The effects of shelf life on the compressive strength of resin-modified glass ionomer cement

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Abstract. Resin-modified glass ionomer cement (RMGIC) is a restoration material composed of powder and liquid whose stability is affected by its shelf life. This is an issue that has not been taken into consideration by customers or sellers. To observe the effects of shelf life on the compressive strength of RMGIC, 30 cylindrical (d = 4mm and t = 6mm) specimens of RMGIC (Fuji II LC, GC, Tokyo, Japan) were divided into three groups with different storage times and their compressive strength was tested with a universal testing machine. Results were statistically analyzed with the one-way ANOVA test. There were significant differences (p<0.05) between the three groups of RMGIC. There is a decrease in the compressive strength value along with the duration of storage time.

1. Introduction
Over the past few decades various types of dental restoration materials have been developed. At this time, biocompatible and tooth-colored restorations are the most desirable. Glass ionomer cement is an example of a biocompatible and tooth-colored restoration material. Glass ionomer cement (GIC) is formed from the reaction between polyalkenoate acid liquid and aluminosilicate powder. GIC has several advantages including the ability to form a chemical bond with the tooth structure. It also has a coefficient of thermal expansion that is similar to the tooth structure. Additionally, its ability to release fluoride plays a role in the prevention of caries. Therefore, it is frequently used with patients who have a high risk of caries [1]. GIC has low mechanical properties and is sensitive to moisture. In 1988, GIC was modified to develop a new material called resin-modified glass ionomer cement (RMGIC). The difference in composition between GIC and RMGIC is due to the addition of a hydrophilic monomer called hydroxyethyl methacrylate resin (HEMA). Moreover, a photoinitiator is used to generate an acid-base reaction and the polymerization of resin and light. RMGIC was developed to overcome the shortcomings of GIC while maintaining the advantages of conventional GIC [1]. Some studies have shown that RMGIC has better mechanical properties than conventional GIC [2]. It is important to ensure that the mechanical properties of the material are strong enough to withstand the mastication load [3]. One of the most important mechanical properties of RMGIC is compressive strength [2]. Compressive strength is the ability of a material to withstand the force that leads to fractures [1]. Compressive strength is needed to bear the force in the mastication process [1].

The mechanical properties of RMGIC can be affected by various factors such as temperature and humidity [4]. Most factories have been set up to ensure that their packaging and storage procedures are in accordance with temperature and humidity standards in order to maintain the quality of materials. However, the conditions during storage and delivery are not always ideal. This can affect the shelf life...
of the materials [5]. Shelf life is the period during which the material will maintain its physical and mechanical properties [6]. In addition to temperature and humidity, shelf life can also be affected by the duration of storage. Hondrum showed that there was a decline in the compressive strength of conventional GIC that was stored past its expiration date [7]. Currently, RMGIC which is nearing its expiration date is still on the market. This can affect the quality of materials. Therefore, research on the effects of shelf life on the compressive strength of RMGIC should be performed. Hopefully, through this research the effects of shelf life on the compressive strength of RMGIC can be identified.

2. Materials and Methods

30 specimens were divided into three groups of tubes with a diameter of 4 ± 0.1 mm and a height of 6 ± 0.1 mm, then filled into a stainless steel mold in accordance with ISO standard 9917-1:2007. Resin-modified glass ionomer cement was used from a sealed package with the following specifications: LOT Number, 2 digits indicating the production year, 2 digits indicating the production month, 2 digits indicating the production date, and the last digit indicating the batch number.

| No. | LOT Number | Production Date       | Expiration Date | Description                                                   |
|-----|------------|-----------------------|-----------------|---------------------------------------------------------------|
| 1   | 1503061    | March 6, 2015         | March 2017      | 6 months after the production date (1 year 6 months before the expiration date) |
| 2   | 1402111    | February 11, 2014     | February 2016   | 1 year 8 months after the production date (4 months before the expiration date) |
| 3   | 1311051    | November 5, 2013      | November 2015   | 1 year 11 months after the production date (1 month before the expiration date) |

The research was done in an experimental research laboratory. The specimens used were made of resin-modified glass ionomer cement (Fuji II LC, GC, Tokyo, Japan). The specimens were manipulated with 0.32 grams of powder and 0.1 gram of liquid. Once the specimens were homogeneous, they were filled into a mold using a plastic filling and then light cured using a Light Curing Unit (LED Hilux Max) with an irradiance of 500 mw/cm² for 20 seconds. The filling and light curing were done incrementally for each 2 mm layer. The mold was covered with a plate and then put in the clamp. The specimens were removed from the mold after 1 hour and both ends of the specimens were honed with 400 grit sandpaper. After 24 hours, the specimens were put into Aqua Des and then incubated at a temperature of 37°C for 24 hours. The compressive strength test procedure was done using the Shimadzu Autograph AG 5000E Universal Testing Machine. It was performed 24 hours after mixing. The specimens were placed in an upright position with a compressive load of 250 Kgf and a speed of 1 mm/min. Compressive strength was examined using the following formula:

\[ C = \frac{4P}{\pi d^2} \]

\( C = \) compressive strength (MPa)
\( P = \) maximum pressure (N)
\( d = \) specimen diameter (mm)

The statistical analysis was performed using SPSS and began with the Saphiro-Wilk normality test and then the one-way ANOVA signification. A post hoc test was subsequently performed using the Least Significant Difference (LSD) method to identify the significant difference between the groups.
3. Results and Discussion

3.1 Results
In this study, the compressive strength test was performed in three groups of resin-modified glass ionomer cement specimens with sealed packaging in accordance with ISO standard 9917-1:2007. The groups were divided according to the duration of storage after production. The first group was a control group and consisted of 10 specimens of RMGIC with 6 months of storage time after production. The second group consisted of 10 specimens of RMGIC with 1 year 8 months of storage time after production. The third group consisted of 10 specimens with 1 year 11 months of storage time after production. The compressive strength test was performed using the Shimadzu Autograph AG 5000E Universal Testing Machine. The mean of the compressive strength value in each group can be seen in Table 2.

Table 2. Mean of the compressive strength value and the standard deviation in three groups of specimens

| Group of specimens | Production date | Expiration date | Number of specimens | Mean of compressive strength (MPa)±SD |
|--------------------|-----------------|-----------------|---------------------|--------------------------------------|
| I (6 months)       | March 6, 2015   | March 6, 2017   | 10                  | 150.74 ± 9.46                      |
| II (1 year 8 months) | February 11, 2014 | February 11, 2016 | 10                  | 140.08 ± 9.95                      |
| III (1 year 11 months) | November 5, 2013 | November 5, 2015 | 10                  | 91.75 ± 5.93                       |
| ISO 9917-1:2007    |                 |                 |                     | 100                                 |

*p < 0.05

Table 2 shows that there was a decrease in the compressive strength value along with the duration of storage and when nearing the expiration date. In group III, there was a decrease in the compressive strength value beyond the minimum standards of ISO 9917-1:2007.

Figure 1. Mean of compressive strength of RMGIC
Figure 1 shows the different values of compressive strength between the RMGIC groups. There was a decrease in the compressive strength value along with the duration of storage. In this study, the Saphiro-Wilk and Levene statistical tests were performed to identify data normality. A normal data distribution and homogeneity was obtained for each test. In the three groups of RMGIC the one-way ANOVA was used to identify the significant difference between the groups. The value of significant difference between the RMGIC groups can be seen in Table 3.

### Table 3. The result of the one-way ANOVA test in the RMGIC groups

| Group of specimen | Production date | Expiration date | Group of specimen | Sig.  | Description               |
|-------------------|----------------|----------------|-------------------|-------|---------------------------|
| I (6 months)      | March 6, 2015  | March 6, 2017  | II (1 year 8 months) | 0.010 | Significantly different   |
|                   |                |                | III (1 year 11 months) | 0.000 | Significantly different   |
| II (1 year 8 months) | February 11, 2014 | February 11, 2016 | I (6 months) | 0.010 | Significantly different   |
|                   |                |                | III (1 year 11 months) | 0.000 | Significantly different   |
| III (1 year 11 months) | November 5, 2013 | November 5, 2015 | I (6 months) | 0.000 | Significantly different   |
|                   |                |                | II (1 year 8 months) | 0.000 | Significantly different   |

Based on an analysis of the one-way ANOVA, a comparison of the compressive strength value in the three groups showed a statistically significant difference with a significance value of 0.010 (p<0.05) between group I and group II. Between group I and group III and between group II and group III, there was a statistically significant difference with a significance value of 0.000 (p<0.05).

### 3.2 Discussion

The material used in this research was a resin-modified glass ionomer cement manufactured by Fuji II LC (GC, Tokyo, Japan). The powder and liquid were packaged well, sealed, and had never been opened. The three groups of specimens consisted of resin-modified glass ionomer cement with 6 months of storage time after production, 1 year 8 months of storage time after production, and 1 year 11 months of storage time after production. This research compared the compressive strength of resin-modified glass ionomer cement based material to the duration of storage. The compressive strength test was conducted in accordance with ISO standard 9917-1:2007. In this research, the results showed different compressive strengths in three groups of RMGIC with different storage times. There was a decrease in compressive strength value in RMGIC with 1 year 8 months of storage time after production and 1 year 11 months of storage time after production. The research showed that longer storage times for resin-modified glass ionomer cement result in a decrease in its compressive strength value. In this study there was a physical change in the viscosity of RMGIC with 1 year 8 months of storage time after production and 1 year 11 months of storage time after production. The liquid in both groups was more viscous than the RMGIC group with 6 months of storage time after production. The change in viscosity was caused by the evaporation of water. This finding is supported by Hondrum (1999) whose research showed that the evaporation of liquid over time could increase the viscosity of the fluid [7]. A component of RMGIC that can be affected by the duration of storage is its liquid content [8]. The liquid of RMGIC contains 40-50% water. Water acts on the material by penetrating particles of powder and releasing ions such as calcium and aluminum which play a role in the setting reaction [3]. Water will evaporate in glass ionomer cement that is stored too long. This can result in reduced water components and the release of calcium and aluminum ions that will crosslink with polyalkenoate and cause the setting reaction to decrease [9]. Water deficiencies result in an increase in the viscosity of the liquid [7]. According to Philips (2003), liquid tends to become a gel over time [8].
Viscosity is a benchmark in determining the stability of water-based cement. Hondrum (1999) found that there was an increase in the viscosity of the liquid as well as a decrease in the strength of the water-based cement over time [7].

There is also tartaric acid in the liquid that can be affected by the duration of storage. Tartaric acid helps to increase the material’s working time [8]. A decrease in the functioning of the tartaric acid could reduce the working time. According to Winkler (2003), a decrease in the working time of resin-modified glass ionomer cement occurs a month before it expires [10]. Tartaric acid also acts as an accelerator to assist in releasing ions in fluorooaluminosilicate powder [1]. The setting reaction of RMGIC is dominated by an acid-base reaction. In addition, there is a polymerization of the monomer resin which is activated by the light [3]. A photoinitiator is a component in the liquid that initiates polymerization and is activated by the light. According to Tirapelli (2004), a photoinitiator could cause over polymerization and setting of the resin becoming less optimal [11]. Other factors that can affect the compressive strength of RMGIC are temperature and humidity. High temperature may result in a decline of material properties before the expiration date [4]. Based on ISO standard 9917-1:2007, the expiration date on packaging is referable if the storage condition is at a temperature of 23 ± 2 °C and a humidity of 50 ± 10% [12]. According to Cardoso et al. (2014), most factories have been set up to operate in accordance with this standard, but the conditions during delivery and storage are not always ideal [6].

According to Algera (2006), an increase in temperature can lead to evaporation of the liquid in GIC [4]. A decrease in compressive strength is caused by a failure of the liquid to ionize perfectly due to water loss [7]. The change of composition in liquid could interfere with the setting reaction and thereby decrease the strength of RMGIC [4]. Temperature, humidity, and storage time are factors that can affect the shelf life of the material [1]. Resin-modified glass ionomer cement which is exposed to temperature and humidity that is not in accordance with the standard could deteriorate. The deterioration of the material indicates that the material has past its shelf life. This has an impact on the composition of the liquid in RMGIC that can affect the setting reaction, physical and mechanical properties of RMGIC. The compressive strength of RMGIC with 1 year 8 months of storage time and 1 year 11 months of storage time decreased in comparison with RMGIC with 6 months of storage time. This indicates a change in the quality of RMGIC based on storage time. A decrease in compressive strength could cause the materials to become more prone to fractures. In the RMGIC group with 1 year 8 months of storage time, the compressive strength value was 140.08 MPa. This group showed a decline compared with the group with 6 months of storage time. The group with 6 months of storage time had a compressive strength value of 150.74 MPa. However, RMGIC with 1 year 8 months of storage time can still be used since it corresponds to the minimum of 100MPa based on ISO standard 9917-1:2007. The group with 1 year 11 months of storage time also had a decrease in compressive strength with a value of 91.75 MPa. That group was already beyond the minimum standard of ISO 9917-1:2007. According to Hondrum (1999), these changes will not be immediately apparent in a clinical setting, but can have an impact on the shelf life of materials [7].

4. Conclusion
In conclusion, this study showed that there is a decrease in the compressive strength value of resin-modified glass ionomer cement with 1 year 8 months of storage time and 1 year 11 months of storage time after production. This study also showed that there is a difference in the compressive strength of resin-modified glass ionomer cement with 6 months of storage time, 1 year 8 months of storage time, and 1 year 11 months of storage time after production. Dental practitioners are advised to be more careful in selecting resin-modified glass ionomer cement based on the production date and the expiration date. This is due to a decrease in the compressive strength of resin-modified glass ionomer cement which is near the expiration date. In addition, it is necessary to do advanced research on the effects of shelf life toward other mechanical properties such as the tensile strength of resin-modified glass ionomer cement.
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