

## **Flexural strength and fracture surface characterization of glass-ionomer cements stored in water**

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### **Summary**

**In order to evaluate the effect of water on the microstructural and mechanical properties of glass-ionomer cements, the flexure strengths of six commercial ionomer cement materials stored in water at 37°C for 1 h and 1 week were determined and their fracture surfaces were characterized using electron microscopy techniques. The flexural strength value of the conventional glass-ionomer material stored in water for 1 h was found to be less than those of the resin-modified glass-ionomers and was higher than those of the glass-ionomer for molar filling materials. It was found that the coated glass-ionomer materials display higher flexural strengths than in the uncoated condition. SEM and energy dispersive analysis (EDS) investigations conducted on the fracture surfaces of the flexural specimens revealed crack formations and cavities and porosities resulting from the loss of ionomer particles. The 1 h specimens displayed cracks deeper and more pronounced than those occurred in the 1-week specimens.**

**Keywords: glass-ionomer cements, flexural strength, fracture surface characterization.**

### **Introduction**

Glass-ionomer cements have been used in a variety of clinical applications at an ever-expanding rate since their introduction to dentistry [1]. These materials have certain properties that make them very useful in restorative dentistry. They have the ability to bond physicochemically to both enamel and dentin [2-6], are biocompatible with the dental tissue [6-8], they release fluoride ions which may contribute to the inhibition of secondary decays in the tooth structure [9-11], and they possess a low thermal expansion coefficient, similar to that of tooth structure [12, 13].

Despite these beneficial characteristics, conventional glass-ionomers have poor physical and mechanical properties such as low fracture toughness and flexural strength [14-17], rough surface texture and opacity [18] and susceptibility to moisture contamination and dehydration during initial setting [18, 19]. In the last two decades, research and development investigations have been under way to overcome the disadvantages associated with the traditional glass-

ionomers. Consequently, glass-ionomer materials comprising polymerizable free-radically active metacrylate monomers and/or prepolymers have been developed [20, 21]. These photopolymerizable or resin-modified glass-ionomers offer the advantages of shortened setting time, reduced early moisture sensitivity, increased working time and better mechanical properties than those of the conventional glass-ionomer cements [22-25]. In addition to resin modification, metal powders have been added to conventional glass-ionomers, to improve their physical properties [12, 26]. On the other hand, there exists no systematic investigation regarding the effect of water storage on the microstructures of glass-ionomer materials reported in the literature. The present study aims to fulfill this need.

The objective of the present study was to determine the mechanical properties of glass-ionomer cement materials subjected to different storage periods in water and to investigate the effect of water storage on the microstructure of coated and uncoated specimens. For this pur-

pose, flexure strength values of six commercial ionomer cements stored in water for 1 h and 1 week were determined and the fracture surfaces of the bend specimens were characterized by using scanning electron microscopy (SEM) techniques.

## Materials and Methods

Six commercial and three distinct groups of glass-ionomer cements were used in this investigation. In the first group are the light-cured, resin-modified glass-ionomers Vitremer (3M Dental Products, St. Paul, Minnesota), Ionosit Fil P/L (3M Dental Products, St. Paul, Minnesota, USA) and Photac Fil (ESPE Dental-Medizin GmbH & Co KB Germany). The second group is the ChemFil Superior (Dentsply Caulk, Dentsply Int. Inc., P. O. Box 359 Milford, Delaware 19963-0359, USA) which is a conventional glass-ionomer material. The third group consists of Ionofil Molar (VOCO Postbox 767 27457 Cuxhaven, Germany) and Argion Molar (DMG Chemisch-Pharmazeu. Fabrik GmbH, Germany) which are glass-ionomer cements for molar filling.

Twenty specimens for flexural (three point bending) tests from each glass-ionomer cement sample were placed in an open-ended mould cavity made up of stainless steel, having the inner dimensions of 30 mm x 3 mm x 3 mm. All specimens were packed carefully into the cavity to avoid air bubbles and were covered with a transparent film. A stainless steel lid was squeezed directly on the paste and the excess paste was discarded. After that, the lid was removed and the specimen paste in the mold cavity was prepared in accordance with the instructions given by the supplier for each sample. Following this, all test pieces were divided into 2 groups and stored in distilled water at 37 °C at varying times, viz. the specimens of the first group were stored in water for 1 h whereas those of the second group for 1 week. To investigate the effect of water penetration on the strength of the glass-ionomer materials, uncoated bending specimens of the Vitremer, Chemfil Superior, Ionofil Molar and Argion Molar materials were also prepared.

Flexural (three point bending) tests were carried out on a Model 1195 Instron Universal

Tester. For flexural tests, 10 test specimens belonging to each group were taken out from water; their surfaces were dried with cotton swab and were taken immediately to the universal tester. Bending specimens were mounted on specially designed jigs having a span distance of 25 mm on two supports attached to the universal tester. Increasing loads were applied at a crosshead speed (extension rate) of 0.5 mm/min and the peak load at which fracture occurred was taken as the maximum force exerted on the specimen and the flexural strengths were calculated accordingly [27]. The results were reported as the mean of all measurements.

The results of flexural strength measurements were analyzed by calculating the mean and standard deviation for each sample. The variance in the mean and the standard deviation values for each sample was found to be homogeneous. The data for the conditions of storing 1 h and 1 week for the six samples of the present study were subjected separately to a one-way ANOVA and significance levels of  $p = 0.000 < 0.05$  for differences between the mean values were for both conditions. In addition, the flexure strength values of coated and uncoated samples of Vitremer, Chemfil-Superior, Ionofil Molar and Argion Molar materials stored in water for 1 h and 1 week were analyzed and the values were found to be homogeneous. The one-way ANOVA testing of these values displayed significance levels of  $0.000 < p < 0.05$ .

Morphological characterization of the fracture surfaces of bending specimens were performed by using a JEOL JSM-T330 scanning electron microscope (SEM) operated at 20 kV. SEM investigations were conducted on the varnished (coated) specimens of the Vitremer, ChemFil Superior, Ionofil Molar and Argion Molar ionomers. As per the Ionosit-Fil and the Photac-Fil materials, only the uncoated specimens were used during SEM investigations. Fractured half of one bending specimen from each sample was glued on a specimen holder and the fracture surface was coated with a thin layer of gold for conductivity.

## Results

*Table 1* is a list of the flexural strength values (three-point bending or transverse strength)

of the glass-ionomer specimens stored in distilled water for 1 h and 1 week, respectively. As seen in *Table 1*, the flexural strength values of the resin-modified cements (Vitremer, Ionosit-Fil and Photac-Fil) stored in water for 1 h are almost double the value for the ChemFil, a conventional glass-ionomer material. On the other hand, the flexural strength values of the Ionofil-Molar and Argion-Molar materials stored in water for 1 h are lower than that of the ChemFil material. After storing in water for 1 week, the flexural strength values of the resin-modified glass-ionomers compared to the specimens stored in water for 1 h showed a reduction more than 50% for the Ionosit-Fil and Photac-Fil and about 30% reduction for the Vitremer. However, in the case of the ChemFil, the reduction in the flexural strength value was about 20%. Thus, after storing in water for 1 week, the flexural strength value of a conventional glass-ionomer (ChemFil) material is very close to those measured for two resin-modified glass-ionomer cements (Ionosit Fil and Photac Fil). The effect of coating can be observed in the flexure strength values of the Vitremer and the ChemFil materials. Whereas the flexural strength values of the coated ChemFil are larger than those of its uncoated specimen for both 1 h and 1 week of water storage, these values are substantially larger for the coated Vitremer material compared with its uncoated specimen. Contrary to resin-modified and conventional glass-ionomer materials, the flexural strength values of the coated and uncoated Ionofil Molar and Argion Molar materials increased with storage time. Whereas the strength increase is about 60% for the coated Ionofil Molar that for the Argion Molar corresponded to an increase of 28% going from 1 h water storage to 1 week. The increases in strength values with storage time are even higher for the uncoated specimens corresponding to about 108 % for the Ionofil and 162 % for the Argion Molar material.

Scanning electron microscopy (SEM) techniques were used to progressively characterize the fracture surfaces of the coated bend specimens of the glass-ionomer cement materials.

*Figures 1a* and *1b* are low-magnification SEM fractographs taken from the Vitremer sample stored in water for 1 h and 1 week, respectively. A large spherical cavity about 100  $\mu$ m in size is shown in *Figure 1a* for the case of the 1 h

specimen. The counterfaces of the fracture surfaces of both specimens were examined and characterized by SEM to verify the nature of this cavity and several others seen in *Figures 1a* and *1b*. The SEM investigations of the fractured counterfaces revealed spherical particles in the fracture surfaces and the silicon (Si) and oxygen (O) were the predominant peaks in the energy dispersive spectroscopy (EDS) analyses taken from these particles. On the basis of these results, it can be stated that the cavities in *Figures 1a* and *1b* (both 1-h and 1-week specimens) are the remnants of differently sized glass-ionomer particles debonded from the matrix. Further, as seen in *Figure 1a*, the large glass-ionomer particles acted as a stress concentrator from which crack nucleation and propagation emerged. The fracture surface of the 1-week specimen, seen in *Figure 1b*, is very similar to that of the 1 h specimen. Here, cavity formations due to loss of glass-ionomer particles and crack propagation from a large ionomer particle can be also observed. The governing mechanism for cavity formations is not known and its interpretation is beyond the scope of this work.

*Figures 2a* and *2b* are SEM micrographs taken from the fracture surfaces of the Ionosit Fil P/L material stored in water for 1 h and 1 week, respectively. Similar to the Vitremer sample (*Figures 1a* and *1b*), cavities or porosities formed in the fracture surfaces of both specimens. However, the fracture surfaces of both specimens do not look alike, indicating that water had an effect on the fracture mode. Whereas the fracture surface of the 1 h specimen contain smooth and shiny regions, that of the 1-week specimen is rough and contain dimpled regions. On the basis of *Figures 2a* and *2b*, it can be inferred that the fracture mode for the 1 h specimen is predominantly brittle, occurring in the matrix phase whereas that for the 1-week specimen is governed more by particle debonding from the matrix phase. No cracks or crack formations are seen in *Figures 2a* and *2b* and no crack formations were observed during the SEM investigations from other locations of the fracture surfaces of both specimens of the Ionosit Fil P/L material.

Fractographs taken from the Photac Fil sample are given in *Figures 3a* and *3b* for the specimens stored in water for 1 h and 1 week, respectively. Similar to the Vitremer and Ionosit

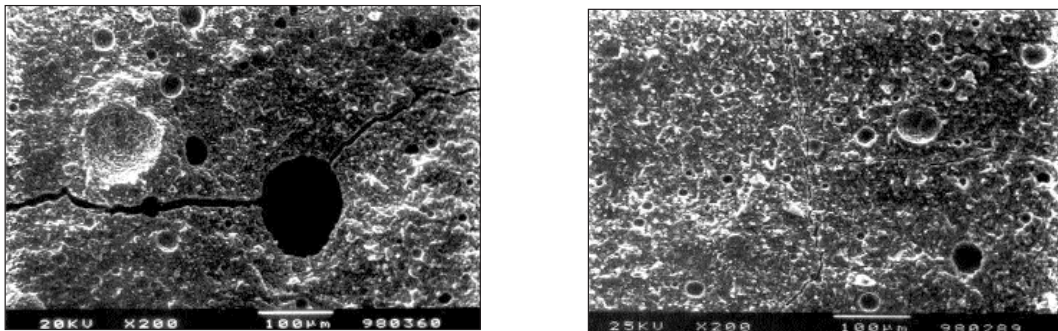
Fil samples, the fracture surface of the Photac Fil material stored in water for 1 h (*Figure 3a*) exhibits the presence and the loss of glass-ionomer particles in a mottled matrix. A deep crack believed to have originated from ionomer particles runs inward from one end of the specimen. *Figure 3b* is a low-magnification SEM fractograph of the Photac Fil material stored in water for 1 week. Similar to the 1 h specimen, a mottled fracture surface can be observed indicating granular particle/matrix separation and displaying barely visible cracks. On the basis of *Figures 3a* and *3b*, it can be mentioned that the separation of glass-ionomer agglomerates take place during fracture and these agglomerates act as nodal/initiator locations where the cracks emerge from.

*Figures 4a* and *4b* are the fracture surfaces of the ChemFil Superior material stored in water for 1 h and 1 week, respectively. As seen in *Figure 4a* for the case of the 1 h sample, round cavities and porosities and surface cracks are present in a mottled microstructure. Some microfissures exist on surface after 1 week of

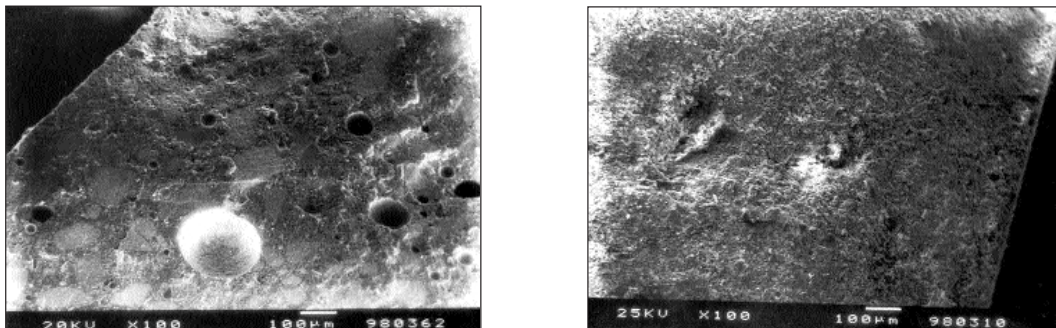
water storage sample with no apparent surface cracks (*Figure 4b*).

*Figures 5a* and *5b* are the SEM fractographs taken from the Ionofil Molar material stored in water for 1 h and 1 week, respectively. It is very obvious that the fracture surfaces of both specimens showed cracks and porosities. The cracks are deeper and more pronounced for both specimens. SEM investigations from other locations of both samples as well as their fracture counterparts revealed that the cracks ran along the particles and all over the matrix and occasionally passed across a particle.

*Figures 6a* and *6b* are the respective SEM fractographs taken from the Argion Molar material stored in water for 1 h and 1 week. The fracture surface of the 1 h specimen contain spherical cavities which are 15-30 mm in size and a deep crack crossing a particle and branching out to several directions. The fracture surface of the 1-week specimen also contains spherical cavities and a thin crack line with some barely visible cracks.



**Figure 1.** Representative scanning electron micrographs (SEM) taken from the fracture surface of the bending specimens of Vitremer stored in water at 37 0C for (a) 1 h, and (b) 1 week



**Figure 2.** Representative scanning electron micrographs (SEM) taken from the fracture surface of the bending specimens of Ionosit Fil P/L stored in water at 37 0C for (a) 1 h, and (b) 1 week



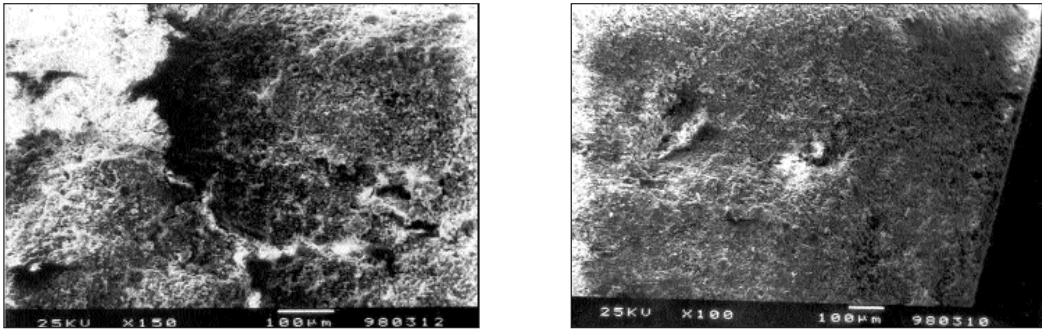


Figure 3. Representative scanning electron micrographs (SEM) taken from the fracture surface of the bending specimens of Photac Fil stored in water at 37 °C for (a) 1 h, and (b) 1 week

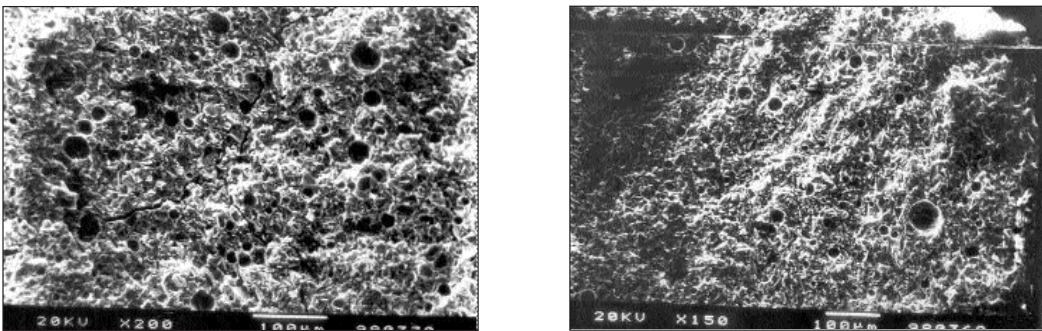


Figure 4. Representative scanning electron micrographs (SEM) taken from the fracture surface of the bending specimens of ChemFil Superior stored in water at 37 °C for (a) 1 h, and (b) 1 week

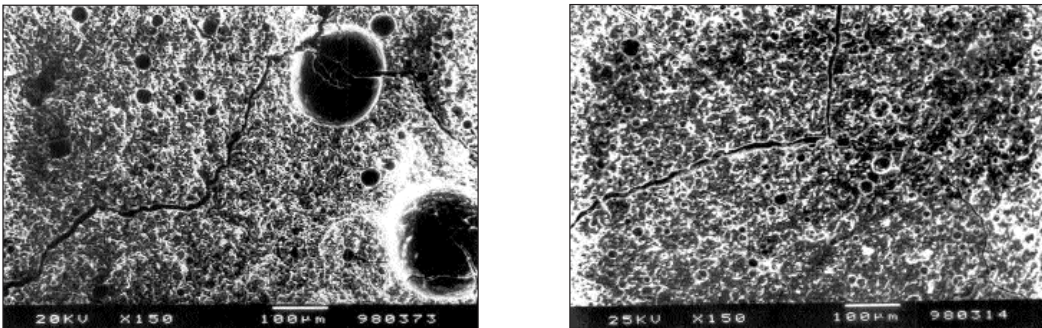


Figure 5. Representative scanning electron micrographs (SEM) taken from the fracture surface of the bending specimens of Ionofil Molar stored in water at 37 °C for (a) 1 h, and (b) 1 week

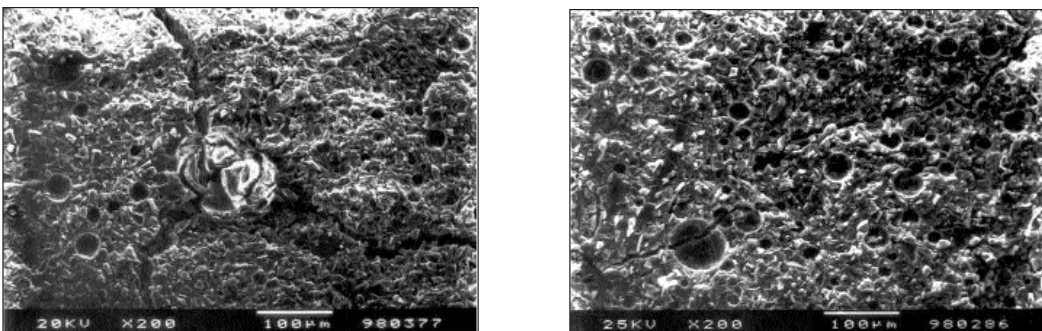


Figure 6. Representative scanning electron micrographs (SEM) taken from the fracture surface of the bending specimens of Argion Molar stored in water at 37 °C for (a) 1 h, and (b) 1 week

**Table 1.** Flexural strengths of glass-ionomer cements stored in water for 1 h and 1 week

Trade name	Coating	Flexural strength after storing	Flexural strength after storing
		1h in water (MPa)	1 week in water (MPa)
Vitremmer	Yes	68.11 ± 2.21	47.67 ± 1.55
	No	53.81 ± 3.76	31.67 ± 1.40
Ionosit Fil P/L	–	71.80 ± 4.50	29.97 ± 1.08
Photac Fil	–	70.85 ± 2.15	31.50 ± 0.76
Chemfil Superior	Yes	34.05 ± 1.28	27.25 ± 2.41
	No	30.65 ± 0.92	23.16 ± 3.96
Ionofil Molar	Yes	17.03 ± 0.97	27.24 ± 3.08
	No	12.26 ± 5.39	25.54 ± 3.65
Argion Molar	Yes	23.83 ± 2.45	30.64 ± 1.22
	No	8.17 ± 3.61	21.45 ± 3.19

## Discussion

The glass-ionomer cements are water-based materials with water playing an essential role in the setting reaction. Water acts as a reaction medium for the metallic cations to react with the polyacid, to form the polyacrylate matrix [28]. The metallic cations brought by water and hydrogen released by the water may also contribute to the strengthening of the cement by cross-linking the polyalkenoic acid chains [29, 30].

On the basis of investigations by several researchers, it can be stated that the reported mechanical properties of the resin-modified glass-ionomers are superior to those of the conventional glass-ionomers [28]. In the present investigation, after 1 h of water storage, the conventional glass-ionomer (ChemFil Sup) displayed a flexure strength value of 34 MPa close to the value reported earlier [31]. On the other hand, as expected, the resin-modified cements (Vitremmer, Photac-Fil and Ionosit-Fil) have flexural strength values around 70 MPa. As pointed out earlier [28], these results indicate the fact that the resin-modified cements are less brittle and are less prone to bulk fracture than the conventional glass-ionomer materials. It has been shown that water sorption performance of resin-modified glass-ionomers increases with storage time [32]. Further, it has been reported that the physical properties of the light-activated cements change dramatically when exposed to moisture. During storing in water, water penetra-

tion at the cement surface and resultant hydrolytic degradation can cause the ionomer cement to become progressively weaker with time [25, 33-36], causing compressive and flexural strength reductions [30, 37, 38]. In the present study, the flexure strength of all resin-modified and the conventional glass-ionomers decreased over a storage time of 1 week, stabilizing around a value of 30 MPa. In agreement with the above-mentioned researchers, we believe that water penetration with storage time plays a dominant role in the reduction of transverse strength values of the conventional and resin-modified glass-ionomer materials of the present study. On the other hand, some researchers have reported that the flexural strength of both resin-modified and conventional glass-ionomers did not change over the storage time [39].

As seen in *table 1*, the flexural strength values of the Ionofil Molar and the Argion Molar materials of the present investigation substantially increased with storage time. This observation is in agreement with the findings of other investigators [26, 31] who reported increases in the flexural values of alloy-reinforced glass-ionomer cements after 1 week of water storage.

Although the effect of water on the flexural strengths of glass-ionomer cements has been reported extensively, there exists no literature pertaining to a systematic electron microscopy (SEM) study of fracture surfaces of glass-ionomer materials. Thus, the most original aspect and contribution of the present study is a series of SEM investigations carried out on the fracture

surfaces of the glass-ionomer cement materials subjected to flexural testing. In general, based on results of these investigations, basic features like cavities or porosities resulting from the loss of ionomer particles and crack formation can be observed in the fracture surfaces of most glass-ionomer samples. Except for the Ionosit Fil P/L material, the fracture surfaces of the 1 h specimens of all samples displayed cracks deeper and more pronounced than those occurred in the 1-week specimens; cracks were thin and barely visible in the latter case. Based on this microstructural observation, it can be stated that the 1-week specimens of a given material display less regions of brittle (catastrophic) fracture than its 1 h samples indicating an apparent toughness increase in the polyacrylic acid based matrix with storage time. The fact that no direct correlation existing between the strength and the toughness [40] should be mentioned here for the sake of the argument. The toughness of a material depends on a combination of strength and ductility [41]. In other words, a material with high toughness value must have both high strength and ductility and that having high strength and a brittle character (low ductility) has low toughness [41, 42]. In the present study, contrary to increases in toughness of the glass-ionomer samples with storage time, while the flexural strengths of the conventional and resin-modified glass-ionomers decrease, those for the molar filling glass-ionomer materials increase.

## Conclusions

In this study, the flexural strength values of six glass-ionomer cements stored in water were determined and their fracture surfaces were characterized. On the basis of the results reported in the present investigation, the following conclusions can be drawn:

1. Whereas the flexural strength value of the conventional glass-ionomer material (ChemFil) stored in water for 1 h is less than those of the resin-modified cements (Vitremer, Ionosit-Fil and Photac-Fil), it is higher than those of the glass-ionomer for molar filling (Ionofil-Molar and Argion-Molar) materials. After storing in water for 1 week, the flexural strength values of the resin-modified and conventional glass-ionomer decreased and those of

the molar filling materials increased. There were no significant differences in the flexural strength values of all glass-ionomer materials after 1 week of water storage except the Vitremer material.

2. In general, the uncoated specimen of a glass-ionomer material displays a lower flexural strength value than its coated specimen. The differences in the flexural strength values of the coated and uncoated Ionofil Molar and Argion Molar materials are higher than those for the resin-modified and the conventional glass-ionomers.

3. Cavities and porosities resulting from the loss of ionomer particles and crack formations are the basic microstructural features in the fracture surfaces of most glass-ionomer materials. Except the Ionosit Fil P/L material, the fracture surfaces of the 1 h specimens of all samples displayed cracks deeper and more pronounced than those occurred in the 1-week specimens.

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