Influence of shelf life on the setting time of type IV gypsum

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Abstract. Although expired materials can exhibit a deterioration in their properties, expired type IV gypsum can still be found on the market. In order to evaluate the influence of the shelf life on its setting time, two groups of type IV gypsum (GC Fuji rock EP) with different expiration dates were used in this research. The setting time tests were done in a mold using a Vicat Needle apparatus. The results of the statistical analysis showed a significant difference (p<0.05) between the two different expiration date groups. Therefore, the shelf life did influence the setting time of the type IV gypsum.

1. Introduction
The general treatments for most tooth and mouth problems are restorations, crowns, and bridges. The crown and bridge fabrication processes require specific materials, including dental gypsum, which is widely used in dentistry. In addition, a dentist must duplicate the condition of patient’s jaw and teeth to establish a diagnosis and determine a treatment plan, so gypsum is often used to create study models for diagnostic purposes, as well as to make dies for casting [1-2]. Type IV dental gypsum has more solid crystals than type III dental gypsum, so it has greater accuracy, strength, and abrasion resistance. Therefore, this type of gypsum is often used to create a model in the fabrication of inlays, crowns, and bridges [2-4]. Overall, the gypsum should exhibit several properties, including a good compressive strength, setting expansion, detail reproduction, and setting time. The setting time is the time that the material requires to react, and an exact setting time is one of the most important characteristics of gypsum, because it can affect the overall working time of the dentist or lab technician [3].

Gypsum powder must be packed in a safe and sealed container, because dampness can change the nature of the gypsum. In American Dental Association (ADA) Specification No. 25 (1989) and International Organization for Standardization (ISO) 6873:1998, one piece of information that must be written on the packaging is the expiration date [3]. Currently, there are many gypsum products on the market that are not in accordance with the ISO standards. In addition, there are gypsum products that are being sold after a long period of storage, near the expiration date, and passed the shelf life.

Other dental materials have also been showed to experienced property changes affected by shelf life [5-9]. When ingredients have passed their shelf lives, they are no longer able to function properly, because there can be changes in the properties of the materials [5,6]. Based on the research by D’Alpino et al., resin composites that have passed their expiration time exhibit a reduced flexural strength value [6]. According to Hondrum et al., alginate which has been stored for a long time shows an increase in the strength, but a decrease in the elasticity, recovery from deformation, and creep compliance of the ingredients [7]. Research conducted by Sinaga et al. indicated that there was an increase in the hardness value of glass ionomer cement that had passed its shelf life, so it became more

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fragile [8]. Another study reported that there was a decrease in the diametral tensile strength in resin composites that had passed their shelf lives [9].

In addition to the shelf life, the climate in Indonesia, which has relatively high humidity, can influence hygroscopic materials like gypsum during the storage and manipulation processes. Exposure to damp conditions can accelerate hardening, but over the long-term it can prolong the setting time of the gypsum. However, many dentists do not care which gypsum materials are used, even though the material could be in poor condition because it has passed its shelf life [1,6,10]. Therefore, further research is needed regarding the influence of the shelf life on the setting time of type IV dental gypsum. Through this research, changing properties are expected to be seen.

2. Materials and Methods
This was a descriptive laboratory research study in which 20 specimens were divided into 2 groups. Group A consisted of type IV gypsum (GC Fujirock EP™) tested ten months after the production time (December 2014), and group B consisted of the same type of gypsum tested 2 years and 3 months after the production time (July 2013). The temperature and humidity of the room during testing were 24.8±0.5 °C and 55±3%, respectively. Distilled water was poured into a rubber bowl, and the gypsum powder was added for 10 seconds (beginning when the gypsum powder first made contact with the water). The mixture was stirred with a spatula for 1 minute until it reached a soft consistency. It was then poured into a mold (70 mm base diameter, 60 mm top diameter, and 40 mm in height) according to ISO 6873:1998 [3]. One or two minutes before the expected setting time, the Vicat Needle apparatus was removed, after penetrating the dough every 15±1 seconds. The position of the mold was changed so that the needle could penetrate anew area a minimum of 5 mm from the mold wall and the previous location. The needle tip was cleaned and positioned to make contact with the dough surface, then it was locked into position. At the appropriate time, the scale was read and the rod was removed. Then, the depth of the needle penetration was recorded. The setting time was noted starting from the beginning of stirring until the needle could not penetrate the specimen at least 2 mm deep.

3. Results and Discussion
3.1 Results
The results of the setting time tests using the Vicat Needle, ADA Specification No. 25, ISO 6873:1998, and factory standards from the packaging for comparison can be seen in Table 1.

| Group name                | Average setting time±SD (minutes) |
|---------------------------|-----------------------------------|
| ADA Specification No. 25  | 12±4                              |
| ISO 6873:1998             | 11.30±2.18                        |
| GC Factory Standard      | 11.30±1.30                        |
| A                        | 18.44±1.16                        |
| B                        | 17.30±0.47                        |

A: Type IV gypsum with a production time of December 2014
B: Type IV gypsum with a production time of July 2013

It can be seen in Table 1 that the type IV gypsum in group B, with a longer period of time from production to use, had a faster setting time than that in group A. Additionally, it can be seen that both groups had longer setting times than the factory standard, ISO 6873:1998, and ADA Specification No. 25. Comparisons between both study groups and the three standards can be seen in Figure 1.
Before the unpaired t-test was conducted for the two type IV gypsum groups, the data was first analyzed with regard to its normality and homogeneity. The results of the Shapiro-Wilk’s normality test showed that the data distribution of the two groups’ setting times was normal, with statistically significant values of 0.402 and 0.430 (p>0.05). A homogeneity test was also done to determine the variance of the two groups’ data. Based on these tests, it was determined that the variance was homogeneous, with a significance value of 0.242 (p>0.05). The averages of the two groups were tested using an unpaired t-test to determine the significance value of the difference between the two groups, with a confidence interval(α) of 0.05 (Table 2).

Based on the results of the unpaired t-test between the two type IV gypsum groups, the significance value was 0.18. There was a significant difference between group A (the type IV gypsum that was tested ten months after production, with an average setting time of 18 minutes and 44 seconds), and group B (the type IV gypsum that was tested 2 years and 3 months after production), with an average setting time of 17 minutes and 30 seconds.

3.2 Discussion
The results of this research showed that both groups had longer setting times than those of the factory standard, ISO 6973:1998, and ADA Specification No. 25. Different results were found in the research by Lucas et al. (2008), which determined that in a control group tested using the standard method and conditions, the setting time often minutes was still in accordance with the standard [11]. Several factors can affect the hardening of gypsum, including the spatulation or stirring process, pH, temperature, and humidity [1]. A setting time test result that is not in accordance with the standard can be expected when the mixing process does not follow the factory recommendations and ADA Specification No.25. Moreover, the mixing speed in this research may not have reached the ADA Specification No. 25 recommended mixing speed, which is 120 rpm. This was because the type IV gypsum properties, requiring only a small amount of water, could have created obstacles to the mixing process.

The stirring process can influence the overall setting time. According to Azer et al., two different mixing methods, a vacuum mixer and mixing manually by hand, did not show a significant difference between the setting times [12]. However, the speed of the manual stirring process in this research was slower, which could prolong the setting time. When the calcium sulfate hemihydrate powder makes contact with water, a chemical reaction occurs and calcium sulfate dehydrate is formed. During the
mixing process, the calcium sulfate dehydrate is divided into smaller crystals and forms new center core. The more the mixing speed or duration is improved, the more nuclei are formed, so that the conversion from hemihydrate to dihydrate increases more quickly; therefore, the setting time is faster [1]. However, in this research, the mixing speed was slower than that of the standard, so that there was less core formation and the setting time was longer, when compared with the factory standard and ADA Specification No. 25. In this study, there was a significant difference between the mean setting times of the type IV gypsum groups with production dates of December 2014 and July 2013. There was an acceleration of the setting time in the type IV gypsum with the longer period of time since production.

The humidity in which the gypsum is stored, as well as the delivery conditions, can affect the setting time, along with the period of time that has passed since production. The humidity of the storage space can be influential because gypsum is hygroscopic, which means that the gypsum particles easily absorb water vapor from the air. Based on the research by Torrance et al., calcium sulfate hemihydrate stored in an airtight container runs a greater risk of converting to dihydrate when exposed to relatively high humidity [13]. Hydration can occur and some of the crystal hemihydrate will form dihydrate deposits. Setting is faster when there are already dihydrate deposits in the gypsum [14]. Group B was produced first, so it passed through a longer storage time and more dihydrate deposits could have formed in the packaging, causing a faster setting time. Materials nearing their expiration dates will experience changes in their quality, depending on the properties of the materials. The setting time of the type IV gypsum produced in July 2013, which was faster than that produced in December 2014, exhibited this difference in the material quality [5,6]. However, the setting times of the two groups in this research were longer when compared with the factory standards, ISO recommendations, and ADA Specification No. 25. This could have been due to the overall quality of the type IV gypsum used in this study.

A previous study by Eriwati et al. tested the setting times of some of the type IV gypsum brands available on the market at a relatively higher temperature and humidity than the ISO 6873:1998 recommendations, according to the situation in Jakarta, Indonesia. That research showed that the setting time of the GC factory type IV gypsum was longer than the standard, and longer than the setting times of the other brands [14]. This study did have some limitations; for example, the above analysis showed that the longer that gypsum is stored, the faster its setting time becomes. However, even though the gypsum may not have passed its expiration date, long-term storage with a longer exposure to humidity may have caused a longer setting time. In addition, the mixing method in this research did not follow the recommendations from the factory, so further research is required using the factory or ADA Specification No. 25 mixing methods. In addition, the influence of the shelf life on the other type IV gypsum properties, such as the detail reproduction and setting expansion, were not examined. Therefore, further research is required with regard to the overall influence of the shelf life on the properties of type IV gypsum.

4. Conclusion
The results of this research showed that type IV gypsum with different production dates (December 2014 and July 2013) exhibits different setting times. Therefore, it can be concluded that the shelf life can affect the setting time of type IV gypsum. It is recommended that the dentist or lab technician pay more attention to the shelf life of the gypsum materials that will be used. Further research is needed with regard to the right mixing methods and stirring processes, following the factory recommendations or ADA Specification No. 25. Moreover, the materials should be traced to determine the origins and storage conditions before they are used in the research.
References
[1] Sakaguchi R L and Powers J M 2012 *Craig's Restorative Dental Materials.* (Philadelphia: Elsevier Health Sciences) p 300-8.
[2] Bonsor S J, Pearson G 2012 *A Clinical Guide to Applied Dental Materials* E-Book (Philadelphia: Elsevier Health Sciences) p. 348-355
[3] Freitas C A, Zanotti T S, Rizzante F A B, Furuse A Y, Freitas M F A 2014 Linear Setting Expansion of Different Gypsum Products. *RSBO.* 12(1):61-7
[4] Da Silva M A B, Vitti R P, Consani S, Sinhoreti M A C, Mesquita M F and Consani R L X 2012 Linear Dimensional Change, Compressive Strength and Detail Reproduction in Type IV Dental Stone Dried at Room Temperature and in a Microwave Oven. *J. Appl. Oral. Sci.* 20 588–3.
[5] Tirapelli C *et al.* 2004 Radiopacity and microhardness changes and effect of X-ray operating voltage in resin-based materials before and after the expiration date. *Mater. Res.* 7 409-12.
[6] D'Alpino P H P *et al.* 2014 Resin composite characterizations services as following a simplified protocol of accelerated aging as a function of the expiration date. *J. Mech. Behav. Biomed. Mater.* 35 59-69.
[7] Guiraldo R D, Berger S B, Consani R L X, *et al.* 2014. Characterization of Morphology and Composition of Inorganic Fillers in Dental Alginates. *BioMed. Res. Int.* 2014 1-6.
[8] Sinaga K L 2010 *The influence of the shelf life of violence glass ionomer cement type II.* Bachelor Theses Paper (Jakarta: The Library of the Faculty of Dentistry University of Indonesia).
[9] Katherine 2010 *The influence of the shelf life of the strength of the pull diametral resin composites.* Bachelor Theses Paper (Jakarta: The library of the Faculty of Dentistry University of Indonesia).
[10] Climate of the World: Indonesia - UK – Weather online Weather.co.uk (Internet) 2015 [cited 2015 Jul 4]. Available from: http://www.weatheronline.co.uk/reports/climate/Indonesia.htm.
[11] Lucas M G, *et al.* 2009 Effect of incorporation of disinfectants solutions on the settings time, linear dimensional stability, and details of the reproduction in dental stone casts. *J. Prosthodont.* 18 521-6.
[12] Azer S S, Kerby R E and Knobloch L A 2008 Effect of one mixing methods on the physical properties of dental stones. *J. Dent.* 36 736-44.
[13] Chan T K and Darvell B W 2001 Effect of storage conditions on calcium sulphate hemihydrate-containing products. *Dent. Mater.* 17 134-41.
[14] Eriwati, *et al.* 1998 Evaluation of W/P ratio, setup time and compressive strength of dental stone type III and IV marketed in Jakarta. *J. Dent. Indones.* 5 25-34.