ORIGINAL ARTICLE

# EVALUATION OF LOOSELY BOUND WATER LOSS FROM DIFFERENT COMPOSITIONS OF GLASS IONOMER CEMENT

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**Background:** Water is an essential component of glass ionomer cement. Water balance is probably the most important and least understood mechanism with the glass ionomer cement. Excessive water in glass ionomer produce weak cement while less amount of water produce cement which is relatively stronger initially but later results in the weakening of the cement. Water present in glass ionomer cement is classified according to its nature of being held in to the cement as tightly bound and loosely bound. The amount of loosely bound water loss from various composition of glass ionomer cement remains unknown. **Methods:** The study was conducted at the Department of Materials, Queen Mary University of London. Two different composition of glass ionomer cements were used in this experiment in which the amount of water absorbed by the different compositions of cement on 1, 3, 7 and 14 days were evaluated and the loss of water was measured after that period until the loss became constant. A total of 25 samples of each GIC composition, 5 samples were immersed in water for 24 hours, 5 in water for 3 days, 5 for 7 days and 5 for 14 days. The remaining 5 samples were directly placed into the desiccator without immersing it in the water. The total water content of both glass ionomer cements was calculated from its chemical composition. The samples were weighed every hour for first 3 hours and then every 24 hours until the weight of the sample became constant. Samples placed in water for 1, 3, 7 and 14 days were dried before weighing with a tissue. **Results:** The amount of water uptake in all the compositions was not that significant in relation to time. In case of Fuji IX, amount of water loss percentage did not vary with increasing time interval. The water loss was rapid in the first 24 hours but it slowed down with time and became constant after 3 days however in Ketac molar water loss slightly varied with time interval. **Conclusion:** It is concluded that the amount of water uptake in both glass ionomer cement is not significant in relation to time. The loss of loosely bound water becomes constant with time after 24 hours for both compositions of glass ionomer cements.

**Keywords:** Loosely bound water; Glass ionomer cement.

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## INTRODUCTION

Glass ionomer cement is one of the commonly used restorative materials in dentistry. This material attains its name from its combination of a glass powder and an ionomer matrix that contain polyalkenoic acids.[1] Glass ionomer cements sets by an acid base reaction and form a core material surrounded by a matrix.[2]

Water is an essential component in its composition and varies in form as cement ages. Water balance is probably the most important and least understood mechanism with the glass ionomer cement.[3,4] Water act as a main reaction medium and plays a key role in the setting of the cement as it hydrates the reaction products. The strength of the cement is directly proportional to the amount of water present in it.[5] Excessive water in the glass ionomer produces a weak cement while less amount of water produces a cement which is relatively stronger initially but later results in the weakening of the cement.[6]

The water content of the cement is one of the integral parts of its structure. Water in glass ionomer cement not only participate as a reaction medium but also acts as a solvent, plasticizer and essential component in cement formation.[7] Water as the solvent is essential for the setting reaction of glass ionomer cements as water dissociates acid in to respective ions that allow dissolution to occur and permit the calcium and aluminium ions to enter the liquid phase and proceed the reaction by forming polyacrylate chain.[8] Loss of water from glass ionomer cement in the first hour disturbs the setting reaction and affects the strength of the cement and make cement prone to crack and shrinkage.[9] Water present in glass ionomer cement is classified according to its nature of being held in to the cement and divided in to two different states:

a) Loosely bound or evaporable and
b) Tightly bound or non-evaporable.[10]

Bound water is much more stable than unbound water. Around 24% of the set cement contains water and this water balance is disturbed if the cement is exposed too early to the air.[10] It is the reaction medium in which the calcium and aluminium are released and transported to form polyacrylate chains after reacting with polyacid. Water is essential for cement formation and its loss or absence from the reaction leads to a discontinuity of the reaction. Loosely bound water can be removed easily by the process of desiccation while tightly bound cannot be displaced from the glass ionomer cement easily.

Recently, several faster setting, high-viscosity conventional glass ionomer cements have become available called viscous or condensable glass ionomer cements by some authors, these materials set faster and are of higher viscosity because of finer glass particles, anhydrous polyacrylic acids of high molecular weight and a high powder-to-liquid mixing ratio. The setting reaction is the same as the acid-base reaction typical of conventional glass ionomer cements.[11] However, the loss of loosely bound water from these viscous glass ionomer cement remains unknown therefore we want to evaluate the loss of loosely bound water from two different compositions of GIC at different time interval.

Objectives of the study are to evaluate the amount of loosely bound water loss from two different compositions of glass ionomer cement.

## MATERIAL AND METHODS

The study was conducted at Department of Materials, Queen Mary University of London. Two different compositions of Glass ionomer cement were used for this experiment. These were:

1. Fuji IX FAST (GC Corporation, Tokyo, Japan)
2. Ketac Molar (3-M ESPE, Seefeld, Germany) (Table-1)

**Table-1: The difference in chemical composition of Fuji IX and Ketac Molar**

| Composition | Fuji IX | Ketac Molar |
|---|---|---|
| **Powder** | | |
| Si | 13.7% | 12.4% |
| Al | 17.9% | 15.0% |
| Ca | 0% | 10.1% |
| F | 10.2% | 13.3% |
| Na | 1.0% | 1.7% |
| P | 2.2% | 2.0% |
| Sr | 19.9% | 0% |
| La | 0% | 17.6% |
| **Liquid** | | |
| Water | 50% | 47.2% |
| Acrylic acid | 45% | 26.4% |
| Maleic acid | 5% | 26.4% |

Other Materials Used in The Experiment are Desiccant: SILICA GEL (Fluka, 1164963), Silicon Rubber Impression Material (Kerr Corporation, USA)

A Total of 50 samples were included in the study. 25 samples were made from each composition of GIC. Sample divisions were:

- 5 samples in Desiccator without water immersion
- 5 samples in water for 24 hours then placed in the desiccator.
- 5 samples in water for 3 days and then placed in the desiccator.
- 5 samples in water for 7 days and then placed in the desiccator.
- 5 samples in water for 14 days and then placed in the desiccator.

This division applied for both Fuji IX and Ketac Molar Glass ionomer cement.

Fuji IX and Ketac Molar were mixed in the powder/liquid ratio of 3.63:1 and 3.4:1 respectively. The cements were provided in encapsulated forms. The capsules were activated in metal GC capsule applier. Immediately after activation, the capsules were triturated in an electric triturator for 10sec. The mixed materials were then placed in a brass ring and then clamped. The peripheries of the samples were sealed with silicone rubber to prevent any water loss. All samples were then placed in oven at 37 °C for one hour.

The purpose of the experiment was to weigh the amount of water absorbed by the different compositions of cement at 1, 3, 7 and 14 days and then measure the loss of water after that period until the loss of water becomes constant. So, from 25 samples of every composition, 5 samples were immersed in water for 24 hours, 5 in water for 3 days, 5 for 7 days and 5 for 14 days. The remaining 5 samples were directly placed into the desiccator without immersing in the water. Before placing the samples in the desiccator, silica gel was placed at the bottom in an adequate quantity. Filter was placed into the desiccator and covered. Vaseline was applied to seal the desiccator properly to avoid any water exchange. The purpose of placing the cement discs (ISO specification 4049: 12-1998) in the desiccator (Bel Art™ SP Scienceware™ Space-Saver, Fisher Scientific) was to remove the evaporable water which was absorbed by silica gel.

The total water content of each dental cement can be calculated from its chemical composition.

The water uptake (U) was calculated by using following formula:

$U = W2 - W1$

Where W1 is the weight of cement disc before water immersion and W2 is the weight of water disc after water immersion. The total initial water content can be calculated by adding the water uptake (U) into the initial water content of the liquid (can be calculated from Powder/Liquid ratio). The samples were weighed every hour for first 3 hours and then every 24 hours until the weight of the sample became constant. Samples placed in water for 1, 3, 7 and 14 days were first dried with a tissue before weighing.

## RESULTS

Results of percentage of loss of loosely bound water (by weight) from KETAC Molar and Fiji XI samples in desiccator without water immersion and in water immersion after 24 hours, 3 days, 7 days, 14 days are given below: (Figure 1–10)

The results of the study showed the uptake of water with time for two different compositions of glass ionomer cements. For all the compositions, there wasn't any substantial change in the water uptake with time. (Figure 11-12)

Result also showed the percentage of loosely and tightly bound water present in different cements and amount of loss of loosely bound water with time. (Figure 13-14)

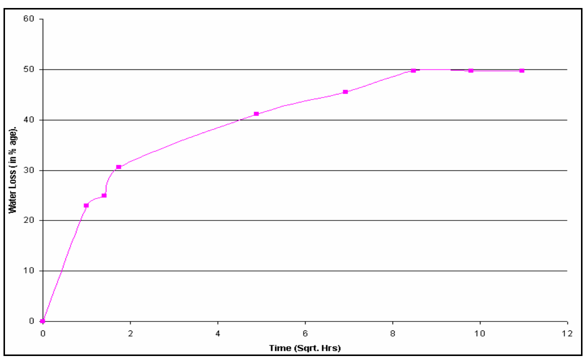


**Figure-1: Mean value for the loss of loosely bound water (in % age) from KETAC Molar samples in DESICCATOR without water immersion**

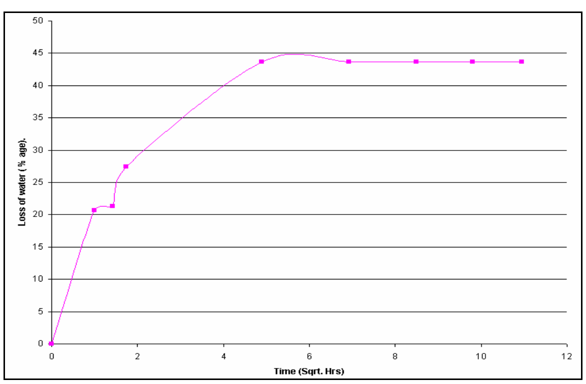


**Figure-2: Mean Value for loosely bound water (in % age) for FUJI IX samples placed in DESICCATOR without water immersion**

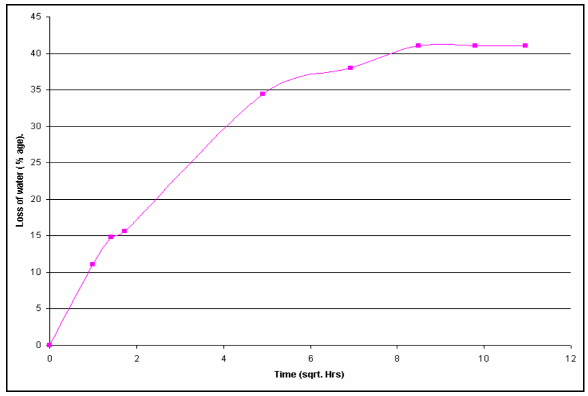


**Figure-3: Mean Value for loss of loosely bound water (in % age) from Ketac molar samples immersed in water for 24 hrs.**

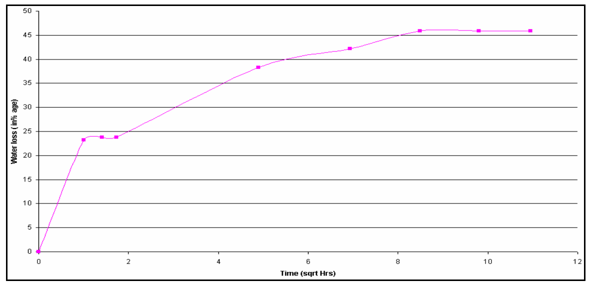


**Figure-4: Mean values of Fuji IX samples immersed in water for 24 hrs.**

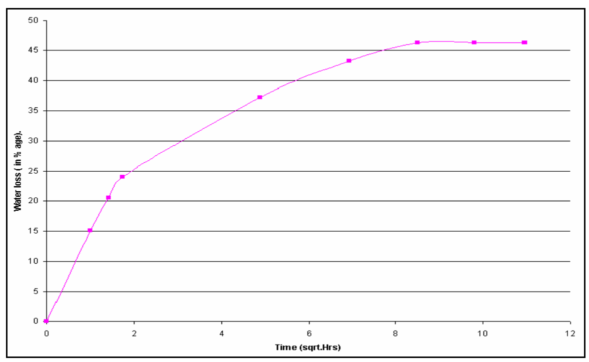


**Figure-5: Mean value for loss of loosely bound water in Ketac Molar samples immersed in water for 3 days.**

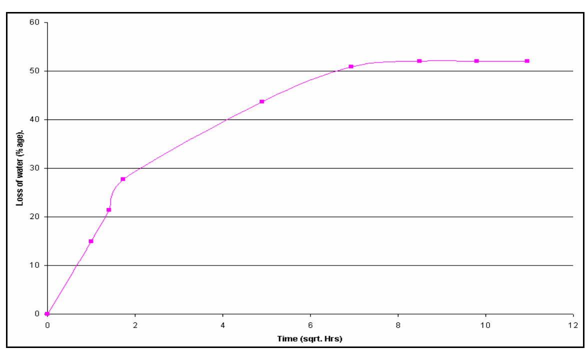


**Figure-6: Mean values of Fuji IX samples immersed in water for 3 days.**

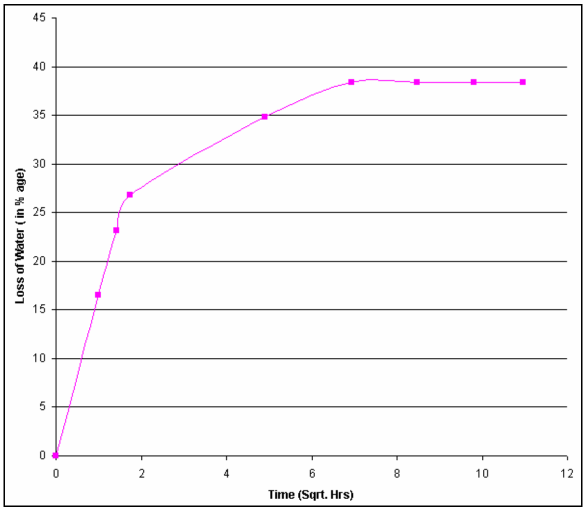


**Figure-7: Mean Value of loosely bound water loss (in % age) of Ketac Molar samples immersed in water for 7 days.**

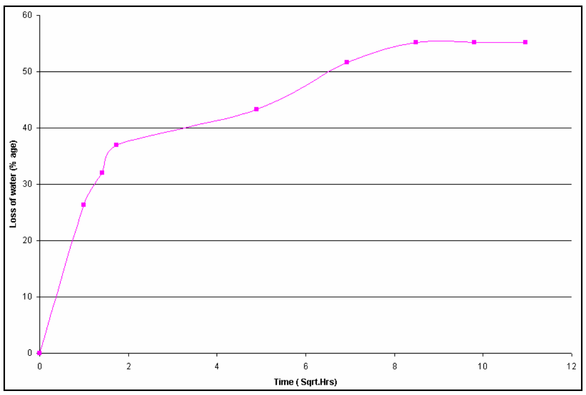


**Figure-8: Mean value (% age) loss of loosely bound water from Fuji IX immersed in water for 7 days.**

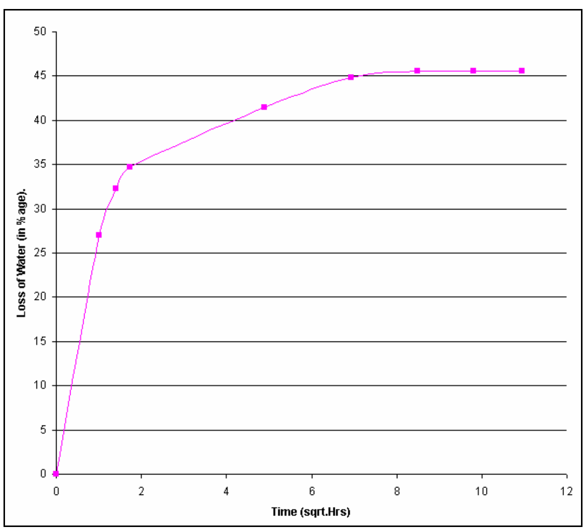


**Figure-9: Mean Value (in % age) of Ketac Molar samples immersed in water for 14 days.**

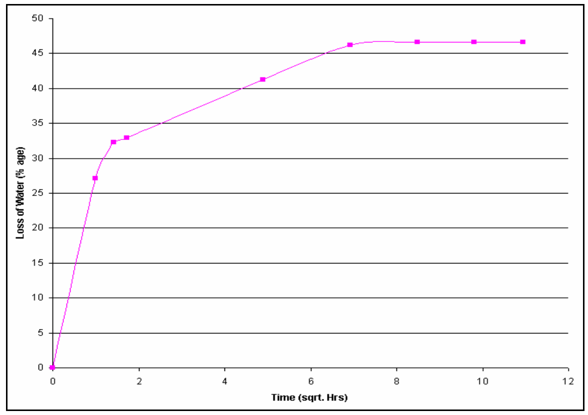


**Figure-10: Mean Values of Fuji IX samples immersed in water for 14 days in water.**

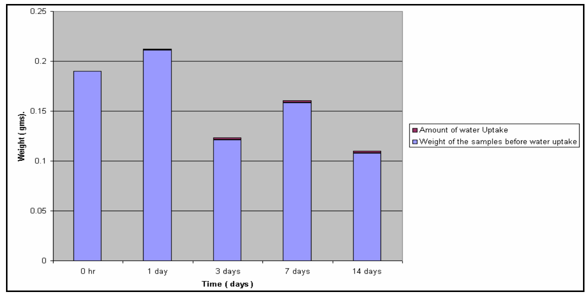


**Figure-11: Chart showing the amount of water uptake by Ketac Molar samples after Immersion in water for 0, 1, 3, 7 and 14 days.**

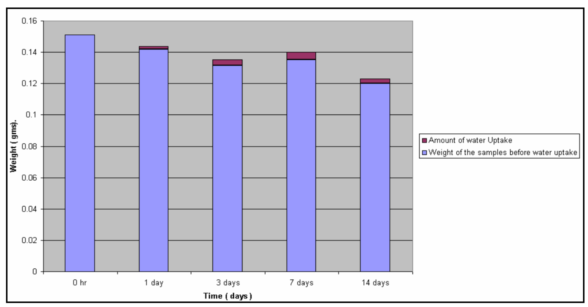


**Figure-12: Chart showing the amount of water uptake by Fuji IX samples after immersion in water for 0, 1, 3, 7 and 14 days.**

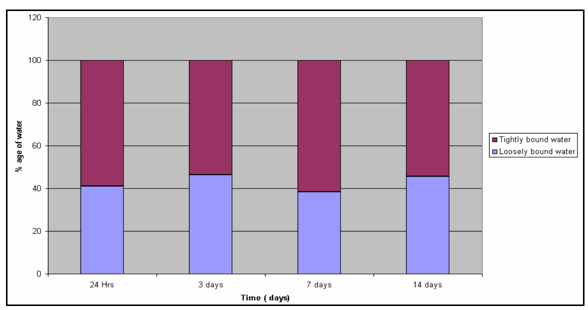


**Figure -13: Mean amount (% age) of loosely and tightly bound water present in Ketac Molar samples placed in water for 1, 3, 7 and 14 days.**

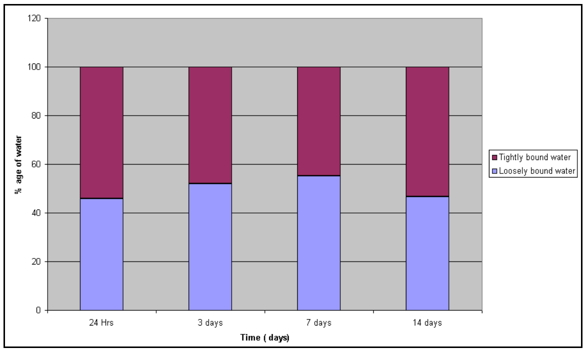


**Figure-14: Mean amount (% age) of loosely and tightly bound water present in Fuji IX samples placed in water for 1, 3, 7 and 14 days.**

## DISCUSSION

Different types of Glass ionomer cements used in this experiment were Fuji IX Fast (GC Corporation, Tokyo, Japan) and Ketac Molar (3-M ESPE, Seefeld, Germany). These are also supplied in encapsulated form and both are the most common commercially available material used in the clinics.

Fuji IX fast (GC Corporation, Tokyo, Japan) and Ketac Molar (3-M ESPE, Seefeld, Germany) have different composition. The liquid of Ketac Molar contains 47.2 % water and Powder/liquid ratio was 3.4:1. Besides water the liquid contains 52.8 % acids (Maleic acid and acrylic acid in proportion of 50:50). Liquid of Fuji IX contains 50 % of water and 50 % of acid (out of which 90% is acrylic acid and 10% is Maleic acid). Powder to liquid ratio was 3.63:1. The powder content of these two Glass ionomer cements also varies. Fuji IX powder contains strontium instead of calcium while Ketac molar contains Lanthanum & calcium in place of strontium. Other components are almost same. For both Glass ionomer cements, we have noticed water uptake, loss of water in percentage and ratio of loosely/tightly bound water present in it. In case of Fuji IX, amount of water loss percentage does not vary with increasing time interval. The water loss is rapid in the first 24 hours but it slows down with time and becomes constant after 3 days, which suggest initial rapid loss in first 24 hours may be due to setting reaction. It also suggests that as cement ages, lesser the water loss. Paddon *et al*[12] suggested that hydration becomes slow with aging. In this study, water uptake was almost constant with increasing time intervals and this indicates that cement do not take up water in high quantity regardless of time intervals. Around 50% of the water present in Fuji IX is tightly bound which is one of the reasons for its higher strength. Percentage of loosely and tightly bound water was unaffected with increasing time intervals.

For Ketac Molar the amount of water loss was almost the same as Fuji IX. The amount of water loss did not vary much with increasing time interval. The loss water was rapid in first 24 hours but the loss started to decline with time and eventually became constant after 3 days indicating the completion of setting reaction. Water uptake was also found to be constant with increasing time interval indicating that their water absorption capacity does not vary with increasing time intervals. The percentage of loosely and tightly bound water was also found to be unchanged with increasing time interval. Around 58% water present in Ketac Molar is tightly bound which imparts a higher strength to the cement.

The findings of our study are comparable with the findings of Paddon *et al* who reported that the cements become highly hydrated and stronger as they aged. However, in our study there was no substantial change in cement hydration with increasing time interval.

Water contamination should be avoided during the initial stage of set as most of the fluoride and aluminium ions at this stage are unreacted and can go into solution. So, if there is any water contact at this time matrix formation would be hindered and disintegration of the cement occurs.[13] Crisp *et al*[10] and Nicholson JW[14] have suggested that the formation of calcium polyacrylate is the reason of initial hardening, most of the aluminium ions are unreacted in the early stages, as being in complex form. However in later stage of cement maturation formation of aluminium polyacrylates occurs.[14]

Water plays an important role for proper maturation of a GIC, both water contamination and dehydration during the initial setting stage can compromise the physical properties of GIC therefore it is advisable to exclude water during the initial stage of setting which last for at least one hour even until 2 weeks.[13,15] Various separating medium like wax, vaseline, cocoa butter, waterproof varnishes, and even nail varnishes have been recommended in the past as suitable surface coating agents.[16] However; in this study we preferred vaseline over varnish because varnish over GIC can severely impede fluoride release as compare to vaseline over GIC.[17]

Difference in chemical composition of glass ionomer cement reflect the difference in their physicochemical properties like water sorption and solubility. Farias et al. found Ketac Molar being less sensitive to water sorption in comparison to conventional GIC, which can be explained by the large number of carboxylic acid groups in the liquid of such cements.[18] Carboxyl groups also prevent hardening of cement because of strongly bonded hydrogen to carboxylic acid groups and increase density of cross-links.[2] The hardening and precipitation reaction continues for at least 24 hours. However, the setting process continues at slow pace for a substantial period of time with minimum expansion and translucent appearance. Strength also tends to increase with time, Crisp *et al*[10] has suggested that it may be directly proportional to time.[10] Water uptake and loss is reduced as the cement ages as water becoming bound into the cement and thus less readily lost.

In future, further work should be done to analyse the effect of hydration at humid conditions similar to oral cavity and in artificial saliva instead of water. Analysis and comparison of hydration of glass ionomer cements with their mechanical properties should also be done to provide an understanding of their association.

## CONCLUSION

It is concluded that the amount of water uptake in both glass ionomer cement is not significant in relation to time. The loss of loosely bound water becomes constant with time after 24 hours for both compositions of glass ionomer cements.

## AUTHORS' CONTRIBUTION

MA: Topic selection, conceptualization of the study design, performed procedures. SM: Data collection, data analysis. MAA, ZC, NM, FN: Drafting of manuscript and abstract.

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