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Original Article

The effect of microwave/laboratory light source postcuring technique and wet-aging on microhardness of composite resin

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ABSTRACT

Background: Although composite restorations are really valuable for esthetic zones, they have shown less longevity rather than amalgam restorations. Since it may be related to the method used for curing the composite, postcuring could increase the degree of conversion and result in more long-lasting composite restorations. This study was planned to evaluate the effect of two different postcuring techniques on microhardness of indirect composite resin after wet-aging and comparing them with the direct type.
Materials and Methods: In this experimental study, 99 composite disk-shaped (6.5 × 2.5 mm) specimens of composite (Gradia GC, Japan) were prepared in split mold. The indirect composite specimens were postcured by laboratory light source (Labolite LV-III GC Corp, Japan) or microwave unit (MC 2002 JR, LG, Korea). Then, the aging procedure was done for 24 h, 30 and 180 days in distilled water. The Vicker's Hardness test (VHN) on surface of specmens was measured by Wolpert microhardness tester and the data were analyzed by the two-way analysis of variance (ANOVA) and Tukey's *post hoc* tests. ($P \leq 0.05$).
Results: The statistical analysis revealed that surface microhardness of postcured composite by microwave and laboratory light source was more than that of direct composite ($P = 0.0001$) and postcuring by microwave was more effective than postcuring by laboratory light source ($P = 0.004$). The 30 days stored composite demonstrated significant decrease of VHN compared with the 24-h stored samples ($P = 0.0001$), with a more significant VHN decrease after 180 days of aging ($P = 0.045$).
Conclusion: Postcuring increased the surface microhardness and aging reduced the surface microhardness of indirect composite.

Key Words: Composite resin, laboratory light source, labolite, microhardness, microwave, postcure, wet-aging

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INTRODUCTION

The introduction of indirect composite resins has primarily made to compensate for the drawbacks of direct composite resins. Among the proposed advantages are the potential for achieving positive interproximal contacts, less polymerization shrinkage, and better marginal sealing because of the polymerization process that takes place in a laboratory setting.[1]

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Factors which directly affect the physical and mechanical properties of composite resins are chemical composition of the materials, the organic or inorganic portion, type, morphology, and filler content.[2,3]

Thickness of the increment inserted into the cavity, intensity, and irradiation time, light spectrum and distance of the tip of the light-curing unit are important factors which affect the polymerization pattern,[4] exposing the composite and adhesive resin to different media and material affect the stability of resin and make them more vulnerable to degradation.[5,6]

In order to improve the mechanical and physical properties of the composites, including hardness, elastic modulus, flexural strength, stiffness, hygroscopic expansion, and solubility, extraoral cure (postcure) is suggested. Extraoral treatment happening under light, heat, and pressure is shown to increase the degree of conversion of dental composites.[7]

The findings have supported the alternative curing methods, such as ultraviolet radiation curing, microwave curing, electron-beam curing, and infrared curing methods. They have demonstrated the superiority of microwave curing over the thermal curing.[8]

Since, restorative materials are exposed to saliva in oral cavity, some researches focus on how aging or water storage affects mechanical properties of direct and indirect composites. The reason behind the chemical degradation of composite resins is mainly because of the diffusion of molecules and ions of nonreacted resin monomers. In fact, once composite resins are put into solutions, the polymer chains will absorb the water, contributing to a swelling which may in turn decrease the bond strength of the polymer chains.[9]

The quality and stability of the silane coupling agent are important in minimizing the deterioration of the bond between the filler and polymer and the amount of water sorption.[10]

Some researches focused on the effects of postcuring (using autoclave and microwave for postcure treatment[7] and postirradiation dry aging at different periods of time[11] that resulted in increased microhardness of composite resin). Some articles studied the effect of water storage (storing specimens in distilled water for 24 h and 30 days that caused decreasing the amount of flexural strength[9] and wet-aging of denture base polymers reinforced with short glass fibers that showed less water sorption and solubility of the reinforced denture base polymer[12]) on physical properties of composite resins, while the objective of this study was to evaluate the influence of two postcuring methods and different water storage periods on the microhardness (VHN) strength of direct and indirect composite resins. Since, the present study focused on the effect of postcuring and wet-aging on microhardness of composite resin the evaluation of postcuring effect was considered by using both the direct and indirect composite resins.

MATERIALS AND METHODS

A total of 99 specimens were fabricated as disks in brass mould [(6.5 × 2.5 mm) [Figure 1]. Direct composite resin (Gradia GC/GC corporation, Tokyo, Japan) (*n* = 33) and indirect composite resin (Gradia GC/GC corporation, Tokyo, Japan) (*n* = 66) was inserted into split mold and then a transparent strip (Jr-Rand Corporation, New York, USA) and a glass slide was placed on top surface until the additional amount of composite was released. Although all the specimens were early cured (EC) by a halogen light-curing unit (400-420 mw/cm^2, Coltolux II, Coltene/Whaledent Inc., USA) for 40s on the top and bottom of each specimen. A total of 33 specimens of indirect composite samples were postcured (one by one) under irradiation of 12 fluorescent lamps in laboratory light source (Labolite LV-III GC Corp, Japan) which scattered light from three directions over specimen, a postcuring unit which is manufactured and recommended by GC corporation, Tokyo, Japan for their products, and 33 remainder specimens were postcured (one by one) under irradiation of microwaves with power of 540 W for 7 min in microwave unit (MC 2002 JR, LG, Korea). Then, all of the samples (*n* = 99) were polished (by the fine diamond bur and the silicon rubber) and were stored in distilled water 37° using incubator (Behdad, Tehran, Iran) and were divided to nine groups as follows:

Group 1: EC + 1 day water storage
Group 2: EC + 30 days water storage
Group 3: EC + 180 days water storage

Figure 1: Split mold

Group 4: EC + microwave 7 min + 1 day water storage
Group 5: EC + microwave 7 min + 30 days water storage
Group 6: EC + microwave 7 min + 180 days water storage
Group 7: EC + labolite 5 min + 1 day water storage
Group 8: EC + labolite 5 min + 30 days water storage
Group 9: EC + labolite 5 min + 180 days water storage

*EC = Early cured, *Labolite or labolight = A trade name for laboratory light source

The hardness tests VHN (which is reliable for testing microhardness of composite resins)[7,11] were counted via a Wolpert (500 gr/10 s, Wolpert, Darmstadt, Germany) microhardness tester using a Vickers diamond indenter. The indentations were performed under a 500 gr load on three random points on the top surface of each specimen. The dimensions of the indentations were measured using the measuring eyepiece of the microscope of the hardness tester, and hardness values were given from standard tables. The data of hardness were analyzed by two-way analysis of variance (ANOVA) and Tukey's *post hoc* test (*t*-test) ($P < 0.05$).

RESULTS

The statistical analysis two-way ANOVA revealed curing technique and aging time are two influential factor (P.V. ≤ 0.001). Curing technique and time affected each other mutually (P.V. ≤ 0.001). Direct composite selected as a control group in order to compare with indirect composite. T-test revealed type of composite could be an influential factor (P.V. ≤ 0.001). However, to ignore factor of composite type, two-way ANOVA and *post hoc* Tukey's test was used.

Two-way ANOVA and Tukey's *post hoc* test revealed high significant differences of hardness values among postcured indirect composite and EC direct composite ($P = 0.0001$), microwave curing technique was more effective than the other technique ($P = 0.004$) [Table 1]. Within this statistical analysis wet-aging decreases surface microhardness of direct and indirect composites: 24-h-stored specimens were significantly harder than 30-day-stored ($P = 0.0001$) and 180-day-stored ($P = 0.0001$), and microhardness of 180-day-stored specimens had been reduced by aging rather than 30-day-stored ($P = 0.045$) [Table 2]. According to Figure 2 and Table 3, the micro-hardness amount of specimens post-cured by microwave is more than labolite and early-cured groups. In addition, increasing of aging time reduced the micro-hardness.

DISCUSSION

Microhardness test has been chosen for this experiment because it is very common and reliable technique. Surface microhardness is believed to be a relevant factor indicating the mechanical strength of a resin and has a substantial correlation to the material's rigidity.[11]

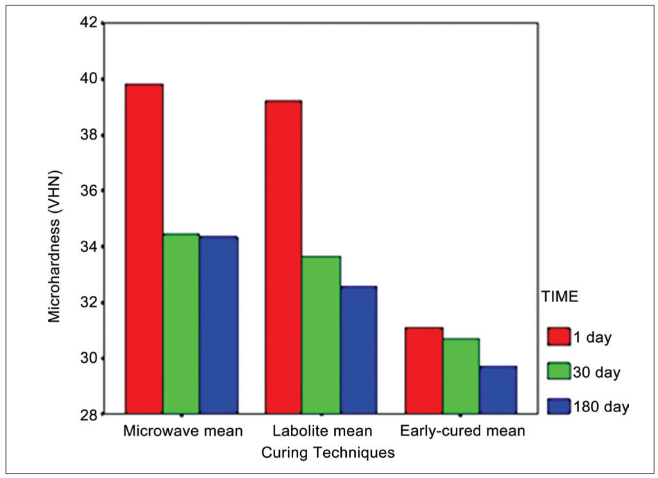

Figure 2: Microhardness (VHN) of direct and indirect composite resin after different aging time

Table 1: Comparison of microhardness (VHN) of all groups with different curing technique

Cure (I)	Cure (J)	Mean difference (I-J)	Std. error
Microwave	Labolite	1.0182 (*)	0.30894
	Early-cured	5.6818 (*)	0.30894
Labolite	Microwave	–1.0182 (*)	0.30894
	Early-cured	4.6636 (*)	0.30894
Early-cured	Microwave	–5.6818 (*)	0.30894
	Labolite	–4.6636 (*)	0.30894

*A trade name for laboratory light source, VHN: Vicker's hardness test

Table 2: Comparison of microhardness (VHN) of all groups with different aging time

Time (I)	Time (J)	Mean difference (I-J)	Std. error
1 day	30 day	3.7939 (*)	0.30894
	180 day	4.5424 (*)	0.30894
30 day	1 day	–3.7939 (*)	0.30894
	180 day	0.7485 (*)	0.30894
180 day	1 day	–4.5424 (*)	0.30894
	30 day	–0.7485 (*)	0.30894

VHN: Vicker's hardness test

Table 3: The microhardness (VHN) amount of each specimen measured after post-curing or early-curing (control group) and aging

S-N C-T, A-T	S1	S2	S3	S4	S5	S6	S7	S8	S9	S10	S11
E.C, 1-day	32.7	33	30.9	33.9	28.6	29.3	30.1	31.2	32	30.2	30.3
E.C, 30-days	32.3	28.1	31.2	29.9	30.1	32.5	32.2	31	30.1	30.3	30
E.C, 180-days	30.4	28.9	29.5	29.1	31.9	27.6	28.5	30	30.5	30.9	29
MIC, 1-day	40.6	38.2	38.5	42.5	40.2	39.9	39.7	38.1	39.4	40.9	39.8
MIC, 30-days	34	35.9	33.6	36	35.1	33.7	34.4	31.8	35.1	33.6	35.3
MIC, 180-days	34.5	32.8	35.9	35.7	33.4	34.2	34.2	34.9	34.6	33.1	34.1
LAB, 1-day	38.7	39.5	38	41.1	39.3	38.5	39.2	38.4	40	39.1	39.9
LAB, 30-day	33.5	34.1	34.3	32.8	34.7	34.1	32.6	31.5	35.5	34.2	33
LAB, 180-day	30	31.8	32.9	34.4	32.7	31.1	34.6	34	33.2	31.3	32.1

*S: Specimen; *S-N: Specimens number; *C-T: Curing type; *A-T: Aging time; *MIC: Microwave; *LAB: Labolite; *EC: Early cured; VHN: Vicker's hardness test

A number of indirect resin restorative system with highly improvements has been presented during the last years.[12] Currently, the main concern of modern restorative composites is focused on polymer matrix or fillers modifications to reduce polymerization shrinkage and stress as well as increasing the degree of monomers conversion and improving their general qualities.[11] In the present study, direct and indirect composite specimens, with different curing methods and different time of water storage, are compared and significant differences in the hardness are detected. Indirect composite showed a higher VHN than direct composite. These differences probably could be related to the effect of postcuring and filler content on improving hardness of the composite resins.

Particular postcuring methods have been promoted to increase the polymerization, such as curing in nitrogen atmosphere under heat and pressure or curing in inlay systems proposed by the manufacturer.[13]

In most restorative composite resins, only 45-70% monomer conversion occurs following the initial light curing process.[14] Postcuring above the glass transition temperature of the resin matrix can enhance the degree of cure and improve the mechanical properties of the composite.[15] Free-radical polymerization reactions usually occur with unsaturated molecules containing double bonds.[10]

Lombardo *et al.*,[16] reported that stove postcuring technique (dry) increases microhardness of composite resin, while autoclave had adverse effect on it, concluding that postcuring technique is very crucial. The postcuring process increases the microhardness, but the polymerization in the autoclave decrease microhardness. Hence, exposing to moisture during postcuring probably can decrease the microhardness of the resin.

In the present study exposing, the specimens to light (12 fluorescent lamp) in laboratory light source unit which is used in laboratory increased microhardness. We realized that microwave was more effective than high intensity light of laboratory light source unit in increasing the microhardness. Poskus *et al.*,[7] also reported that microwave is a useful technique for postcuring of composite. They stated that microwave postcuring technique was more effectual than oven and autoclave techniques to enhance marginal adaptation and increased microhardness of Filtek Z250 inlay restoration.

Sharafeddin and Ghahramani[17] study showed that postcuring by microwave and oven techniques increases microhardness of composites resin, which apparently confirms the present study results. Kumar *et al.*,[8] study also revealed the same results in which microwave curing is better than thermal curing.

In the present study, the 30-day-stored specimens illustrated a significant decrease in the surface microhardness rather than 24-h-stored specimens and 180-day-stored specimens revealed a significant decrease in the surface microhardness in comparison to 24-h and 30-day-stored specimens.

One of the characteristics of dental materials is water sorption. Materials which absorb water are swollen. Water sorption has irreversible weakening effect on the mechanical properties of composite.[18]

Therefore, the major cause of decrease in surface microhardness of the composites after water storage may be water sorption. When composite resin is immersed in water, two phenomena occur. There is a quick separation of uncured monomer or oligomers which presumably completes within several days.[19] Water sorption is a slow process comparing to polymerization shrinkage and stress development.

The hygroscopic expansion phenomenon starts 15 min after the initial polymerization. Most resins entailed 7 days to reach stability and about 4 days to show the preponderance of their expansion.[10]

Concurrently, water is diffused in to the polymer matrix of composites, swells the polymer, and fills the space between the main chains and crosslinks, as well as occupying the microvoids created during the polymerization phenomenon.[20] Time or aging is expected to be a significant factor associated with the amount of water sorption; moreover, composites which absorb more water, will show more decreased surface microhardness. The present study confirmed the latter clarification.

Carreiro *et al*.,[21] evaluated the effect of aging in distilled water on the hardness and compressive strength of a direct composite resin and three indirect composites. The results showed that aging in water reduced the hardness for all composites, proving the results of present study.

De Moraes *et al*.,[22] study described the effect of 6 months of aging in water on the surface roughness and surface/subsurface hardness of two microhybrid resin composites. Eventually, the results revealed that the 6-month period of storage in water presented a significant softening effect on the surfaces of the composites. These results are also in line with the results of the present study.

Hahnel *et al*.,[23] investigated the aging manners of dental composites regarding surface roughness and Vickers hardness. They explained that artificial aging causes a significant decline in mechanical properties. This report could also substantiate the results that we obtained in our present investigation.

In Oliveira *et al*.,[24] study, they selected three types of acrylic and one composite resin. The Vickers hardness and roughness were measured following 24 h storage in distilled water. They concluded that storage in distilled water had not any considerable effect regarding the evaluated properties. Comparing to the results of present study, this difference can be due to the short time of storage, since it has been proved that water sorption is a slow process[10] and more time is needed to measure the effects of this process.

Within the limitation of this *in vitro* study, the postcured treatment was efficient in increasing the VHN. So, postcuring with microwave and labolite must be effective for increasing the degree of conversion which results in long-lasting composite restorations. Additional clinical studies are necessary to analyze the success of two different postcuring techniques and aging of restoration in oral cavity.

CONCLUSION

This investigation attested that the postcuring process increases the surface microhardness of composite resins. The postcuring process by microwave was more effective than laboratory light source in increasing the surface microhardness. Moreover, aging in distilled water decreases the surface microhardness of the composite resins.

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