

INFLUENCE OF POST CURING HEAT AND PRESSURE ACTIVATION FOR RESIN COMPOSITE ON IT'S SURFACE HARDNESS

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ABSTRACT

This study was conducted to evaluate the influence of post curing heat and pressure activation of resin composite on it's surface hardness.

Materials and methods: A total of 30 disc specimens, 7mm in diameter and 2mm thick, were constructed from 3 different types of contemporary resin composite restoratives (10 each). For each restorative, half the numbers of the constructed specimens (5 discs) were served as directly cured composite, following their storage in water at $37\pm 1^{\circ}\text{C}$ for 24h. The remaining 5 specimens of each restorative were subject to post-curing heat and pressure in type B Lisa autoclave for 41 min using B-universal 121 cycle at 122.5°C and a pressure of 1.16 bar to simulate one simple approach of chair-side indirect composite polymerization. Vickers hardness numbers were determined using a micro-hardness tester (Wilson® *Hardness Tester, Model Tukon 1102, Buehler, Lake Bluff, IL, USA*) using a load of 50 gf for 15 seconds dwell time. The Vickers's hardness number (N/mm^2) was recorded as an average of six readings, three from each surface (top and bottom) for each specimen.

Results: ANOVA was used for the effect of additional heat activation and type of resin composite on it's surface hardness. It was found that the use of an additional heat activation and type of resin composite (matrix type and degree of loading) had a statistical significant effect on VHN of resin composite tested.

Conclusion: The post-curing heat and pressure improves the surface hardness of the tested resin composites.

INTRODUCTION

One of the most critical aspects of a resin composite restoration is the polymerization stage. Inadequate polymerization may result in a resin with inferior surface properties and more liable to staining and even poisonous to the dental pulp due to the presence of free monomers⁽¹⁻⁴⁾.

The photo-polymerization process of resin composites is initiated by absorbing light spectrum in a range of wavelength around 400 to 500 nm. Sufficient light activation transforms the resinous monomers into a complex polymeric structure. Full monomer conversion cannot be achieved, which results in free unreacted radicals⁽¹⁾.

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The degree of conversion of resin composite is determined by the number of carbon double bonds existing in the un-polymerized resin, which are converted into carbon single bond to form the polymeric chain during the polymerization process^(3,5). The degree at which monomers react to form the polymer during the polymerization reaction has a major effect on the physical and mechanical properties of resin composites⁽⁶⁻⁹⁾.

Due to comprehensive research and continuing manufacturers' developments, resin composite restorations have achieved wide popularity through the few last decades.⁽¹⁰⁾ The modifications of resin composite materials normally came in order to overcome the commonly experienced and the inherited disadvantages of those materials⁽¹¹⁾. The polymerization shrinkage stresses and their subsequent sequels were among those shortcomings and that is why many resin composites with minimal shrink formulae have been developed⁽¹²⁾. Most of the resin composite manufacturers introduced nano-sized fillers to their directly cured composite formulations. This approach basically changed the resin composites' matrix- filler ratio that had a positive effect on the materials' physical and mechanical properties⁽¹³⁾. Indirect curing of resin composite is also a well-known approach to improve their mechanical properties and overcome the subsequent effect of shrinkage stresses induced as a result of direct curing^(14,15).

Different means of additional extra-oral polymerization were proposed, including the use of photonic energy, dry heat, and even moist heat, were suggested in order to improve, and enable the use of direct-use resin composites in indirect restorations. This attempt was to extend the indication of resin composites and the clinical longevity of restorations^(16,18). The autoclave has been suggested as a moist heat source since it is basic equipment in the dental offices. Therefore, even at areas remote from dental laboratories, it would be beneficial to

develop simple technique that could allow the use of indirect restorations at lower costs.

Resin composite surface hardness is dependent primarily on their microstructure and composition, which is in a direct correlation with the degree of conversion of monomers^(19,20). The increase in hardness is related to a higher degree of monomer conversion and consequently higher mechanical properties of polymeric materials. Thus, the study of the microhardness of materials consists of an indirect method to evaluate the effect of different treatments on the properties of dental composites⁽²⁰⁻²²⁾. The use of Vickers hardness test as an indicator of improvement in materials' properties was proposed by different authors^(19,23-26). The aim of this work was to evaluate the effect of additional activation using an autoclave on microhardness of three commercially available direct resin composites.

MATERIALS AND METHODS

Specimen's preparation

A total of 30 disc shaped specimens, 7mm in diameter and 2mm thick, were constructed from 3 different types of contemporary resin composite restoratives (10 each), Filtek Z250 XT, 3M ESPE, St. Paul, MN (Z250, Group A); Filtek Z350, 3M ESPE (Z350, Group B) and Ceram X mono, Dentsply Detrey GmbH, Konstanz, Germany (Ceram X, Group C). The detailed description and the manufacturers of the materials used were listed in table (1).

A metal ring was specially constructed to hold a split Teflon ring 2mm thick with a central hole of 7 mm in diameter (Fig 1-a & b). Resin composites were packed; using gold-plated applicator, in the split Teflon ring then covered with thin glass slide to produce standardized smooth top and bottom surfaces of the specimens. A load of 400 gm was placed over the glass slide (Fig 1-c) to generate standardized packing force for 10s. Resin composite

was light cured for 20s using *LED* curing light having intensity of 1200 mW/cm² and wave length of 430-480 nm (*Elipar S10, 3M ESPE AG, Seefeld, Germany*) (Fig 1-d). After releasing the cured specimens out of the split ring, the margins of their bottom surfaces were lightly scratched with hand cutting instrument to produce groove landmarks to help identify the top and the bottom surfaces.

For each restorative, half the numbers of the constructed specimens (5 discs) were served as directly cured composite references (*Subgroup I*) following their storage in water at 37±1°C for 24h (*FUNCTION Line, Thermo Electronic Inc., Lagensbold, Germany*). The remaining 5 specimens of each restorative material (*Subgroup*

II) were subject to post-curing heat and pressure⁽²⁷⁾ in type B Lisa autoclave for 41 min (*W&H Sterilization Srl., Brusaporto (BG), Italy*) using B-universal 121 cycle at 122.5°C and a pressure of 1.16 bar to simulate one simple approach of chair-side indirect composite polymerization.

Hardness Test

Vickers hardness numbers were determined using a micro-hardness tester (*Wilson® Hardness Tester, Model Tukon 1102, Buehler, Lake Bluff, IL, USA*). The test was carried out by using a load of 50 gf for 15 seconds dwell time. The Vickers’s hardness number (N/mm²) was recorded as an average of six readings, three from each surface (top and bottom) for each specimen (Fig 2).

TABLE (1) Resin composites used.

Material/Manufacturer	Description	Composition	Lot. No.
Filtek Z250 XT 3M ESPE, St. Paul, MN	Nano-hybrid, Methacrylate-based resin composite	Matrix: bisphenol A glycol dimethacrylate (<i>BIS-GMA</i>); bisphenol A ethoxylated, methacrylate (<i>BIS-EMA</i>), urethane dimethacrylate (<i>UDMA</i>), Triethylene glycol dimethacrylate (<i>TEGDMA</i>) and Polyethylene glycol dimethacrylate (<i>PEGDMA</i>). Fillers: 1. Surface-modified zirconia/silica with particle size of 0.1 - 10 microns (median approximately 3 microns or less). 2. Non-agglomerated/non-aggregated 20 nanometer surface-modified silica particles. The filler loading is 81.8% by weight (67.8% by volume).	N560782
Filtek Z350 XT restorative 3M ESPE, St. Paul, MN	Nano-filled, methacrylate-based resin composite	Matrix: BIS-GMA, UDMA, BIS-EMA, PEGDMA and TEGDMA resins. Fillers: combination of non-agglomerated/non-aggregated 20 nm silica filler, non-agglomerated/non-aggregated 4 to 11 nm zirconia filler, and aggregated zirconia/silica cluster filler (comprised of 20 nm silica and 4 to 11 nm zirconia particles). The inorganic filler loading is about 72.5-87.5wt%.	N134165
Ceram.X mono Dentsply Detrey GmbH, Konstanz, Germany	Nano-ceramic, methacrylate-based resin composite	Matrix: Methacrylate resins, ethyl-4 (dimethylamino) benzoate Fillers: Methacrylate-modified polysiloxane (organically modified ceramic), Barium-aluminum-borosilicate glass, silicone dioxide nano-fillers Others: UV stabilizer, stabilizer, Camphroquinone, fluorescent pigment, iron oxide pigment, titanium oxide pigment and aluminum sulfo-silicate pigments.	1406000345

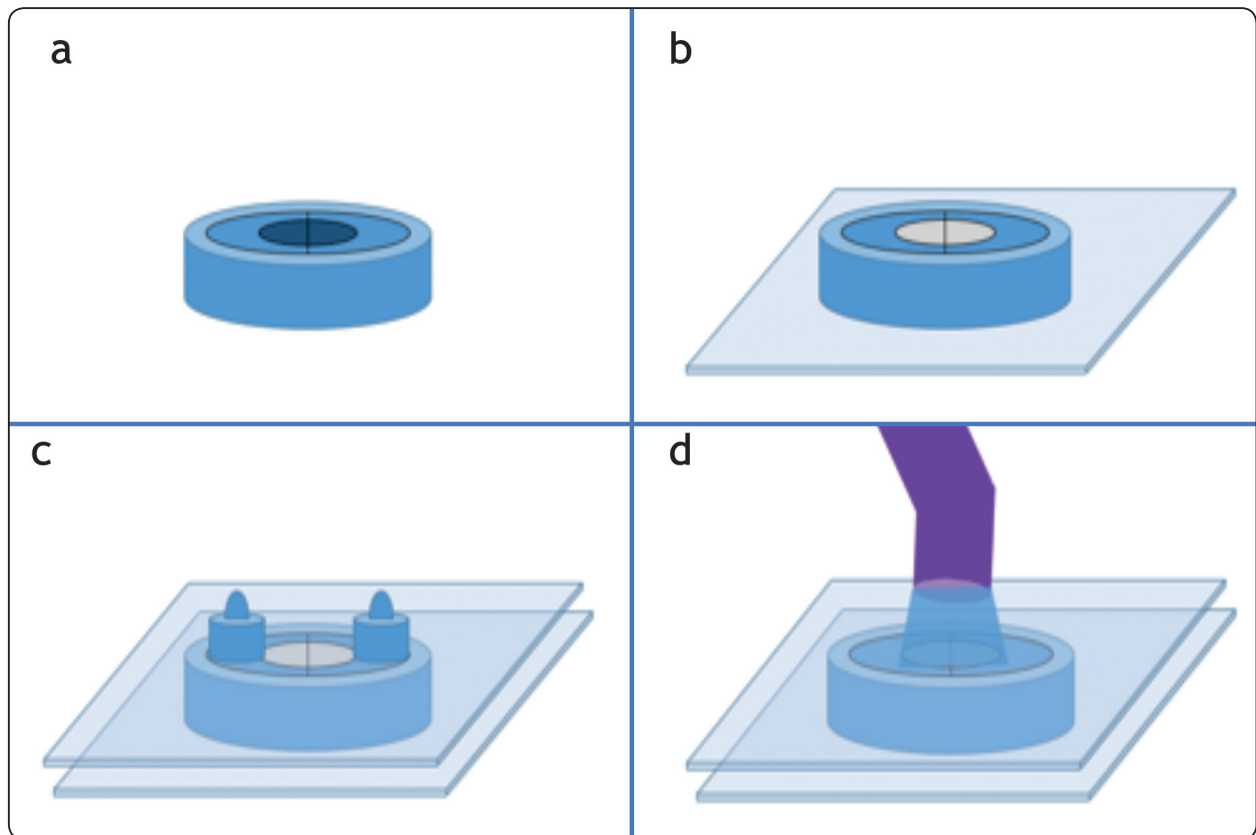


Fig. (1) Schematic diagram showing the construction of composite specimens: (a) Positioning of the split Teflon ring in the metal ring, (b) Positioning of a glass plate under the assembled split Teflon and metal rings, (c) after packing of resin composite another glass plate was positioned on top of the assembled split Teflon and metal rings with application of constant weight and (d) Light curing of specimens.

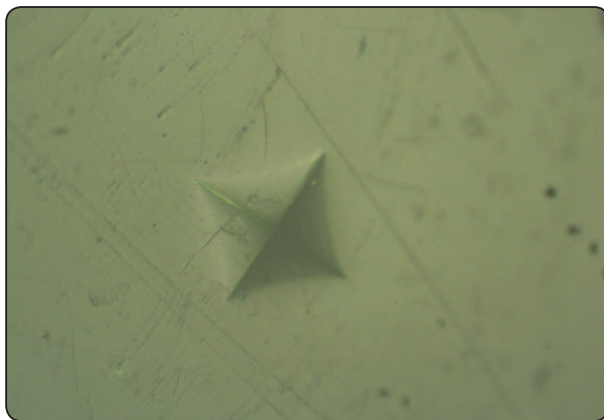


Fig. (2) Indentation on surface of specimens

Statistical analysis

The data obtained from the means were subjected to analysis of variance (ANOVA) followed by Tukey's test at a 5% level of significance.

RESULTS

The VHN mean values and standard deviations of tested groups are presented in (Table 2) and fig 3. Two-way ANOVA was used to test the two main effects namely, the types of resin composites and the mode of curing. The first main effect (types of resin composite) had three levels namely, Z250, Z350 and Ceram X. The second main effect (modes of curing) had two levels namely, direct curing and post curing heat and pressure. Two-way ANOVA

Table (2) The VHN, mean values, standard deviations and significance of tested resin composite.

Variables	Filtek Z250 XT		Filtek Z350 XT		Ceram X mono	
	Mean	St. Dev.	Mean	St. Dev.	Mean	St. Dev.
Direct curing	77.7400 ^(b)	0.63482	77.1000 ^(b)	0.56125	56.8000 ^(d)	3.02738
Post-curing heat and pressure	91.3000 ^(a)	1.52807	89.3600 ^(a)	1.16533	69.5960 ^(c)	2.20660

The same superscript capital letters in the first left column indicates insignificant difference between composite types

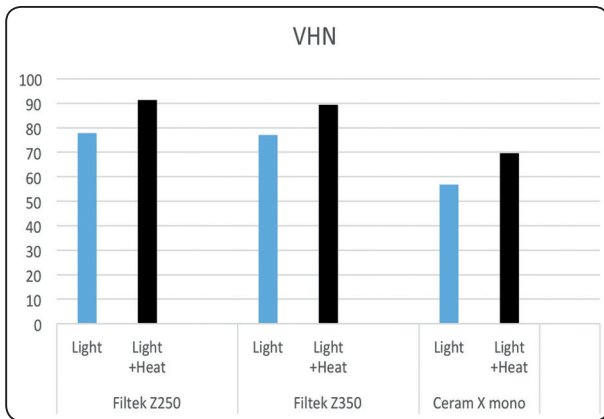


Fig. (3) The VHN of tested resin composite.

revealed a statistical significant difference in the two main effects, the types of resin composites ($P=0.0001$) and modes of curing ($P=0.0001$). There was no statistical significant interaction between the two main effects ($P=0.71$). Post-hoc Tukey’s test at $\alpha = 0.05$ revealed that VHN mean values of Ceram X mono was significantly lower than that of Filtek Z350 ($P= 0.001$) and Filtek Z250 XT ($P= 0.001$), while no significant difference was revealed between Filtek Z350 and Filtek Z250 XT ($P= 0.113$).

DISCUSSION

The results of the current study indicate that the use of an additional activation method significantly improved the micro-hardness of the tested resin composites. These results are consistent with other studies, which reported that photo-activated polymerization is never capable of achieving 100% monomer conversion in resin composite

mass ^(28,34,35). Yet the degree of conversion can be boosted by subjecting resin composite to a temperature of 125°C ^(19,29, 34-39).

All three materials tested in the current study showed a significant increase in VHN mean values when subjected to post curing heat and pressure. Such finding could be attributed to the thermal energy and pressure delivered by the process increased the degree of conversion. Others, who tested the effect of heat treatment on the degree of conversion of resin composite, also observed an improvement the mechanical properties of the tested materials, which may be due to exhaustion of residual double carbon bonds in the polymer ⁽³⁰⁻³³⁾.

Trujillo et al (2004) ⁽³⁴⁾ stated that additional heat treatment of resin composite, significantly improve the kinetics of activation, and increases the rate of conversion of resin composites improving their physical properties ⁽³⁵⁾.

The increase in VHN can be explained by the fact that the thermal energy used caused a rise in temperature close to the glass transition temperature, which increases the kinetic energy of the resinous monomers and the amount of free unreacted radicals. The greater mobility within the polymer chain triggered new reactions of the activated radicals causing a greater number of crosslinks in the organic matrix. The continuation of the activation process leads to higher degree of conversion, greater stability and hardness of the resin composite ^(16-17, 35).

Another benefit is the enhanced biocompatibility, as a result of a significant decrease in the amount of unpolymerized resin and, accordingly, decreased levels of leachable monomers that promote oral cytotoxicity⁽¹⁹⁾. The use of heat also causes an increase in crosslinking of monomers and evaporation of the residual monomers^(34,39).

The effect of additional activation and the different VHN of the resin composites is affected mainly by material composition and the degree of filler loading. Bis-GMA has a low degree of conversion because of its high molecular weight, high viscosity and low flexibility. By adding a low molecular weight monomer (EGDMA or TEGDMA) to Bis-GMA the mobility and conversion rate is boosted. Also UDMA is considered a substitute to Bis-GMA, due the similarity in molecular weight but difference in viscosity^(30,37).

It has been shown that although the monomer TEGDMA assist in forming a thicker polymeric chain, yet it is the most flexible and with a greater rate of water absorption. Bis-GMA forms a more rigid chain but absorbs less water; however, it absorbs more water than the UDMA/Bis-EMA combination. Hydrolysis of intermolecular bonds weakens the polymer. In UDMA based composites, hydrogen bonds increase the rate of conversion and improve mechanical properties. Upon replacing TEGDMA by UDMA and/or Bis-EMA (during co-polymerization with Bis-GMA), the absorption of water is decreased. Such characteristics influence the rate of conversion and the mechanical properties of resin composites⁽²⁷⁾. The above mentioned information explains the different behaviors between different resin composites after additional activation: Filtek Z250 XT & Z350 XT contains nearly the same organic matrix Bis-GMA, UDMA, TEGDMA, Bis-EMA and PEGDMA, while Ceram.X mono contains Methacrylate resins, and ethyl-4(dimethylamino) benzoate.

In the above discussion, it was reported that it was possible to say that additional activation

by thermal treatments was found to improve the hardness of resin composite tested irrespective of their composition. Autoclaving was found to be a very effective method of activation and gives better results. Putting in consideration that the autoclave is routinely found as equipment at dental offices therefore it is possible to develop a simple, low cost technique for production of indirect restoration especially in cases of difficult access to special equipment.

CONCLUSION

Under the limitation of the current study, it could be concluded that post curing activation using heat and pressure has a positive influence on the surface hardness of the resin composites

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