The Effect of Heating and Ultrasound on the Shear Bond Strength of Glass Ionomer Cement

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A B S T R A C T

The aim of this study was to compare the influence of externally applied »command set« methods (heat, ultrasound) on shear bond strength to enamel of several glass ionomer cements (GIC). The vestibular surfaces of 180 extracted premolars were wet ground until a flat enamel surface was created, and divided into three groups. Three restorative GICs (Fuji IX GP Fast, Fuji Triage, Ionofil Molar AC) were cured in three ways: standard (SC), ultrasonic excitation (UC) and by an external heat source (HC). In each group, teeth were conditioned in two ways: 30 with 10% polyacrylic acid and 30 without conditioning. The GIC were used to fill teflon molds (3x4 mm). The samples were loaded in a Universal testing machine (Lrx Material Testing Machine) at a 1 mm/min crosshead speed. Results showed that heat cured Fuji IX on conditioning enamel had significantly greater shear bond strength (13.3 MPa) than all other tested groups (8.6–10.8 MPa) ($p<0.001$). The mean shear bond strength in GIC with SC and without enamel conditioning was 3.6–5 MPa and had significantly lower bond strength. Heating of GIC increase bond strength, improves the properties of GIC restoration and can be recommended for use as a »command set« method.

Key words: *glass ionomer, shear bond strength, heating, ultrasound*

Introduction

Glass ionomer cements (GIC) are increasingly used to treat both primary and permanent teeth. The chemical adhesion to enamel in combination with the fluoride-releasing property and its excellent biocompatibility gives glass ionomer cements an important place. GIC offers the advantages of adhesion to enamel and dentine and has cariostatic properties due to sustained fluoride release[1,2]. There are also some disadvantages such as the slow curing reaction and the weaker bond strength compared to composites[3,4]. An effective bond between GIC and tooth substance is important for the clinical success of glass ionomer restorations. A vast amount of research has been conducted in an effort to improve the strength of GIC by modifying the chemical composition of both the glass and polyelectrolyte components[4,5]. Some in vitro studies have investigated the influence of varying conditions such as humidity, temperature, and heat treatment on the bond strength[3,6]. The use of heat to improve the mechanical properties of resin composites has been also investigated[7–9]. The treatment of glass ionomer material with heat was described by Brune and Sidhu[6,10]. The idea of this procedure was to heat material at 50–70 °C after mixing, which enables command set of classical (chemically cured) material and increases the mechanical properties of material. As heat will generally accelerate chemical reactions, the application of heat is also expected to increase the acid-base reaction rate as well[3]. Sidhu et al. describe the increase of mechanical properties due to contraction and loss of water in material during heating[10]. It is considered that the compressive strength of restorative GIC will be increased at temperatures above 37 °C[4,11]. Some studies also showed increased bond strength of glass ionomer to hard dental tissues in such circumstances[3]. Heat treatment of Vitremer resulted in an increase in compressive strength[4]. Very few studies have been published on the effects of heat on the strength of newer glass ionomer.

Ongoing developments have led to new technique based on the application of ultrasonic energy[12,13]. The use of ultrasonic vibrations to change the setting of glass ionomer cements (GIC) was reported by Towler et al. It has been established that application of ultrasound to a GIC produces a marked acceleration in setting and en-

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hanced mechanical properties of GIC and increases adhesion to the tooth substances[12]. The effect on chemical setting processes has not been reported.

It has generally been observed that a surface conditioner is beneficial for bonding glass ionomer materials to enamel. For conventional glass ionomer, Hotz et al. found that a citric acid pretreatment of the enamel resulted in approximately 60% increase in bond strength compared to untreated enamel[14]. Powis et al. also examined the efficacy of the various surface treatments on bond strength to enamel and found that all the treatments resulted in increased bond strength. In particular, a 25% solution of polyacrylic acid (PAA) was found to be very effective[15].

The aim of this in vitro study was to examine the effect of enamel surface treatment and the influence of externally applied heat or the ultrasound on the GIC material in order to improve performance on shear bond strength to enamel for several glass ionomer cements (GIC). Also, aim of the study was to introduce the »command set« method for curing of GIC.

Materials and Methods

Shear bond strength

Commercially available materials used in this investigation are listed and described briefly in Table 1. One hundred and eighty extracted human premolars have been stored in deionized water. They were embedded in self-curing acrylic resin (AcryFix Kit, Struers A/S, Ballerup, Denmark, Batch No: 7222-0000). The vestibular surfaces of 180 extracted molars were wet ground in a polishing block on a polishing machine (RotoPol-11, Struers, Denmark) with use of abrasive disks ending with 1200 grit silicon carbide papers until a flat enamel surface was created. After the specified enamel pretreatment, a split Teflon mold with a cylindrical hole, 3 mm in diameter and 4 mm deep, was clamped over the treated area and filled with the appropriate glass ionomer material. Each glass ionomer was mixed according to manufacturer's instructions. Three restorative GICs: GC Fuji IX GP Fast (GC, LOT 0704023), GC Fuji Triage (GC, LOT 0703051), VOCO Ionofil Molar AC (VOCO, LOT 712064) were cured in three different ways: standard (SC) (manufacturers' recommendation method), ultrasonic excitation (UC) and by an external heat source (HC). The teeth were randomly divided into three groups of sixty samples *per* each and were conditioned in two ways: 30 with 10% polyacrylic acid (Dentin Conditioner, GC Corporation, Tokyo, Japan) and 30 without conditioning. Each of these groups comprised ten samples treated in different ways: SC, UC or HC. The GIC were mixed and applied following the manufacturers' instructions and used to fill teflon molds. Heating of glass ionomer cement was performed with conventional polymerization unit Elipar Trilight (3M ESPE Dental Products, Seefeld, Germany) standard mode at 750 mW/cm^2 for 40 s. The ultrasound was applied for 20 s using a flat bladed scaler attachment of a Soniflex (Kavo, Dental GmBh, Biberach/Riss, Germany). An infrared thermometer PCE-889 (PCE Group, Meschede, Germany) was used for recording temperature changes on the surface of the GIC material. Due to its high optical resolution of 50:1 an object's temperature can be measured at a substantial distance. Area of 2 mm was measured at 10 cm distance with thermometer laser diode output lower than 1 mW, wavelength of 630~670 nm and resolution of 0.1 °C. The distance of the infrared thermometer from the object and the size of the measurement point are related. All specimens were left undisturbed for 30 minutes, at which time the clamp was removed and the samples were stored in deionized water at 37 °C in incubator (Cultura, Vivadent) for 24 h. The shear bond strength between the glass ionomer and enamel was then measured on a Universal testing machine (Lrx Material Testing Machine, AMATEK Lloyd Instruments Ltd, Hampshire, UK) at a crosshead speed of 1mm/min. A shear load was applied to the base of the bonded GIC cylinder with a knife-edge rod (width, 0.5 mm). The surfaces at a fracture site were analyzed using stereo microscope and SEM. Obtained data were analyzed using three-way analysis of variance (ANOVA) and Tukey HSD test. The software used was SPSS Inc. SPSS for Windows Version 7.5 [Computer program manual](SPSS Inc. 444 N. Michigan Avenue, Chicago, Illinois 60611, U.S.A.).

Determination of fracture mode

Visual examination of failure mode of the bonding specimens was accomplished by viewing all of the debonded specimens under a light microscope at 12x magnifi-

TABLE 1
MATERIALS USED IN THE STUDY

Product	Type of glass ionomer cement	Manufacturer	LOT	Ratio powder/liquid
GC Fuji Triage Capsule	Highly viscose	GC Corporation, Tokyo, Japan	0703051 Exp 2009-03	P: 0.3 g L: 0.15 g
GC Fuji IX GP Fast	Highly viscose	GC Corporation, Tokyo, Japan	0704023 Exp 2009-04	P: 0.4 g L: 0.11 g
VOCO Ionofil Molar AC	Highly viscose	VOCO, Cuxhaven, Germany	712064 Exp 2010-02	P: 0.43 g L: 0.125 g

P – powder; L – liquid

TABLE 2
RESULTS OF ANALYSIS OF VARIANCE FOR SHEAR BOND STRENGTH OF GIC ON DIFFERENT TREATED ENAMEL AND CURED ON DIFFERENT WAY (HEAT, ULTRASOUND)

	SS	df	MS	F	p	
Main effects	Model	901.0	5	180.2	33.5	<0.001*
	Material (MAT)	118.8	2	59.4	11.1	<0.001*
	Enamel treatment (TR-ZU)	524.0	1	524.0	97.5	<0.001*
	Curing mode of GIC (TR-MAT)	258.2	2	129.1	24.0	<0.001*
Interaction 2-factorial	(Total)	169.5	8	21.2	3.9	<0.001*
	MAT * TR-ZU	6.6	2	3.3	0.6	0.544
	MAT * TR-MAT	52.1	4	13.0	2.4	0.050*
	TR-ZU * TR-MAT	110.8	2	55.4	10.3	<0.001*
Interaction 3-factorial	MAT * TR-ZU * TR-MAT	89.8	4	22.5	4.2	0.003*
Model		1160.4	17	68.3	12.7	<0.001*
Rezidual		870.4	162	5.4		
Total		2030.8	179	11.3		

*p<0.01, MAT – material, TR-ZU – enamel treatment, TR-MAT – curing mode of GIC

cation. Failure was identified as 'adhesive' if no observable glass ionomer remained on the enamel surface and 'cohesive' if bulk amounts of glass ionomer remained on the enamel surface. The fractured specimens were subjected to scanning electron microscope (SEM) (TESCAN VEGA TS5136LS) analysis.

Results

Shear bond strength

Heat application

The curing mode of GIC (F=24.0; p<0.001) and the enamel conditioning (F=97.5; p<0.001) had a significant effect on the debonding force. These are shown in Table 2. The result from Tukey HSD showed that glass ionomer cured with heating showed the best shear bond value (Table 2–4). All tested materials react equally on the application of heat. The heat cured Fuji IX GP Fast on conditioned enamel had significantly greater shear bond strength (13.3 MPa) compared to other tested groups (p<0.001). The mean shear bond strength in GIC with SC and without enamel conditioning ranged from 3.6–5 MPa and had significantly lower bond strength when compared to other groups. The bond strength of HC glass ionomer and conditioned enamel with 10% polyacrylic acid was significantly higher than other tested groups (Figure 1). All three materials showed statistically significant increase (p<0.001) in bond strength on enamel conditioned with 10% PAA compared to the enamel surfaces without conditioning.

Ultrasound application

The effect of application of US to shear bond strength of GIC is shown in Figure 1. The effects appear to material specific, with Fuji IX showing a greater effect than Fuji Triage and Ionofil Molar. These are shown in Table 3 and 5.

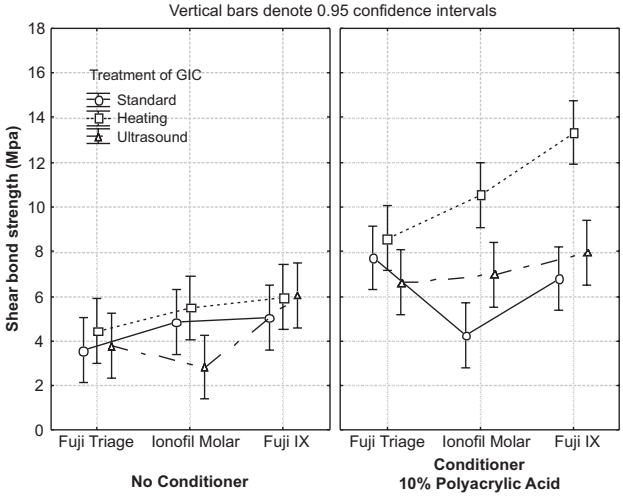

Fig. 1. Mean shear bond strengths (MPa) of glass ionomer cements depending on curing mechanism.

Fracture mode

Results of the failure mode analysis for the bond strength specimens are depended on enamel treatments and curing mode of GIC. Adhesive failure was observed only in group without conditioner and with standard curing mode of GIC. For all specimens, a statistically significant more cohesive failures were established when PAA was used, which is in correspondence with the bond strength and proposed effect on smear layer (p=0.005). Three-way ANOVA analysis showed that enamel pretreatment had a significant influence on the cohesive failure. The cohesive mode of failure for GIC also significantly depends on curing modes of GICs (p<0.001). The highest percentage of cohesive mode of failure was observed in the heat cured and ultrasound cured specimens

TABLE 3
TUKEY HSD TEST FOR SHEAR BOND TEST OF FUJI TRIAGE

Material	Cond[i]	Curing mode	1	2	3	4	5	6
1 Fuji Triage	No[ii]	No		n.s.	n.s.	0.007928*	0.000215*	n.s.
2 Fuji Triage	No	HC			n.s.	n.s.	0.008431*	n.s.
3 Fuji Triage	No	US				n.s.	0.000502*	n.s.
4 Fuji Triage	Cond	No					n.s.	n.s.
5 Fuji Triage	Cond	HC						n.s.
6 Fuji Triage	Cond	US						

* $p<0.01$, n.s. – non significant, [i] Cond – 10% polyacrylic acid conditioning, [ii] No – without treatment, HC – heat curing, US – ultrasound curing

TABLE 4
TUKEY HSD TEST FOR SHEAR BOND TEST OF IONOFIL MOLAR

Material	Cond[i]	Curing mode	1	2	3	4	5	6
1 IonofilM	No[ii]	No		n.s.	n.s.	n.s.	0.000040*	n.s.
2 IonofilM	No	HC			n.s.	n.s.	0.000174*	n.s.
3 IonofilM	No	US				n.s.	0.000036*	0.008510*
4 IonofilM	Cond	No					0.000036*	n.s.
5 IonofilM	Cond	HC						0.055881*
6 IonofilM	Cond	US						

* $p<0.01$, n.s. – non significant, [i] Cond – 10% polyacrylic acid conditioning, [ii] No – without treatment, HC – heat curing, US – ultrasound curing

TABLE 5
TUKEY HSD TEST FOR SHEAR BOND TEST OF FUJI IX GP FAST

Material	Cond[i]	Curing mode	1	2	3	4	5	6
1 Fuji IX	No[ii]	No		n.s.	n.s.	n.s.	0.000036*	n.s.
2 Fuji IX	No	HC			n.s.	n.s.	0.000036*	n.s.
3 Fuji IX	No	US				n.s.	0.000036*	n.s.
4 Fuji IX	Cond	No					0.000036*	n.s.
5 Fuji IX	Cond	HC						0.000060*
6 Fuji IX	Cond	US						

* $p<0.01$, n.s. – non significant, [i] Cond – 10% polyacrylic acid conditioning, [ii] No – without treatment, HC – heat curing, US – ultrasound curing

compared with standard cured specimens in the group with conditioned enamel. The fracture usually occurs in the glass ionomer cements, just above ion-exchange layer or interaction zone (Figure 2). For standard curing GIC groups without enamel conditioning, the failure mode was approximately equal of cohesive and adhesive.

Discussion

Heat treatment

It is generally believed that glass ionomer, particularly conventional GIC, bond to enamel by an ionic interaction with the mineral phase. However, the exact mechanism is not clear[16]. Recently, the latter technique was used to provide evidence for a chemical interaction between calcium and carboxyl ions for both human enamel and hydroxyapatite. Conventional glass ionomers bond to enamel, even with the presence of a smear layer, but surface conditioners have been found to improve the bond strength. Previous studies have shown that conditioners such as citric acid improve bond strength and PAA was also shown to be an effective conditioner[15,17,18]. In the current study, conditioners improved the bond strength of all tested GIC to enamel, in agreement with some previous studies. The role of the conditioner involves effective removal of the smear layer and provides good wetting of the surface by glass ionomer, an essential requirement for good bonding[19].

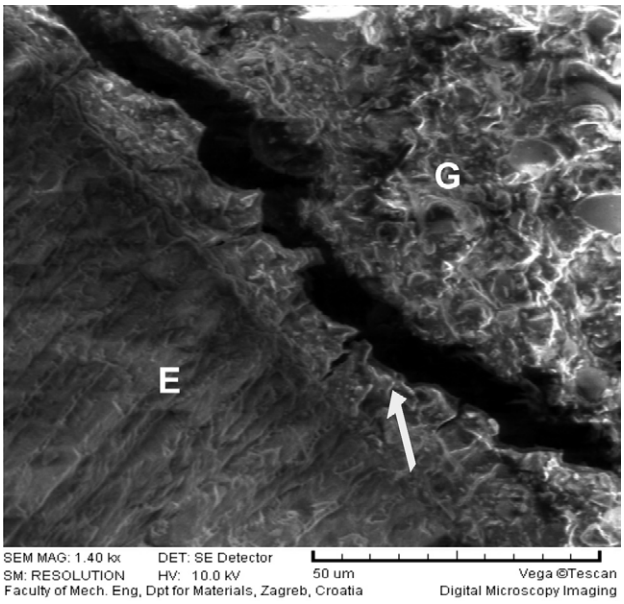

Fig. 2. Scanning electron micrographs of fractured test specimens prepared on enamel. The arrow shows failure which occurred in the glass ionomer cement. E-enamel, G-glass ionomer cement.

In the group of teeth without enamel pretreatment, conventional GIC should penetrate the smear layer through a self-etching process and affect a bond to the under laying enamel. The degree to which this occurs will influence the measured bond strength and the fracture mode[16]. In this study, in the group with pretreatment, approximately 93% of the bond failure was cohesive at the enamel surface. Explanation of this result relates to the removal of the smear layer by conditioning and the ability of the materials to interact with the under laying enamel and a stronger bond is achieved. Ion exchange layer or interaction zone is well attached to the surface and failure usually occurs above this layer.

Heat treatment applied to GIC in different in vitro studies was approximately 70–120 °C[3,4,20]. Improvement of the mechanical properties was observed in tested GIC, but that high temperature is not feasible in the real clinical situation. GC Europe N.V. was introduced sealing material Fuji VII and later Fuji Triage as material that uses heat as »command set« method in the clinical practice. In the same principle the heat generated by polymerization unit was applied in this study on two other chemically cured GIC. Measurement of the temperature rise on the surface of the material by the laser pointed thermometer revealed that the surface temperature of filling was between 2–3 °C, which can be clinically tolerant. Significant increase in bond strength of GICs to enamel was observed compared to standard chemical curing. Acidic conditioning is beneficial in achieving better bonding to enamel for conventional GIC. Significant problem in clinical practice might be temperature raise and influence of the heat on the pulpal tissue. Santini et al.[21] describe the pulp temperature rise during resin composite polymerization. Significantly higher temperature rise (3–7.1 °C) was established using LED units comparing to halogen unit (2.8–4.9 °C). According to Zach et al.[22] the pulp vitality can be compromised when the pulp temperature rise is 5–6 °C. But, it is difficult to apply these measured temperature rise in vivo. Blood flow through pulp tissue and absorption of the heat by gingival tissue is a mechanism of heat dissipation in vivo[21]. Also inclusion of the water in the GIC material prevents the pulp temperature rise. On the contrary, during bleaching procedure pulpal temperature rises up between 6 and 11.7 °C[23]. Skrinjaric et al. showed that heat treatment of GIC sealants has no influence on retention rate in clinical conditions[24]. Up to now, there is no study that uses heat in the same way, as »command set« method for GIC restoration, so it is difficult to comment and compare obtained results. It seems that changes in molecular kinetic energy due to elevated temperature lead to rearrangement of the molecules in the material during setting[20]. In spite of relatively modest increase of temperature on the surface of the filling, this molecular rearrangement can enable better adhesion of the material or achieving more stable zone of ionic exchange. The mechanical movement of the tip improves the mixing of the particles and polyalkenoic acid chains, resulting in homogenous reaction kinetics. As a consequence, the total reactive surface increases, which can enhance the setting time[20].

Ultrasound treatment

The effect of ultrasonic radiation in enhancing chemical reactions has been widely reported for the last 70 years. It is also shown that treatment of GIC during setting with ultrasound can improve mechanical properties of GIC. Ultrasound may contribute to acceleration of the reaction by de-clustering glass particles and enhancing the diffusion of the reaction components[3]. Several studies have confirmed the effect of ultrasound on GIC; both in respect of effect on the initial setting reaction and on the properties of the set cement[3,20]. The mechanism by which ultrasound enhances the setting rate of cements is not clearly established. Towler et al. suggest that, in glass ionomers, ultrasound may increase powder surface area by breaking up aggregates or breaking down glass particles and this may account for increased reactivity[14]. The effect ultrasound on GICs enhances the bonding to tooth surfaces[3] and release of fluoride[1]. Application of ultrasound in this study did not have significant influence on shear bond strength values of the tested materials. Obtained values were slightly higher compared to standard chemical curing of material but the difference was not statistically significant.

Conclusion

It can be concluded that adding energy to setting GIC i.e. temperature increase will improve shear bond strength. The results of this study indicate that heating of GIC with polymerization unit for 40s during setting increases

bond strength to enamel conditioned with 10% polyacrylic acid. The same improvement was achieved in group of GIC cured with ultrasound. An effective and improved bond between conventional GIC and tooth substance is important for the clinical success of restorations.

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UTJECAJ GRIJANJA I ULTRAZVUKA NA SNAGU VEZIVANJA STAKLENO IONOMERNOG CEMENTA

SAŽETAK

Svrha ovog istraživanja bila je usporediti utjecaj postupaka inicijacije stvrdnjavanja (grijanje, ultrazvuk) na snagu vezivanja nekoliko stakleno ionomernih cemenata (SIC) na caklinu. Vestibularne površine od 180 izvađenih premolara su polirane uz hlađenje vodom do postizanja glatke površine cakline i podijeljeni su u tri grupe. Tri restorativna SIC-a (Fuji IX GP Fast, Fuji Triage, Ionofil Molar AC) su tretirana na tri načina: standardno (SC), ultrazvukom (UC) i vanjskim izvorom topline (HC). U svakoj skupini, zubi su tretirani na dva načina: 30 s 10% poliakrilnom kiselinom i 30 bez kondicioniranja. SIC-ima su se ispunjavali teflonski kalupi (3x4 mm). Uzorci su testirani na aparatu za univerzalna ispitivanja (LRX Material Testing Machine) pri brzini glave od 1 mm/min. Rezultati pokazuju značajno veću snagu vezivanja toplinom tretiranog Fuji IX (13,3 MPa) na kondicioniranu caklinu od svih ostalih testiranih grupa (8,6 do 10,8 MPa) ($p<0,001$). Srednja snaga vezivanja SIC-a u SC grupi i bez kondicioniranja cakline je 3,6–5 Mpa, a time je i značajno niža čvrstoća vezivanja. Grijanje SIC-a povećava snagu vezivanja, poboljšava svojstva SIC ispuna i može se preporučiti za korištenje tijekom stvrdnjavanja materijala.