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Effect of delayed light-curing on solubility, color stability, and opacity of Fuji II LC glass ionomer cement: original article

Efeito da fotopolimerização tardia na solubilidade, estabilidade de cor e opacidade do cimento de ionômero de vidro Fuji II LC: artigo original

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ABSTRACT

Objective: Resin-modified glass ionomer (RMGI) cements are among the commonly used restorative materials in low stress-bearing areas and also for temporary restorations. The competition between acid-base reactions and light polymerization reactions in delayed curing of RMGIs can affect their physical and mechanical properties, as well as their degree of conversion. Since solubility, color stability, and opacity are among the main physical properties affecting the durability and clinical service of RMGI restorations, this study aimed to assess the effect of delayed curing on solubility, color stability, and opacity of Fuji II LC RMGI. **Material and Methods:** This in vitro, experimental study evaluated 80 Fuji II LC RMGI specimens (10 specimens per each in 4 groups) in terms of solubility, color stability, and opacity at 6 months later. Specimens were cured immediately or were cured with 1, 5 and 10 min delay. **Results:** Maximum solubility and minimum change in opacity and color stability at 6 months were noted in the group with delayed curing by 10 min. A significant difference was noted in the solubility of specimens cured after 10 min and 1 min. Significant differences were also noted in the opacity and color stability of specimens cured after 10 min and all other groups ($P < 0.05$). **Conclusion:** Delayed curing by 1 min decreased the solubility of RMGI specimens compared with immediate curing or curing after 5 min. Although this difference did not reach statistical significance. Color stability and changes in opacity are mainly influenced by the acid-base reactions rather than polymerization reactions.

KEYWORDS

Glass Ionomer cement; Fuji II LC cement; Solubility; Color; Opacity.

RESUMO

Objetivo: Cimentos de ionômero de vidro modificado por resina (CIVMR) estão entre os materiais restauradores mais comumente utilizados em áreas de baixa tensão e também para restaurações temporárias. A competição entre reações ácido-base e reações provenientes da fotopolimerização tardia dos CIVRMs podem afetar suas propriedades físicas e mecânicas, bem como seu grau de conversão. Uma vez que a solubilidade, estabilidade de cor e opacidade estão entre as principais propriedades físicas que afetam a durabilidade e o tempo de serviço clinico de restaurações de CIVMR, este estudo teve como objetivo avaliar o efeito da fotopolimerizaçao tardia na solubilidade, estabilidade de cor e opacidade do CIVMR Fuji II LC. **Material e Métodos:** Este estudo experimental in vitro avaliou 80 espécimes de CIVMR Fuji II LC (4 grupos com 10 espécimes cada) em termos de solubilidade, estabilidade de cor e opacidade apos 6 meses. As amostras foram fotopolimerizadas imediatamente ou com 1, 5 e 10 min de atraso. **Resultados:** Máxima solubilidade e mínima alteração na opacidade e estabilidade da cor em 6 meses foram observadas no grupo com fotopolimerização tardia em 10 min. Uma diferença significativa foi observada na solubilidade das amostras fotopolimerizadas após 10 min e 1 min. Diferenças significativas também foram observadas na opacidade e estabilidade de cor das amostras fotopolimerizadas após 10 min e em todos os outros grupos (P <0,05). **Conclusão:** A fotopolimerizaçao tardia em 1 min diminuiu a solubilidade das amostras CIVMR em comparação com a fotopolimerizaçao imediata ou após 5 min. Embora essa diferença não tenha alcançado significância estatística. A estabilidade da cor e as mudanças na opacidade são influenciadas principalmente por reações ácido-base, em vez de reações causadas pela polimerização.

PALAVRAS-CHAVE

Cimento de ionômero de vidro; Cimento Fuji II LC; Solubilidade; Cor; Opacidade.

INTRODUCTION

Glass ionomer restorative materials can be used for conventional restorative treatments in low stress-bearing areas such as the cervical region or in primary teeth, and also for temporary restorations (for caries control, or temporary filling of endodontic access cavity).

Resin components, and conventionally 2-hydroxyethylmethacrylate (HEMA), are added to glass ionomers to improve their properties. Resin-modified glass ionomers (RMGIs) have higher mechanical strength, higher bond strength to enamel and dentin, lower moisture sensitivity, and longer working time than the conventional glass ionomers [1]. Nonetheless, light-curing is never complete, and the degree of conversion (DC), which refers to the percentage of reduction of double bonds during polymerization, is 33% to 75% for RMGIs after 24 h [2]. The polymerization process of RMGIs includes three types of setting reactions namely (I) the acid-base reaction between the poly alkenoic acid and fluoroaluminosilicate glass particles, which is a slow reaction and starts upon mixing and continues for 24 h or even 1 week, (II) a faster reaction initiated by light irradiation, and, (III) self- cure polymerization, which II and III are related to the HEMA monomer polymerization.

It is believed that acid-base reactions can induce chemical bonding of glass ionomer to dentin, and serve as the main bonding mechanism of glass ionomers [3]. The polymerization reaction is based on the presence of monomers in the cement, their freedom of movement, and their diffusion, which are all affected by the volume of the previously cross-linked matrix subjected to acid-base reactions. Thus, it seems that one reaction can affect the rate and speed of the other reaction. Changing the time of initiation of polymerization can affect the balance between the acid-base reactions and polymerization reactions. For instance, if photo-activation is delayed, the frequency of acid-base reactions exceeds that of polymerization reactions; thus, the final properties of the set RMGI would be more similar to those of conventional glass ionomers. However, if polymerization is initiated earlier, the final properties of the set material would be closer to those of composite resins [4]. Previous studies have reported an increase in DC following a delay in initiation of light curing, which was maximally 10 min [1,4].

Solubility and water sorption of restorative materials are among their important clinical properties. High solubility and water sorption can lead to loss of marginal adaptation, debonding of restoration, recurrent caries, decreased surface properties, and compromised esthetics, causing restoration failure [5,6]. Optical properties (such as color stability and opacity) are among other critical factors for restorations in the esthetic zone. Color mismatch of restorations with the adjacent tooth structure can cause patient dissatisfaction and treatment failure. Considering the effect of delayed light curing on physical and mechanical properties of glass ionomer cements, and gap of information regarding the effect of delayed curing on solubility, color stability, and opacity of glass ionomers, this study aimed to assess the effect of delayed curing on the solubility, color stability, and opacity of Fuji II LC RMGI cement.

MATERIAL AND METHODS

This in vitro, experimental study evaluated the solubility, color stability and opacity of Fuji II LC RMGI cement.

Solubility

According to previous studies which used ISO 6876 for solubility of GI [7,8], 40 discs measuring 8 mm in diameter and 2 mm in thickness [9] were fabricated from RMGI. After mixing of the capsules in an amalgamator (Owzan, Iran) for 10 s, glass ionomer was injected into each mold using a gun. The mold was placed on a glass slide and filled with glass ionomer. Next, 4 mm of an 8-cm stainless steel ligature with 0.001-inch thickness (G&H orthodontics, Franklin, USA) was embedded in the glass ionomer for its suspension in distilled water. By doing so, all specimen surfaces were equally exposed to distilled water, and the solubility was measured more accurately. The two sides of the mold were covered with transparent Mylar strips to prevent adhesion to glass slide. Another glass slide was placed over the mold and gently pressed in order for the excess material to leak out, and eliminate the voids. Experiment was done in a dark room under precise controls. The specimens were light-cured with Optilux curing unit (Kerr, USA) with a light intensity of 850 mW/cm^2 for 20 s from each side in groups with immediate curing, and delayed curing by 1, 5 and 10 min after mixing [9]. Excess material was removed using 1000-grit silicon carbide abrasive paper. The dimensions of the specimens were measured by a digital caliper and they were then placed in a glass container (A) containing 50 mL of distilled water. The containers were incubated at 37°C for 6 months [10]. After this time period, the specimens were removed from the water, the water was filtered using a filter paper (Whatman filter paper, 42 Grade, Germany) and transferred into another glass container (B). The B containers were rinsed previously with distilled water, dried for 2 h, and placed in a desiccator for 20 min [10]. They were then weighed with a digital scale (Acculab, USA; AL-124, Max:120 g, d=0.1 mg). The weight was recorded as M1. After transferring the water from container A to container B, the container B was placed in an oven and heated at 100°C for 2 h. Next, they were placed in a desiccator for 2 h, and weighed with a digital scale. This weight was recorded as M2 [11]. The solubility of the specimens was calculated using the formula below:

$$M1\text{-}M2/S \qquad (1)$$

S: indicates the specimen surface area.

Color assessment

A total of 40 cylindrical specimens measuring 2 x 8 mm^3 were fabricated using stainless steel molds [9]. After mixing of the capsules in an amalgamator (Owzan, Iran) for 10 s, glass ionomer was injected with a gun into the mold and compressed between the two plastic Mylar strips and glass slabs with uniform pressure in order for the excess material to leak out and obtain a smooth and homogenous surface. Next, the specimens were cured with Optilux curing unit with a light intensity of 850 mW/cm^2 for 20 s from each side in immediate curing and delayed curing groups (1, 5 and 10 min). Light curing was performed at the center of each specimen from both sides. All specimens were placed in the dark prior to light curing. Next, excess material was eliminated with circular movements of 1000-grit silicon carbide abrasive paper under water coolant [9]. Each specimen was polished for 20 s, and they were then incubated at 37°C for 24 h. The L*, a*, and b* color parameters of specimens were then measured by a spectrophotometer (Ci64; X- Rite, Grandville, USA) against a white background. This device had an 8-mm aperture that measured the color parameters by light irradiation with a tungsten lamp under D65 lighting conditions at 8° for 2 s. The measurements were repeated 6 months after storage of specimens in distilled water, and the color change (ΔE) of each specimen was calculated using the formula below:

$$\Delta E^* = \cdot\left(\Delta L^{*2} + \cdot\Delta a^{*2} + \cdot\Delta b^{*2}\cdot\right)^{1/2} \qquad (2)$$

To test the opacity (Y), each specimen was analyzed by a spectrophotometer once against a black background and the second time against a white background. Y is a color parameter in color space. The opacity of specimens was calculated using the formula below:

$$\text{Opacity} = \cdot Y \cdot \text{ black background } / \ Y \text{ white background } x \cdot 100 \qquad (3)$$

Statistical analysis

Normal distribution of data was evaluated using the Kolmogorov-Smirnov test. One-way ANOVA was used to compare the groups. In case of presence of a significant difference, pairwise comparisons were performed using the Tukey's HSD test.

RESULTS

The Kolmogorov-Smirnov test confirmed the normal distribution of data.

Solubility

Table I presents the mean solubility of specimens in immediate and delayed curing groups (Figure 1). The range of weight loss was the same in immediate and 1 and 5 min delayed groups; 1 min delayed group showed lowest (0.0049 g/mm^2), and 10 min delayed group showed highest solubility (0.0060 g/mm^2). One-way ANOVA indicated a significant difference in solubility of the groups ($P = 0.001$). Pairwise comparisons of the groups by the Tukey's test revealed a significant difference in solubility only between the 1 min and 10 min groups ($P < 0.001$).

Color assessment

The color parameters and opacity of the specimens were measured to determine their color stability.

Measurement of ΔE

After the measurement of L*, a* and b* color parameters, ΔE was calculated according to the aforementioned formula. Table II presents the ΔE of the groups. Maximum change was noted in L (value). Minimum ΔE was noted in the 10 min group, while other groups showed almost similar ΔE values (Figure 2). The results of one-way ANOVA indicated a significant difference among the groups ($P < 0.01$). Pairwise comparisons of the groups by the Tukey's HSD test showed significant differences between the 10 min group and all other groups ($P = 0.001$).

Measurement of opacity

Table III presents the difference in the opacity measured immediately after curing and

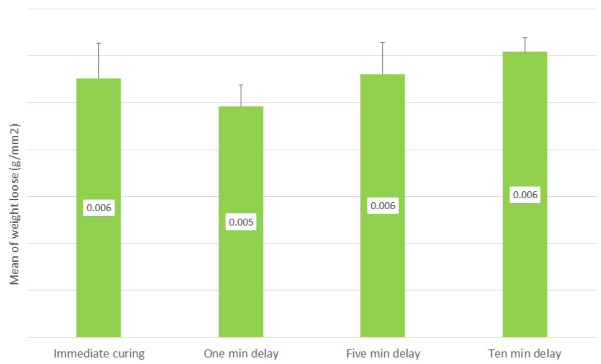

Figure 1 - Mean weight loss in experimental groups.

Table I - Solubility of each experimental group during 6 months

Time of Delay	Mean (g/mm$^{2)}$	Std. Deviation	Minimum	Maximum
Immediate	.005520	.0007436	.0044	.0070
1 min	.004920	.0004541	.0043	.0058
5 min	.005610	.0006806	.0044	.0070
10 min	.006080	.0003120	.0057	.0067

Table II - Mean values of ΔE measurements in experimental groups

Time of Delay	Mean	Std. Deviation	Minimum	Maximum
Immediate	5.2585	1.42126	3.62	7.01
1 min	5.2446	.96111	4.02	6.45
5 min	5.2711	1.10985	3.98	6.85
10 min	3.1286	.96907	1.85	4.53

Table III - Opacity difference between immediate after light-curing and six months later

Time of Delay	Mean	Std. Deviation	Minimum	Maximum
Immediate	.08680	.033634	.035	.139
1 min	.10943	.021745	.069	.137
5 min	.09321	.013962	.082	.127
10 min	.04752	.012741	.030	.073

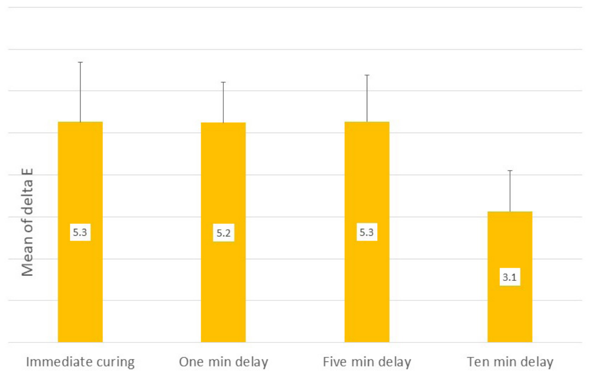

Figure 2 - Mean delta E in experimental groups.

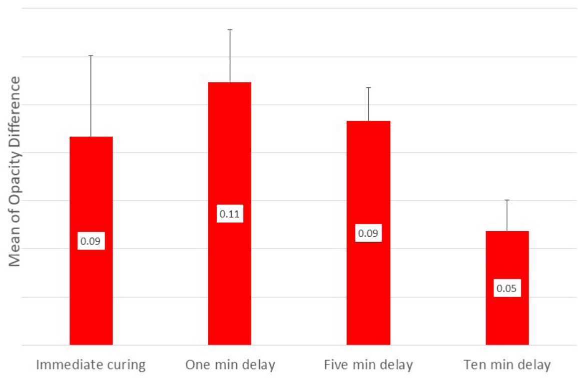

Figure 3 - Mean Opacity difference in experimental groups.

6 months later in the study groups (Figure 3). Minimum change in opacity was noted in the 10 min group while maximum change was noted in 1 min group. One-way ANOVA indicated a significant difference in this respect among the groups ($P < 0.001$). The Tukey's HSD test indicated significant differences between the 10 min group and all other groups ($P < 0.002$).

DISCUSSION

The interactions of acid-base and light polymerization reactions can affect the properties of RMGI cement. Nicholson and Anstice [12] and Wan et al. [13] were the first to suggest the theory of competition of acid-base reactions with visible light polymerization reactions, such that by delaying the curing of cement, acid-base reactions occur more frequently than polymerization reactions. As the result, the properties of the cement would be more similar to those of the conventional glass ionomers. However, in case of immediate curing of cement, its properties would be closer to those of composite resins [4].

Dursun et al. explained that delaying the curing by up to 10 min increased the DC due to phase separation by HEMA. HEMA induces the polymerization upon initiation of light-curing [2]. However, when curing is delayed for a longer time (over 10 min), polyacetate is formed, which limits the movement of HEMA monomers, and inhibits the formation of poly-HEMA, which decreases the DC. Gee et al. [14] evaluated the structural integrity of three RMGI cements with delayed or no photo-activation, and showed that despite the similarities in the structure of glass ionomers available in the market, different behaviors can be expected from them following different delayed curing protocols. Evidence shows that the physical properties of RMGI cements can affect the outcome of delayed curing and subsequent competition of acid-base and polymerization reactions.

Despite the relatively long-term use of RMGIs in dentistry, there is a gap of information regarding their solubility and color stability following delayed curing.

A previous study [1] reported that maximum mechanical properties at 24 h after curing were noted in the specimens cured with 5 min delay. Thus, delayed curing by 5-10 min can bring about maximum mechanical properties in short-term. However, this delay does not improve the mechanical properties in long-term.

Solubility

Water sorption and high solubility can lead to loss of marginal adaptation, debonding of restoration, recurrent caries, and decreased surface properties and esthetics, causing eventual failure of restoration [6,15]. Thus, solubility is an important factor for assessing the quality of a luting, base, liner, or restorative material.

Considering the moisture of the oral cavity, the results after water storage demonstrated that [1] shortest delay in curing yielded the best mechanical properties after 12 months. Decreased mechanical properties of specimens with delayed curing for 1 min can be due to the separation of polymer chains by the water molecules, leading to organic matrix softening due to hydrolysis. In 5, 10 and 15 min delays, the mechanical properties further decrease due to the lower rate of formation of poly-HEMA, which is in favor of hydrolysis.

In this study, encapsulated Fuji II LC RMGI was used. According to Table IV, poly acrylic

Table IV - Fuji II LC composition and filler content

Filler	Composition details
55 vol % FSG /	Powder:
Poly – HENA	Alumino-fluorosilicate glass 100%,
Average 5.9 µm	Liquid:
	polyacrylic acid,
	20-22%
	2-hydroxyethylmethacrylate(HEMA) 35-40%,
	2,2,4-trimethyl
	hexamethylene decarbonate 5-7%,
	triethylene glycol dimethacrylate 4-6%

acid is the main constituent of its liquid phase. According to the literature, glass ionomer cements with poly acrylic acid base have lower solubility than those with poly maleic acid base [3]. Also, it has been reported that the solubility of RMGIs depends on their DC and the ratio of resin in the composition of cement [16]. On the other hand, presence of HEMA hydrophilic molecules is responsible for higher water sorption and higher solubility [10]. According to Table IV, it accounts for 35-40% of the liquid content of the Fuji II LC capsules.

Another study [2] showed that DC and HEMA release had a direct correlation with delayed curing of cement and competition between the acid-base and polymerization reactions in encapsulated Fuji II LC RMGI cement. Delayed curing significantly increased the release of HEMA. DC significantly increased by delayed curing, and maximum DC was recorded following delayed curing by 10 min, and then decreased. No significant correlation was noted between the monomer release and DC. Savas et al. [9] confirmed that the composition of glass ionomer cements plays a key role in their solubility/water sorption in different environments.

In this study, immediate curing and 1 and 5 min delayed curing groups experienced the same range of weight loss. The 1 min delayed group showed minimum and the 10 min delayed group showed maximum solubility among all (Table I). However, a significant difference in solubility was only found between the 1 min and 10 min groups. Since no significant difference has been found between immediate curing and other curing protocols [2], it may be assumed that delayed curing does not significantly improve the clinical outcome of cements for solubility. However, it has been suggested that delayed curing by 1 min can insignificantly decrease the solubility of cement. Several factors can affect the results such as the sample size, presence of salivary enzymes in the oral environment, presence of thermal cycles in the oral environment (thermocycling), and pH alterations following the consumption of acidic or alkaline foods. Thus, future studies are required to take into account the role of these parameters in the solubility of cements [17].

Color assessment

Color match of dental restorations is an important parameter in their clinical success, particularly in the anterior region and visible areas (cervical restorations in patients with high lip line) [18]. Color of restorations is affected by their composition and exposure to moisture [9].

In this study, minimum color change was noted in the 10 min group (3.1) while other groups showed almost equal color change (around 5.2) (Table II). The difference in this respect was significant between the 10 min group and all other groups.

$\Delta E \leq 3.7$ is clinically acceptable according to Johnson and Kao [19]. In immediate curing and 1 and 5 min delay groups, the mean ΔE was higher than 3.7, and the color change was therefore clinically visible. However, in 10 min delay group, ΔE was lower than 3.7. Thus, only the 10 min delay group showed insignificant color change; while the color change was significant in other groups.

To the best of the authors' knowledge and literature search, no study has assessed the effect of delayed curing on color stability of glass ionomer cements. Inokoshi et al. [20] evaluated the color stability of encapsulated Fuji II LC glass ionomers within 4 weeks of storage in distilled water at 60°C. They calculated $\Delta E=13$, and reported a reduction in value (lightness) in addition to color change of RMGIs. However, the mean ΔE in our study was 3.1-5.2 and the color change in our study was mainly due to the reduction in value (-3.97). Difference between their results and ours can be due to the fact that they did not assess color stability following delayed curing. They only assessed the color stability of specimens after 4 weeks of immersion in distilled water at 60°C while we assessed the color stability of specimens after 6 months of incubation at 37°C.

Opacity

Translucency and opacity are two sides of a coin. Translucency has a pivotal role in esthetic considerations. Materials with higher opacity have lower passage of light, and therefore, mask the underlying defects such as old and discolored restorations, brown arrested caries, and cast cores. Thus, preserving the opacity over time can play an important role in success of tooth-colored restorations. To date, no study has assessed the changes in opacity of glass ionomer cements following delayed curing. In this study, as shown in Table III, all groups showed a reduction in opacity, and minimum change in opacity was noted in 10 min group while maximum change in opacity was noted in 1 min group. The difference in this respect was significant between the 10 min group and all other groups. However, other groups were not significantly different in this respect during the 6-month study period. In line with our findings, Inokoshi et al. [20] reported a sudden reduction in opacity and higher translucency of Fuji II LC conventional and encapsulated cements after 4 weeks of water storage at 60°C. As a clinical point, considering the inherent reduction in opacity of glass ionomers, they suggested that clinicians should select a lighter shade of glass ionomer since it would become darker over time; this is particularly important in immediate curing with shorter delay.

One of advantages of our study was using of capsular form of Fuji II LC. Several factors affect the physical properties of glass ionomer cements such as the powder to liquid ratio, the concentration of poly acid, and size of powder particles [21]. The advantages of encapsulated glass ionomers include optimal powder to liquid ratio, easy application, less void formation, and more appropriate mixing time.

Our results reveal that 1 min delay curing can be suggested to clinician because of more solubility resistance in comparison to more time of delay. Although 10 min delay curing showed higher color and opacity stability. This time of delaying seems be unpractical because of moisture control and isolation consideration, patient and practitioner comfort (chair-time). In critical esthetic situation, GI restorations can be alternated by composite one or GI can be covered by a composite layer (closed sandwich technique).

It should be noted that the current results were obtained in vitro and in absence of the effects of oral enzymes, microbial flora, and clinical conditions. Also, absence of a significant difference in solubility or optical properties of glass ionomers may be due to the small sample size or absence of confounders. Increasing the sample size and taking into account the effect of confounding factors such as the clinical oral conditions may change the results. Thus, future studies with a larger sample size are required to take into account the confounding factors.

CONCLUSION

A 1-min delay in curing of RMGI cement had insignificant higher solubility resistance compared with other late curing protocols. The results revealed that color stability and changes in opacity are mainly influenced by the acid-base reactions rather than the polymerization reactions of RMGI cement.

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Not attainable.

Authors' Contributions

MN: conceptualization and methodology, data curation, writing, original draft preparation; NP: conceptualization and methodology, writing, original draft preparation; AG, HT: conceptualization and methodology.

Conflict of Interest

The authors have no proprietary, financial, or other personal interest of any nature or kind in any product, service, and/or company that is presented in this article.

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Regulatory Statement

The study is an invitro experiment, University ethical committee evaluated the project and No IR.SBMU.DRC.REC.1398.124 code was assigned for it.

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