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Comparative evaluation of heat versus microwave irradiation on the microhardness of glass ionomer cements

Monadle Raouf Hadi*

*Department of Conservative Dentistry, College of Dentistry, University of Alkafeel, Holly Najaf, Iraq.

Email: Monadle.almansoor@alkafeel.edu.iq

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Abstract

Objective: To compare the effects of conventional oven heat and microwave treatments on the surface microhardness of conventional glass ionomer cement (GIC). **Materials and Methods:** Thirty disc-shaped specimens (10 mm diameter × 4 mm thickness) were fabricated from self-cured conventional GIC (Cavex) and randomly divided into three groups (n=10). The control group received no heat treatment and was stored at room temperature for 24 hours. The oven group was subjected to 50°C in a convection oven for 15 minutes after a 24-hour setting. The microwave group was exposed to microwave radiation for 10 minutes after the identical setting. Surface microhardness was evaluated using a Vickers hardness tester (25g load, 5 seconds, five indentations per specimen). Statistical analysis included descriptive statistics, pairwise correlation, and post-hoc Tukey HSD testing. **Results:** Microwave-treated specimens showed the highest mean microhardness, closely approaching or slightly exceeding controls. Oven-treated specimens exhibited the lowest values. Significant differences were found between microwave and control groups ($p<0.05$), with high correlation. Microwave samples showed the greatest variability; controls showed an optimal balance of high mean values, consistency, and predictability. **Conclusion:** Microwave treatment can maintain GIC microhardness, but variability limits reliability; the conventional 24-hour setting remains safest and most predictable.

Keywords: Glass ionomer cement, microhardness, microwave irradiation, heat treatment.

INTRODUCTION

With its special capacity to chemically attach to tooth structure and release fluoride over an extended period of time, glass ionomer cement (GIC) is a multipurpose dental restorative material. Since its 1972 introduction by Wilson and Kent, it has established itself as a fundamental component of contemporary dentistry, especially in applications where its unique qualities are beneficial [1].

GIC is a water-based cement that sets via an acid-base reaction between two main components: a powder of calcium fluoroaluminosilicate glass and polyalkenoic acid (e.g., polyacrylic acid, itaconic acid, or maleic acid). The acid assaults the glass particles when the powder and liquid are mixed, releasing ions (Ca^{2+}, Al^{3+}, F^{-}). The remaining glass particles are encapsulated in a hydrogel matrix created by the calcium and aluminum ions cross-linking with the polyacid chains, resulting in a firmly set cement. Although it starts quickly, this reaction takes around 24 hours to fully develop [2].

GICs chemically bond to both enamel and dentin without the need for a separate adhesive. This bonding is achieved through an ionic exchange at the interface, where carboxylate groups from the polyacid chelate with calcium ions from the tooth.

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The setting reaction is mild and exothermic, causing minimal pulp irritation, making it suitable for use in deep cavities. The release of fluoride ions helps inhibit caries, reduce demineralization, and promote remineralization of adjacent tooth structure. Furthermore, GICs can act as a "fluoride reservoir," absorbing fluoride from toothpaste or mouth rinses and re-releasing it. Glass ionomer cement's coefficient is close to that of tooth structure, minimizing microleakage at the restoration margins. Conventional Glass ionomer cements are opaque and have a less natural appearance than composites, but newer resin-modified and high-viscosity types offer improved aesthetics [3].

Timing of heat application has primary effects on clinical outcomes. During setting of the cement, it can lead to accelerated setting reaction, reduced working time, altered viscosity, difficult handling, risk of incomplete adaptation, and potentially weaker final structure. After setting, it may lead to dehydration and cracking, thermal expansion mismatch, and degradation of the matrix. Surface roughness, microleakage, marginal failure, secondary caries, and reduced restoration longevity [4].

Heat can significantly affect the properties of glass ionomer cement both during and after its setting phase [5]. One of the most critical effects is related to the material's inherent hydrophilicity. Glass ionomer cements undergo an initial "water sorption" phase, which is essential for their structural stability. When intense heat is applied, such as during dry polishing without the use of a water coolant, it rapidly drives water out of the cement matrix. This rapid dehydration causes the material to shrink considerably, leading to surface crazing (formation of micro-cracks), increased surface roughness, and a severe reduction in mechanical strength. Heating set glass ionomer cements to 100°C resulted in a significant and irreversible loss of surface hardness caused by dehydration [4].

Heat treatment of glass ionomer cement during its setting phase enhances microhardness primarily by accelerating the acid-base reaction between the polyacid and the glass particles. This faster reaction promotes improved cross-linking within the polyacid matrix, resulting in a denser and harder material. Studies demonstrate that applying heat in the range of 37°C to 60°C can significantly increase the surface hardness of GIC, making it more durable and resistant to wear [6,7,8]. Higher temperatures can decrease the viscosity of the unset cement, making it more fluid initially. However, this is quickly followed by a rapid set [5].

Low-power microwave irradiation significantly speeds up the setting reaction of GIC. Glass ionomer cement sets via an acid-base reaction between fluoroaluminosilicate glass and polyacrylic acid [9]. Water is crucial as the reaction medium [10]. Microwave energy rapidly heats the water in the cement paste, accelerating hydration and strength gain. Microwaves (a form of non-ionizing radiation) cause dielectric heating; they make water molecules and ionic species within the cement matrix oscillate rapidly, generating internal heat [11,12]. The dielectric heating mechanism in the context of cementitious systems. It explains how water molecules (the primary dipole) and dissolved ions are the main agents for converting microwave energy to heat [13].

Microwaves can be used as a post-setting treatment to quickly improve the material's characteristics, mimicking or speeding up the sluggish maturation process that typically takes weeks. Enhanced early mechanical qualities and quicker maturation: internal heat from microwaves speeds up the acid-base interaction and encourages more cross-linking of the polyacrylate matrix. This causes the elastic modulus, surface hardness, and compressive strength to rise quickly. At room temperature, these attributes would normally take a day or longer to attain their values, but they can do so in minutes [14].

The purpose of this work was to compare the effect of heat and microwave radiation on the microhardness of glass ionomer cement. One of the objectives of this work is to study the effect of heat and microwave energy on increasing the hardness of glass ionomer cement in order to plan the future use of the material as an inlay or onlay made outside the human mouth.

MATERIALS AND METHODS

In this research, the glass ionomer cement material used was a self-hardening type (Cavex), and the samples of cement were made with 30 samples that were divided into three groups as follows:

The first group, ten samples, was considered the control group, where the cement was mixed and placed in the designated mold and allowed to set for 24 hours without any treatments.

For the second group, ten samples were prepared by mixing the cement, placing the mixture into designated molds, and allowing it to set for 24 hours. Subsequently, the samples were cured in a convection oven at 50 °C for 15 minutes. The ten specimens in the third group were prepared by mixing the cement, placing the mixture into designated molds, and allowing them to set for 24 hours. Following the setting period, the specimens were subjected to microwave treatment for 10 minutes.

A mold was fabricated from heavy-body polyvinyl siloxane impression material using a wax disc (10 mm diameter × 4 mm thickness) as a pattern. The impression material was prepared by manually mixing the base and accelerator, then formed around the wax disc. The resulting mold was used to produce glass ionomer cement specimens of the same dimensions. Polyvinyl siloxane impression material was selected for mold fabrication due to several advantageous properties. It allows for easy mixing without chemical hazards or adverse reactions with the cement. The material sets via a cold reaction, generating no significant heat that could deform the wax disc during mold formation. Furthermore, it exhibits sufficient durability, resisting damage, rupture, or dimensional changes, while enabling easy removal of the cement disc. The mold can also be reused for fabricating multiple cement specimens.

The cement powder and liquid were mixed in a 2.2:1 weight ratio, as specified by the manufacturer. The powder was placed on a designated mixing pad, and the appropriate number of drops of liquid were added. Mixing was performed manually for 30 seconds using a plastic mixing spatula. Following mixing, the cement was packed into the mold using a stainless-steel spatula. To prevent the formation of air bubbles, the cement was introduced incrementally and pressed against the sides of the mold until the mold was filled. Following a 24-hour setting period, the cement specimens were removed from the molds. Excess material was removed from all surfaces, and the upper and lower surfaces were smoothed using diamond burs with cooling water. These surfaces were subsequently polished to a smooth finish with a rubber cup and pumice to prepare the specimens for hardness testing.

After preparation was completed, the specimens were randomly assigned to three groups. The first group was maintained without any heat treatment and served as the control. The second group was placed in a clean convection oven and heated at 50 °C for 15 minutes. The third group was placed in a microwave oven and exposed to a power output of 800 W at a frequency of 2450 MHz, set to the lowest power level (10%), for 10 minutes. After that, the samples were kept in a dry place at room temperature until a microhardness test was performed.

A Vickers microhardness testing machine was used to evaluate the specimens after a 24-hour interval. On each specimen, five indentations were made at distinct locations on the surface. To prevent plastic deformation of the polymer matrix a factor that could compromise the accuracy of the surface

microhardness measurements a load of 25 g was applied with a dwell time of 5 s. The microhardness values were recorded directly from the digitally controlled testing apparatus, which automatically aligns the pointers to the indentation edges and computes the Vickers hardness value following the specified load and dwell duration.

RESULTS

Microhardness measurements were conducted under a load of 25 g with a dwell time of 5 s. Five indentations were made on each specimen, and the individual microhardness values were recorded. For each specimen, the mean value was calculated from the five readings. Subsequently, the group mean microhardness value was obtained by averaging the five specimen means per group, as summarized in Table 1.

Table 1. Descriptive Statistics of the tested groups.

Group	Mean	Median	Standard Deviation	Range (Min–Max)
Oven treated	69.2	73.2	11.3	51.1 – 87.4
Control Group	74.5	76.3	10.9	51.1 – 94.7
Microwave treated	75.8	78.0	12.6	43.2 – 89.9

Microhardness values were generally similar across the experimental groups, although distinct differences were evident among them. The lowest values were recorded for specimens in the oven-treated group, while microwave-treated specimens demonstrated values comparable to, or marginally greater than, those of the control group. Overall, microwave treatment produced higher microhardness values than oven heat treatment. Table 1 presents the mean values and standard deviations for each group, expressed as Vickers hardness numbers (VHN). Statistical tests were performed to investigate the presence of differences and their degree of significance between the results obtained from the test samples. The bar chart (Figure 1) compares group means, showing that the values of microhardness of samples treated with microwave radiation were the highest, while the values of samples treated with oven heat were the lowest.

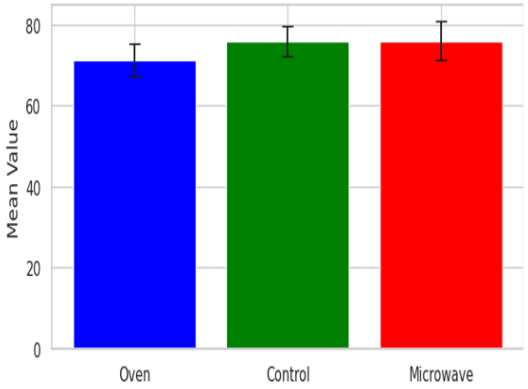


Figure (1) Bar Chart compares group means with confidence intervals.

A high degree of correlation is evident in the pairwise correlation matrix (Table 2) when comparing the microhardness values of the microwave-treated samples with those of the control group.

Table 2: Pairwise Correlation Matrix

	Oven	Control	Microwave
Oven	1.00	0.45	-0.12
Control	0.45	1.00	0.33
Microwave	-0.12	0.33	1.00

From the post-hoc Tukey HSD test, it was clear that there was a significant difference between the microwave-treated values and those of the control samples, as shown in Table (3).

Table (3) Post-hoc Tukey HSD Test determines significant differences between groups.

Comparison	Mean Difference	p-value
Oven vs. Microwave	-6.6	**0.01**
Oven vs. Control	-5.3	**0.04**
Control vs. Microwave	1.3	0.65

Finally, from the box plot shown in Figure (2), there were high spread values of the microwave-treated samples, while the control group values showed the lowest spread values.

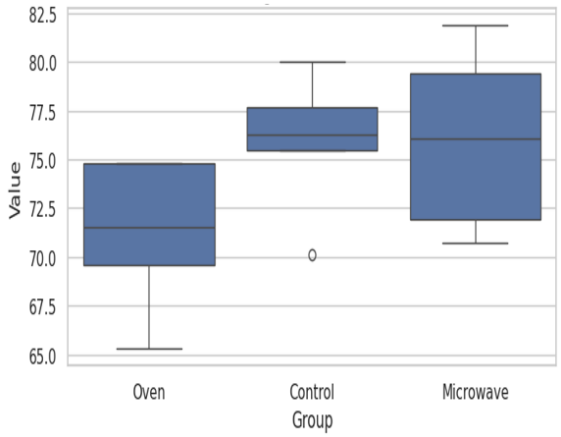


Figure (2) Box Plots by group highlight distributions and outliers.

DISCUSSION

The purpose of this study was to assess how various post-curing heat treatment techniques, particularly microwave and oven heat, affected the microhardness of glass ionomer cement (GIC). A restorative material's microhardness is a crucial characteristic since it is correlated with wear resistance, clinical durability, and the material's resistance to abrasion and intraoral forces. The findings showed that, in comparison to the control group, microwave treatment produced noticeably higher microhardness values, whereas oven heat treatment produced the lowest. These results imply that the heat application technique has a significant and unique effect on the physicochemical characteristics of the set cement.

The nature of microwave radiation can be used to explain the observed rise in microhardness in the microwave-treated group. Microwave radiation interacts with polar molecules in the material, such as water and ionic species, creating fast molecular oscillation and volumetric heating, in contrast to traditional oven heating, which depends on conduction and convection to transport heat from the surface inward. It is thought that this effective energy transfer will speed up the acid-base setting reaction and improve the GIC's maturation process. A denser, more cross-linked polysalt matrix results from a more thorough interaction between the fluoroaluminosilicate glass particles and the polyalkenoic acid. This result is in line with other research showing enhanced mechanical characteristics in GICs after exposure to microwaves. For example, Tantbirojn et al. (2004) showed that by encouraging a more complete setting reaction, microwave irradiation could considerably raise the surface hardness of traditional GICs. In a similar vein, Kleverlaan et al. (2004) observed that, if the temperature rise is regulated to prevent dehydration, rapid curing at higher temperatures can improve the early mechanical strength of GICs [15].

On the other hand, the group that was oven-treated had the lowest microhardness values. The process of heat transfer is less consistent and may result in surface dehydration, even though oven heating also supplies thermal energy to speed up the setting reaction. The cement matrix most likely lost too much water as a result of the extended exposure to high, dry heat in a traditional oven. Ionic transport and the matrix's ongoing hydration in GICs depend on water. A brittle, porous, immature structure with diminished mechanical integrity might result from premature or severe dehydration, which can stop the setting reaction. This phenomenon is well-documented. Cattani-Lorente et al. (1999) emphasized that long-term maturation and mechanical strength of GICs depend on maintaining an ideal water balance. The production of the calcium and aluminum salt bridges necessary for the cement's structural integrity is disrupted by excessive desiccation, such as that which can happen with uncontrolled heat application. This compromises the hardness of the cement [16].

The microwave-treated and control groups differed significantly, according to the post-hoc Tukey HSD test. This is an important discovery since it shows that the increase in hardness is a real material improvement rather than just a statistical phenomenon. The robustness of this difference is further supported by the pairwise correlation matrix. The usual clinical setting is represented by the control group, which was allowed to settle at room

temperature without any external heat treatment. Although this group produced acceptable hardness values, the microwave treatment seems to provide a way to improve the qualities attainable by traditional setups, which could result in a restoration that is more long-lasting.

In comparison to the control and oven-treated groups, the box plot analysis showed a wide range of values within the microwave-treated group, suggesting more variability in hardness. The intrinsic heterogeneity of the GIC material and the sensitivity of the microwave treatment parameters could be the causes of this variability. Localized variations in the degree of setting could result from factors including the exact water content of individual specimens, their placement within the microwave cavity (which can impact the homogeneity of the electromagnetic field), and the specific microwave power output. This implies that although microwave treatment has a lot of potential, thorough standardization of the exposure protocol would be necessary for its clinical implementation in order to guarantee reliable and consistent outcomes. Because of its smaller spread, the control group's results are less ideal but more predictable [17-20].

CONCLUSION

The findings of this study show that applying microwave radiation is a better way to increase GIC's surface microhardness than traditional oven heat treatment. The microwave-treated group performed better because of the efficient, volumetric heating that speeds up the acid-base reaction without producing severe dehydration. On the other hand, the poor performance of the oven-heated group highlights the negative impact of uncontrolled surface desiccation on the mechanical properties of the cement. Microwave therapy may be a useful method for enhancing the clinical performance of GIC restorations, especially in high-stress-bearing applications, as indicated by the notable improvement over the control group.

RECOMMENDATION

If the goal is to maximize performance reliably, the control group conditions appear optimal. The microwave treatment group may be worth exploring further if variability can be controlled. But the oven treatment seems the least effective, and heat control must be noted.

CONFLICT OF INTEREST: **NIL**

FUNDING: **NIL**

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