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Effect of ultrasound application during setting on the mechanical properties of high viscous glass-ionomers used for ART restorations

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ABSTRACT

This study was conducted to evaluate the effect of ultrasound application on the surface microhardness (VHN) and diametral tensile strength (DTS) of three high viscous glass-ionomer restorative materials (HVGIRMs). For each test (VHN and DTS), a total of 180 specimens were prepared from three HVGIRMs (Ketac-Molar Aplicap, Fuji IX GP Fast, and ChemFil Rock). Specimens of each material ($n = 60$) were further subdivided into three subgroups ($n = 20$) according to the setting modality whether ultrasound (20 or 40 s) was applied during setting or not (control). Specimens within each subgroup were then equally divided ($n = 10$) and tested at 24 h or 28 days. For the VHN measurement, five indentations, with a 200 g load and a dwell time for 20 s, were made on the top surface of each specimen. The DTS test was done using Lloyd Testing machine at a cross-head speed of 0.5 mm/min. Ultrasound application had no significant effect on the VHN. Fuji IX GP Fast revealed the highest VHN value, followed by Ketac-Molar Aplicap, and the least was recorded for ChemFil Rock. Fuji IX GP Fast and Ketac-Molar Aplicap VHN values were significantly increased by time. ChemFil Rock recorded the highest DTS value at 24 h and was the only material that showed significant improvement with both US application times. However, this improvement did not sustain till 28 days. The ultrasound did not enhance the surface microhardness, but its positive effect on the diametral tensile strength values was material and time dependent.

Introduction

Glass-ionomer restorative materials (GIRMs) are acknowledged for their ability to bond to dental structures as well as their capacity for fluoride release and uptake [1,2]. However, like all dental materials, GIRMs have certain drawbacks, chiefly their water sensitivity and insufficient mechanical properties [3]. Thus, attempts were done to overcome the slow setting reactions, in order to decrease the moisture sensitivity as well as to improve the mechanical strength at early stages of the acid-base reaction [4]. Consequently, there have been considerable modifications in the formulations, physical, mechanical and handling properties of this group of materials to enhance their clinical applications. High viscous glass-ionomer restorative materials are one of the results of these improvements. Meanwhile, modifications in clinical application technique were also carried out. Ultrasound (US) is routinely used for setting cement in the building industry and authors

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[5–9] have previously shown that glass-ionomer restorative materials can be command set by a similar process. An external energy source can be conducted through ultrasonic excitation generated from dental scaler [7] which could enhance the materials' physical and mechanical properties.

The reported increase in surface hardness of GIRMs during early setting time after US application could help in the resistance of the material to moisture contamination [4] but, whether this effect remains over time or not, still needs confirmation. Surface hardness property is defined as the resistance of a material to indentation or penetration [10]. Many studies have been done using Vickers hardness (VHN) test to assess the surface hardness of GIRMs [4,11–13]. Moreover, the mechanical strength is an important factor that has to be analyzed for clinical success of dental restorations. The US application could be effective in achieving a homogenous set throughout the bulk of the material enhancing its resistance to force of mastication. The diametral tensile strength test (DTS), which has been used by many researchers [12–14], provides a simple method for indirect measurement of tensile strength of brittle materials such as GIRMs.

Although there is increasing attention concerning the effects of US application during setting, there has been a lack of studies to elucidate its concurrent effect on physical and mechanical properties of HVGIRMs and alteration of these properties with time. The null hypotheses tested were as follows: (1) The US application has no significant effect on either VHN or DTS values of the used HVGIRMs at both testing times. (2) The difference among the tested HVGIRMs has no significant effect on any of the evaluated properties with any setting modality at all testing times. (3) The testing time has no significant effect on the recoded VHN and DTS values of all tested materials with any setting modality.

Material and methods

The three high viscous glass-ionomer restorative materials investigated in this study as well as their composition, manufacturers and lot numbers are listed in Table 1. All specimens were prepared at room temperature (23 ± 1 °C) in a relative humidity of 50 ± 5% in conformance with ISO 9917-1:2003 [15].

Specimen preparation

Mold and base fabrication

A split Teflon mold (2 mm in thickness) was specially fabricated with a central hole of 4 mm in diameter [14]. An accessory Teflon ring with an elevated central button was supplied with the mold to help in specimens' separation from the mold without contamination. A Teflon base with a circular depression corresponding to the external dimension of the mold was also fabricated to support and hold the Teflon mold assembly in position during US application (Fig. 1).

Material insertion

All glass-ionomer capsules of the tested materials were activated and mixed mechanically by an amalgamator (Linea Tec.S.R.L, Montegrosso, Italy) according to the manufacturer's instructions. Thus, Ketac-Molar Aplicap and Fuji IX GP Fast GIRMs were mixed for 10 s with the exception of ChemFil Rock which was mixed for 15 s. Immediately after mixing; the paste was injected into the split Teflon mold until being slightly overfilled. Two polyester strips were used to cover both sides of the mold. A microscope glass slide was hand pressed against the top of the mold to completely pack the material into the mold and to obtain flat and smooth surface.

Specimen grouping

A total of 360 specimens were prepared. The specimens were divided into three groups (n = 120), according to the type of HVGIRMs used. Specimens of each group were further allocated into three subgroups (n = 40) according to different setting modalities; either control (standard setting method) or command set with US application for 20 or 40 s. Specimens of each subgroup were further subdivided into two classes (n = 20) according to the time of testing (24 h and 28 days). Half of the specimens within each class were subjected to surface microhardness measurement and for the other half diametral tensile strength testing was performed.

Preparation of control group specimens (standard setting)

For the control group, specimens were allowed to set under load application of 150 g to ensure an equal pressure was applied for all specimens. Specimens were then incubated at 37 °C for 15 min [16]. Then, specimens were unloaded and left for another one hour under the same conditions [17]. Afterward, specimens were separated from the molds and fine flashes were removed with caution [16]. The specimens were checked with a magnifying lens (10×, Wellpromo.com, magnifying lens, China) for any cracks or air bubbles. Specimens with visible defects were discarded. The specimens' correct dimensions were verified using a digital caliber to an accuracy of 0.01 mm [13] and weighed using a sensitive balance (Kern Precision Balance, Avon Corporation Ltd., India). Each specimen was then stored in a plastic test tube containing 5 ml of de-ionized water, labeled and incubated at 37 °C.

Table 1 Material brand names/manufacturers, compositions and lot numbers of tested glass-ionomer restorative materials.

Material brand names/manufacturers	Composition	Lot number
Ketac-Molar Aplicap (3M ESPE, Sheifeld Germany)	Powder: Alumino-fluoro-silicate glass, Liquid: polycarboxylic acid, tartaric acid and water	404500
Fuji IX GP Fast (GC Company, Tokyo, Japan)	Powder: Alumino-fluoro-silicate glass, Liquid: polycarboxylic acid, tartaric acid and water	1008091
ChemFil Rock (Dentsply, Konstanz, Germany)	Powder: Calcium-aluminum-zinc-fluoro-phosphor-silicate glass, Liquid: polycarboxylic acid, iron oxide pigments, tartaric acid and water	1105001122

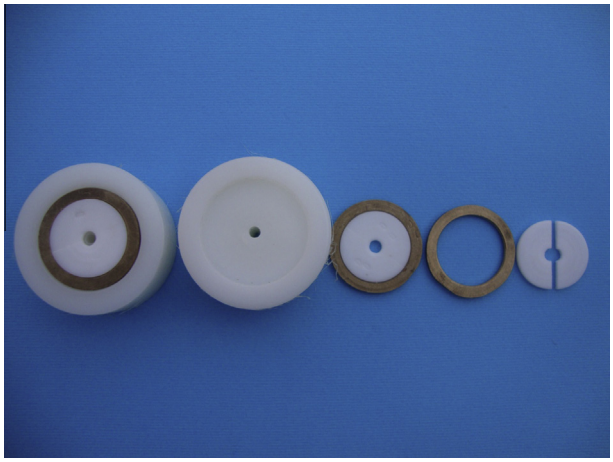

Fig. 1 The split Teflon mold and the supporting Teflon base.

Preparation of the specimens subjected to ultrasound application during setting

The specimens were left after mixing for 40 s before US application [18]. The US application was done either for 20 or 40 s using a dental scaler (Ultrasonic Scaler (DTE-D5), Guilin, China) with a B-tip instrument [4] at a frequency ranging from 25 to 30 kHz [5]. A specially designed holder was fabricated to enable the B-tip instrument to have a uniform equal contact with the top surface of all test specimens (Fig. 2). Water cooling was not applied during ultrasonic application to avoid interference with the setting reaction [19]. Then, the specimens were handled in the same way as the specimens of the control group until being tested.

Surface microhardness measurement

VHN measurements were taken using a digital microhardness tester (Model HVS-50, Laizhou Huayin Testing Instrument Co., Ltd., Laizhou, Shandong, China) and a 200 g load was applied for a dwell time of 20 s [11]. Five indentations were performed on the top surface of each specimen [20]. The mean VHN of the five readings of each specimen as well as the overall mean VHN for each subgroup was then calculated [20].

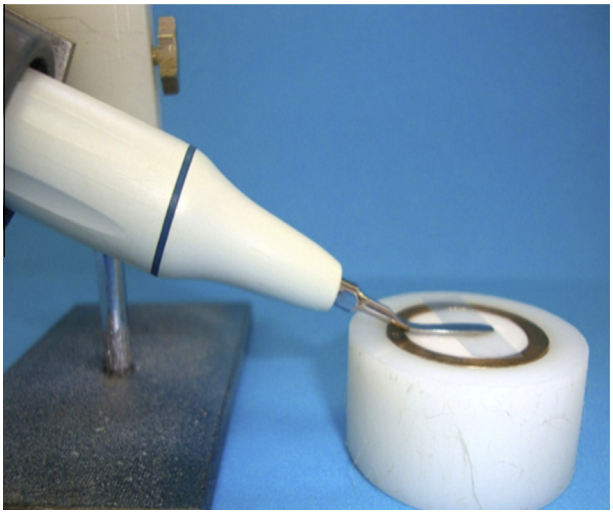

Fig. 2 The holder used for positioning the B-tip instrument.

Diametral tensile strength measurement

Specimens were compressed diametrically until fracture using the universal testing machine (Lloyd instruments Ltd., Ametek Company, West Sussex, UK) at a cross-head speed of 0.5 mm/min. The diametral tensile strength, T was calculated in MPa using the following formula: $T = 2P/\pi dl$ where P is the maximum load applied (Newton), d is the measured mean diameter of the specimen (mm) and l is the measured length of the specimen (mm) [13].

Statistical analysis

Data were statistically described in terms of mean and standard deviation. Multi-way analysis of variance (ANOVA) was done to test the effect of the setting modality, material type and testing time or their interactions on microhardness as well as diametral tensile strength tests. For each test, One-way ANOVA was done to compare the different materials with each setting modality and testing time. Bonferroni post hoc test was used for pairwise comparisons when indicated. Student's t test was used to compare the two testing times with each material type and setting modality. P values less than 0.05 was considered statistically significant. All statistical calculations were done using computer programs SPSS (Statistical Package for the Social Science; SPSS Inc., Chicago, IL, USA) version 15 for Microsoft Windows.

Results

The mean (standard deviation) of VHN and DTS values of the three HVGIRMs subjected to different setting modalities and tested at 24 h and 28 days are presented in Table 2. For the microhardness test, the Multi-way ANOVA revealed significant effects for the material type ($P = 0.02$) and the testing time ($P < 0.01$) and not for the setting modality ($P = 0.05$). The interactions between these variables were not significant except the interaction between the material type and testing time was significant ($P < 0.01$). One-way ANOVA test revealed a significant difference among the tested materials with all setting modalities. Bonferroni post hoc test showed that Fuji IX GP Fast had the highest VHN value, followed by Ketac-Molar Aplicap while ChemFil Rock came with the least value. The *t-test* showed that Fuji IX GP Fast and Ketac-Molar Aplicap had a significant increase in their VHN values at 28 days while there was no significant increase in the hardness of ChemFil Rock by time (Table 2).

Regarding the DTS, the Multi-way ANOVA showed that the setting modality, material type and the testing time had a significant effect ($P < 0.01$, $P < 0.001$ and $P < 0.001$, respectively). Setting modality and material type ($P = 0.24$) as well as setting modality and testing time interactions ($P = 0.27$) were not significant. However, the material type and the testing time interaction had a significant effect ($P = 0.001$). Interaction among all the tested variables (setting modality, material type and testing time) was significant ($P = 0.02$). For the US application, at 24 h testing time, the DTS of ChemFil Rock significantly improved with both US application times (20 and 40 s) while at 28 days testing time this improvement was not sustained. The One-way ANOVA test revealed a

Table 2 The mean (standard deviation) surface microhardness (VHN) and diametral tensile strength (MPa) of the three tested high viscous glass-ionomer restorative materials as a function of setting modalities (ultrasonic application for 20 (20 U) or 40 (40 U) seconds or not (control) during setting) and testing times (24 h and 28 days), $n = 10$.

Test	Tested HVGIRMs	Setting modalities and Testing times								
		Control		P^* value	20 U		P^* value	40 U		P^* value
		24 h	28 days		24 h	28 days		24 h	28 days	
Vicker's hardness test (VHN)	Ketac-Molar Aplicap	77.6 (1.2)a	87.8 (1.3)a	< 0.001	76.1 (2.5)a	90.7 (3.7)a	0.001	73.0 (1.9)a	87.0(0.9)a	< 0.001
	ChemFil Rock	58.5 (0.9)b	58.7 (0.8)b	0.718	58.5 (1.7)b	61.4 (4.1)b	0.239	55.8 (1.8)b	56.3 (1.8)b	0.698
	Fuji IX GP Fast	85.7 (3.3)c	98.4 (7.9)c	0.020	84.9 (1.3)c	99.9 (2.3)c	< 0.001	85.3 (1.7)c	100.3 (2.4)c	< 0.001
	P^{**} value	< 0.001	< 0.001		< 0.001	< 0.001		< 0.001	< 0.001	
Diametral tensile strength test (DTS)	Ketac-Molar Aplicap	11.0 (5.2)a	10.8 (3.5)	0.907	12.7 (2.8)a	12.6 (3.6)	0.936	12.6 (3.5)a	10.4 (3.3)	0.143
	ChemFil Rock	14.5 (3.4)b^{+}	11.2 (4.3)	0.167	21.9 (3.9)b^{++}	11.8 (4.0)	< 0.001	18.6 (6.1)b^{++}	14.4 (0.2)	0.040
	Fuji IX GP Fast	12.6 (3.0)c	9.7 (3.3)	0.047	14.2 (3.4)c	12.7 (4.0)	0.356	13.6 (4.4)c	12.9 (3.1)	0.656
	P^{**} value	0.033	0.645		< 0.001	0.827		0.013	0.045	

Numbers in brackets are standard deviation.
Different small letters indicate statistical significant difference. Values with + and ++ superscripts are statistically significantly different (Bonferroni test, $P < 0.05$).
** One-way ANOVA and post hoc Bonferroni.
* *t*-Test.

significant difference among the tested materials with all setting modalities at 24 h but not at 28 days. ChemFil Rock recorded the highest DTS value, followed by Fuji IX GP Fast and the least was for Ketac-Molar Aplicap (Table 2).

Discussion

Based on the results of the current study, the first null hypothesis that the US application does not improve the surface microhardness and the diametral tensile strength properties of any of the tested HVGIRMs at the two testing times, was partially accepted. Ultrasound application did not improve the surface microhardness at the two testing times. However, it had a positive effect on the diametral tensile strength values and this effect was material and time dependant. Based on these findings, US application cannot be recommended as a routine treatment for ART restorations. Moreover, researchers [21,22] reported conflicting results about the effect of US application on the adaptation of the glass ionomer restorations. The results of the current study revealed that the material type and the testing time had significant effects on the recorded VHN and DTS values, thus the second and third null hypotheses should be rejected.

This study was the first to test the mechanical properties for ChemFil Rock HVGIRM, which was claimed by the manufacturer to behave better with ART restorations in stress bearing areas, when it was subjected to US application. Previous studies supported the positive effect of the 40–55 s ultrasound application [23] on hardness [4] and compressive strength [6] as well as on fluoride release of HVGIRMs [24]. On the other hand, the positive effect of 55 s US application on fluoride release was referred to surface dissociation or de-clustering of particles which did not only render the surface more reactive but also could have a negative effect on the resistance of the surface to degradation. Though this risk, the enhancement of fluoride release could be considered positive in case of using the glass ionomer as a caries control restoration. Nevertheless, this version of highly viscous glass ionomer including the newly introduced ChemFil Rock is indicated for ART restorations in stress bearing areas. Therefore in this study, the two US application times were chosen to test whether better hardness and diametral tensile strength could be achieved without jeopardizing the surface layer quality that could accompany fluoride release enhancement.

Our results reflected that the surface microhardness recorded by Fuji IX GP Fast surpassed those for Ketac-Molar Aplicap and the lowest value was recorded for ChemFil Rock. Variations in the microhardness of different GIRMs were explained based on the maturity state of every material and its setting reaction [25–29]. Preliminary studies [5–8,30] suggested that adding kinetic energy from the ultrasonic device to the material can enhance the rate of setting reaction due to the increase in temperature. The US may also contribute to acceleration of the reaction by de-clustering glass particles and enhancing the diffusion of the reaction components. Moreover, it may offer a reduction in porosities or may result in a closer packing of particles [19]. On the other hand, it can be expected that the increase in viscosity due to the progression in the formation of the polycarboxylate network can steadily reduce the rate of further reaction. Also, US application could cause a temperature rise with subsequent liquid evaporation from the surface layer which may compromise the optimum glass powder to aqueous acidic ratio and affect the extent of co-ordination and chelation of bonded glass networks [4]. These speculations may clarify the lack of improvement in surface microhardness induced by US application during setting of HVGIRMs in the present study. Previous work [4] showed that US application caused an improvement in the microhardness of Ketac-Molar HVGIRM at 0.5 h after setting but not later (4 h and 1 week).

Regarding the DTS, at 24 h, there was a significant difference among the tested HVGIRMs where ChemFil Rock revealed the highest value. The mechanical resistance of GIRMs was reported to be conditioned by numerous factors such as the chemical composition, glass structure [31], nature, concentration [32] and molecular weight of polycarboxylic acid [33], and the proportion of powder/liquid [26]. Filler composi-

tion and particle size have also a significant influence on the mechanical properties [34]. At the same time, the current study showed that, the 24-h diametral tensile strength of ChemFil Rock was influenced by US application during setting. This could reflect that the applied energy to the surface has been transmitted throughout the material bulk despite that the GIRMs are good insulators and exhibit a similar thermal diffusivity to dentin [35]. It seems that compositional differences are also involved in making the US application effective. Some work showed that the type of polyacrylic acid and the percentage of tartaric acid can influence the response of the GIRMs to US application [7]. ChemFil Rock contains zinc in the glass powders as well as has a novel acrylic acid copolymer with increased molecular weight. Both modifications are expected to enhance the setting reaction and to modify the formed matrix. ChemFil Rock contains also itaconic acid that has been reported to increase the DTS [36]. This may clarify the significant increase in the DTS of ChemFil Rock when subjected to US application and not in that of other tested materials.

As for the effect of time, our findings demonstrated a significant increase in the surface microhardness of Fuji IX GP Fast and Ketac-Molar Aplicap after storage. The increase in surface hardness of the glass ionomer by time was recorded in previous *in vitro* [4,11–13] and *in vivo* [37] studies. Change in hardness by time may reflect the progression in the setting reaction [25,29] where further ionic cross-linking formation occurs [38]. Meanwhile, there was stability in surface microhardness of ChemFil Rock over time. This could be attributed to the zinc modified filler particles that allowed fast setting reaction, thus less reactive ions were available for further maturation to take place. Despite there was an increase in surface microhardness by time, the DTS values of Fuji IX GP Fast and Ketac-Molar Aplicap were not affected by time. The lack of time effect on the DTS of HVGIRM was also recorded by others [13,16]. On the other hand, the recorded high DTS of ChemFil Rock at 24 h did not sustain till 28 days. Over the past decade, the metal reinforced GIRMs have been introduced where the reinforcing effects of metal additives were subject of much controversy [14,39]. ChemFil Rock, a zinc filler modified HVGIRM, may suffer from compositional heterogeneity that rendered it more sensitive. This may explain why this material behaved like the metal reinforced materials for being not harder or more durable. Based on the Chemfil Rock results, it seems that it would not behave better than the other available high viscous glass ionomer materials when used as ART restorations. A clinical trial is required to be conducted to validate the obtained *in vitro* findings. Thus, present study findings could support the assumption that the modification in the chemistry of the powder and the change in the fillers composition are crucial for mechanical properties improvement.

Conclusions

The ultrasound did not enhance the surface microhardness, but its positive effect on the diametral tensile strength values was material and time dependent.

Conflict of Interest

The authors have declared no conflict of interest.

Compliance with Ethics Requirements

This article does not contain any studies with human or animal subjects.

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