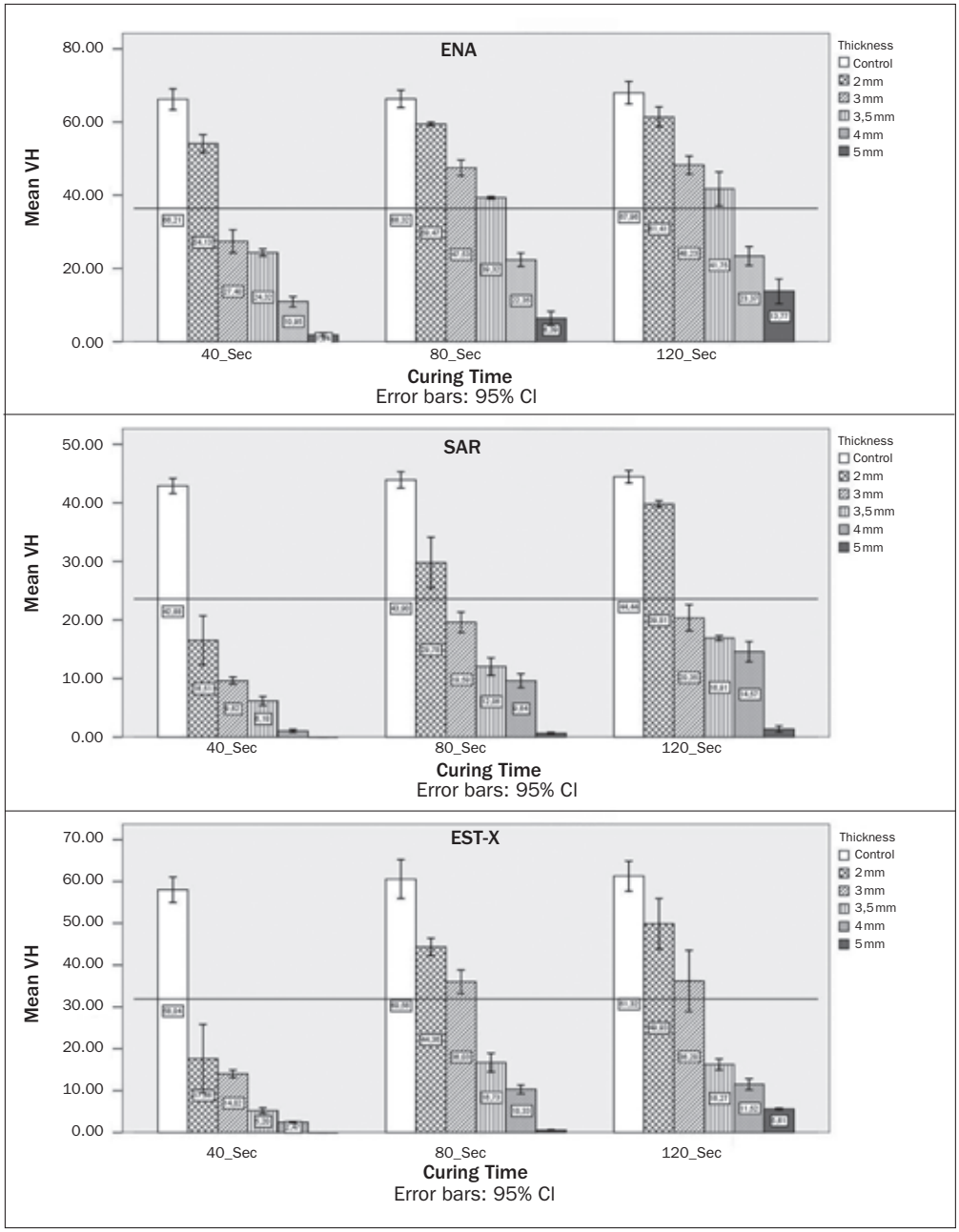

Fig 5 Bar charts summarizing mean Vicker's Hardness (VH) values and 95% confidence intervals obtained for the three materials under investigation (ENA, SAR, EST-X), using different thickness overlays and after 40, 80 or 120 s curing time. The horizontal lines indicate 55% of each respective control group VH value, used as acceptability threshold for each different material.

Measurable VH readings were not performed in 6-mm-thick overlay subgroups, as indentation diagonals were not clearly detectable on the most undercured specimens. A similar finding was also observed in SAR and EST-X 5-mm overlay subgroups subjected to a 40 s curing time.

DISCUSSION

The results of the present study confirmed that resin composite overlays can significantly disperse the light from the curing unit when it passes through, even for thicknesses that may commonly occur clinically. As a consequence, adequate light polymerization of all portions of the light-cured cement does not always appear to be possible. Hence, the clinician should carefully keep this factor under control by modifying the cavity shape with a direct resin composite buildup. The influence of the restoration thickness on the material's degree of conversion was previously investigated by Musanje and Darvell,[19] who showed a dramatic attenuation in irradiance with increasing specimen thickness. This attenuation is expected to be dependent in part on filler volume fraction, particle size distribution, particle

shape, refractive index difference (compared with the matrix) of the resin composite material, and in part on the decrease in light intensity with increasing distance from the composite surface.[1,2]

Based on the obtained data, the null hypothesis must be rejected. In all tested groups, significant differences in the microhardness of resin composites used as a luting agent were observed for the different curing times employed. Longer curing times (at least 80 s) led to an improved degree of conversion of the luting agent, even if no differences concerning the clinical suitability of the polymerization extent were recorded between 80-s and 120-s subgroups.

Different VH results were also observed when corresponding shades from different manufacturers (Enamel Plus HRi UD2, Saremco ELS A2 and Esthet-X HD A2) were used as overlay and luting agent, suggesting that the inherent translucency of the chosen material should be accurately considered. Regardless of the curing time, in the ENA group, a proper polymerization of the luting agent (55% of the control, as suggested by Silikas et al[30]) was achieved with overlay thicknesses ranging between 3.5 and 2 mm; in the EST-X group, overlay thicknesses above 3 mm resulted in an inadequate degree of conversion; in the SAR group, just the 2 mm overlay led to a proper polymerization. As a consequence, when dealing with thick and less translucent indirect composite restorations, a dual-curing cement seems more suitable; in contrast, translucent materials used at the appropriate thickness seem to allow the clinician to employ the same material for the build-up procedures, indirect restoration manufacture, and cementation. This is particularly important at the margins between tooth and restoration. Marginal adaptation of indirect composites before cementation has been reported to range widely, between 20 and 718 μm.[22-25] As a consequence, the material that fills this gap should preferably have mechanical properties (eg, wear resistance) comparable to those of the restoration. To obtain this, a filler content of at least 70%, as in microhybrid composites, would be necessary, whilst common dual-curing cements have a reduced filler content and mechanical properties similar to those of flowable resins. Moreover, with light-curing composites employed as luting agents, the necessary working time for seating the restoration and removing the excess cement may be extended at the discretion of the clinician, overcoming the relatively restricted working time imposed by self-curing cements.

In this study, an indirect method such as hardness testing was used to evaluate curing effectiveness. Various methods exist to assess the degree of conversion of resin composites. Direct techniques include Fourier transform infrared spectroscopy (FTIR) or Raman spectroscopy, which allow a direct measure of differences in the degree of C=C conversion.[13,28] However, these techniques are time consuming, complex[6,28] and, according to some authors,[6,33] more qualitative than quantitative in nature. In contrast, indirect methods are relatively easy to perform.[6,20] Moreover, it has been shown that the hardness tests were more sensitive than FTIR to detect small changes in degree of conversion after the network was cross linked.[29] Rode et al[27] showed a high correlation between resin composite microhardness and degree of conversion. Thus, hardness measurements are widely used to evaluate resin composite cure and provide a good estimate of the degree of conversion of composite materials.[11,16,32]

Ilie et al[14] showed that at 2 mm depth, an increase in polymerization time increased all the measured properties, including hardness, modulus of elasticity, creep, and elastic-plastic deformation. Moreover, mechanical properties varied strongly as a function of the spatial regions and a significant correlation was found between all of them. In a recent study, Bhamra et al[3] evaluated hardness and flexural properties of four commercially available resin composite restorative materials subjected to different light-curing procedures using irradiation times recommended by manufacturers: no detectable effect on strength or stiffness was recorded, although indentation hardness showed clearly that while the top surface was always adequately cured, the bottom surface at the recommended maximum increment thickness was not. A bottom hardness that is 80% of the top hardness value is generally considered adequate for a 2-mm increment.[10]

However, in the present study, to imitate the clinical procedure of luting an indirect restoration, irradiation of the composite layer under investigation was performed through a heat-cured overlay. This study design did not allow the measurement of any top hardness value to be used as reference. Silikas et al[30] suggested that degree of conversion values below 55% might be contraindicated. As a consequence, although degree of conversion and hardness are not identical, as they are two different properties that can just be related one to the other, VH values below 55% of the respective control group were not considered clinically suitable in this study.

Several types of light-curing unit (LCU) are currently used for photopolymerization of light-activated composite resins: conventional quartz-tungsten-halogen (QTH), light-emitting diode (LED), plasma arc, or laser. The most commonly used are the QTH and LED light.[24] Ceballos et al[6] concluded that the type of light-curing unit (LED vs QTH halogen) had no influence on curing effectiveness; instead, microhardness results were significantly affected by the interaction between the type of curing light and exposure time and also by the interaction between the type of curing light and depth. However, using an old dilapidated QTH LCU, light-activated dental materials may be less effectively polymerized, resulting in poorer physical properties and an increased risk of premature failure of restorations, assuming no compensation for decreased LCU irradiance.[31] Cavalcante et al[5] showed that the exposure time required to stabilize hardness values of resin composites is affected not only by resin composite formulation but also by the type of LCU. This probably indicates that actual power density and degree of collimation of the LCU used are other important variables to be investigated. In the present study, these variables were not considered, since the same commercially available, new-model LED LCU was used for all specimens.

CONCLUSION

Within the limitations of the present in vitro study, VH values (and thus degree of conversion) of the tested resin composites used as luting agents were affected by the curing time, indirect restoration thickness, and the inherent translucency of the overlay material. With at least 80 s of curing time, a 3.5-mm thickness limit should not be exceeded even when using translucent materials, if a light-curing composite must be used for cementation. For luting thicker and more opaque indirect restorations, a dual-curing luting agent is suggested.

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Clinical relevance: When light curing composites are employed as luting agents, the clinician should be able to keep factors like indirect restoration thickness/translucency and curing time under strict control. Otherwise, a dual-curing resin cement should be preferred, accepting some clinical limitations that this choice may entail and that were discussed in the present paper.