Abstract

Purpose: To evaluate the shear bond strength of a new generation of glass ionomer (Glass Carbomer) to enamel versus a nano-filled resin-modified glass ionomer (Ketac Nano) and a conventional type after different storage periods.

Materials & methods: Crowns of 36 sound and freshly extracted human permanent molars were sectioned mesiodistally into two halves. The convex buccal or lingual surface was gently ground with water cooled 200-, 400-, and 600-grit silicon carbide abrasive papers successively to obtain flat enamel surfaces. The prepared specimens (n = 72) were divided into three main groups (24 each): I (Ionofil Molar), II (Ketac Nano) III (Glass Carbomer). The specimens in each group were subdivided into three subgroups A, B & C according to the storage period in artificial saliva. Shear bond strength between enamel surface and the bonded material was measured using a universal testing machine at a cross head speed of 0.5 mm/min. All the debonded interfaces were examined under both binocular stereo microscope at 40× and SEM at 200× to determine the mode of failure.

Results: Ketac Nano recorded the highest shear bond strength values (9.30 ± 0.67, 12.07 ± 0.76, 6.7 ± 0.73) followed by Ionofil Molar, recording (5.25 ± 0.62, 7.82 ± 1.42, 5.91 ± 0.87) while the lowest values were found in Glass Carbomer specimens, recording (2.17 ± 0.63, 6.66 ± 0.68, 5.72 ± 0.79). There was a highly significant difference in shear bond strength values among the three different storage periods in all the tested materials (P < 0.0001). A positive correlation was recorded (R = 9.3) between the adhesive mode of failure and shear bond strength while a negative correlation was recorded (R = 4.5) between the cohesive mode of failure and shear bond strength using Spearman's correlation test.

Conclusions: Storage time was a factor which significantly influenced both shear bond strength and mode of failure especially in Glass Carbomer specimens.

Keywords: Shear bond strength; Enamel; Storage; Artificial saliva; Ketac N100; Glass carbomer
1. Introduction

Restorative dentistry has seen a paradigm shift from the invasive surgical approach laid by G.V. Black “extension for prevention” to a minimally invasive approach with advancement in diagnostic system and revolution in adhesion technology [1].

An ideal material used for restoration should be adhesive, tooth colored, resistant to wear [2], non-toxic, biocompatible to the tissue [3].

A new era had begun in the world of dentistry since the introduction of glass ionomer cement (GIC) in 1972 by Wilson and Kent, which have been considered to be a leading restorative material so far as they adhere to tooth structure [4], have an antibacterial activity [5], negligible dimensional changes [6], release fluoride [7], and can be used in different clinical situations such as luting of indirect restorations, lining, basing and filling [8].

Unfortunately, the use of GI as a restorative material was limited to areas of low masticatory forces [9] due to their low mechanical properties which were also affected by the powder/liquid mixing ratio of this material [10]. Another drawback is the slow rate of setting reaction which dictates postponing the finishing and polishing procedure to an additional visit [4].

Thus, several modifications and improvements were introduced [11].

In the late 1980's, an attempt to enhance conventional GIs was carried out through developing a hybrid material that combines composite resin and GI [12]. Resin-modified glass-ionomers (RMGICs) are light curable materials that allow for command set, less sensitive to dehydration than conventional GICs, immediate finishing and polishing following light curing [13], extended working time [4] and demonstrate higher flexural and diametral tensile strengths [14].

The shift towards the use of nano-fillers in esthetic restorative materials supported the fact that the small size allows finer polishing and smoother surface. At the same time, the grain size is too small for dislocation and higher strength can be obtained by incorporation of such nano-fillers [15]. This trend was utilized to solve the problems of esthetics and low wear resistance of RMGICs, so another type of glass ionomers based on nano-fillers was introduced, Glass Carbomer which sets chemically [16].

The use of nanotechnology greatly increases the reactive surface of the filling material, which in turn leads to a better reaction. An organic carbon chain additive, which is completely biocompatible, is also added to Glass Carbomer to provide the material with greater strength and increased transparency [17].

Although clinical trials would provide the ultimate evidence of clinical performance of dental restorations, preliminary and safety studies on dental materials should be conducted in vitro [18].

Different methods can be utilized in vitro to evaluate the durability of the bond strength to tooth structure. The shear bond strength test is one of these methods which has been widely used as it was reported to be easily performed [19].

2. Materials & methods

Thirty-six sounds & periodontally involved extracted human permanent molars were selected from patients, after signing a written consent, aging between (35–45) years for this study. Teeth were cleaned from tissue remnants and debris using periodontal curettes then polished with slurry of pumice and water. Teeth were examined to ensure they were free of any visible hypoplastic defects, cracks or white spots (demineralized enamel) using the blue light of a light curing unit [20].

They were stored in refrigerated saline solution for maximum 3 months as recommended by the ISO norms (ISO. Guidance on testing of adhesion to tooth structure.International Organization for Standardization, 1994) [21].

Crowns of the collected teeth were separated from the roots at cemento-enamel junction then, sectioned mesiodistally into two halves by cutting parallel to the long axis and facial surface using a low speed diamond disk under continuous water cooling [22].

Custom made cylindrical metallic molds of 20 mm length and 14 mm diameter were filled with chemically polymerizing acrylic resin [2]. Each metallic mold composed of an external cylindrical part surrounded another split metallic halves of 18 mm height \(\times\) 12 mm diameter (Fig. 1).

These two metallic compartments were adjusted together by means of two external screws to facilitate the insertion and removal the acrylic block from the mold (Fig. 1).

Each crown half was embedded horizontally in the acrylic resin making the buccal or lingual enamel surface facing upwards (Fig. 2). Care was taken to keep the experimental surfaces free of contamination by acrylic resin.

1 Polofil® Lux, halogen light, Voco, Germany.
2 Imicryl, Konya, Turkey.
After setting of the acrylic resin, the specimens were removed from the molds and the convex buccal or lingual surface of the crown was gently and mechanically ground with water cooled 200-, 400-, and 600-grit silicon carbide abrasive papers successively to obtain flat enamel surface [23].

A specially designed custom made split Teflon mold (Fig. 3) 4 mm diameter/2 mm height [24] was secured to the polished, clean and dry flat enamel surface prior to application of the tested materials. This Teflon mold was stabilized in its place by the mean of another split metallic ring fitted inside the external cylindrical metallic mold (Fig. 4).

The prepared specimens \( n = 72 \) were randomly divided into three main groups (I, II and III) according to the material used (24 each):

- Group I: Ionofil Molar (control group), Group II: Ketac N100 and Group III: Glass Carbomer (composition & manufacture of each material are mentioned in Table 1).

The specimens of each group were prepared, applied and cured according to the manufacturer instructions. The recommended powder \( \times \) liquid ratio of the conventional ionofil molar was applied to dry and clean glass slap. The powder was divided into two portions then mixed with the liquid using a plastic or metallic spatula until reaching the proper condensable consistency (mixing time is 50–60 s). The mix was directly applied into the Teflon mold using a plastic instrument. The excess was removed and the material...
was immediately protected against humidity through covering it with the recommended varnish. The material was left to set (4–5 min) before separation of the specimen from the mold.

For the Ketac Nano specimens, primer was applied for 15 s on the prepared dry tooth surface. Using a fiber tip, then dried for 10 s using an air syringe followed by light curing for 10 s maintaining the shiny appearance. Equal volumes of each Ketac Nano paste were dispensed onto a mix pad. Using a metal cement spatula, the pastes were mixed together for 20 s until a uniform color was achieved. A delivery tip was immediately back loaded with Ketac Nano then dispensed directly into the Teflon mold using a capsule dispenser. One increment of 2 mm thickness was dispensed and light cured for 20 s using LED curing unit (blue phase C5).3

The capsule of glass carbomer was tapped on a hard surface to loosen the powder then activated by insertion into a universal capsule gun. The capsule contents were mixed using a high frequency mixer (amalgamator) for 10–20 s. The mixed capsule was placed in a universal applicator after removing the stop from the nozzle then the material was extruded directly from the capsule into the mold. A cotton pellet was used to cover the surface of the material with surface gloss then placed under finger pressure and light cured with the LED curing device (1000 mW/cm²) for (60–90) sec where light cure device was used to enhance setting reaction of glass carbomer not as photo initiator.

All specimens were load cycled and were thermal cycled for 600 cycles from 5 °C to 55 °C with 30 s dwell time, 20 s transfer time resembling 6 months of clinical condition [25].

The specimens of each group were randomly subdivided into three subgroups A, B and C (8 specimens each) according to the storage period (24 h, 3 months and 6 months respectively).

Specimens of each subgroup were stored in artificial saliva in an incubator at 37 °C. The storage medium (artificial saliva) was changed weekly to minimize the risk of bacterial growth and maintain the PH of saliva at 7.6 [26].

2.1. Shear bond strength test

At the end of each storage period, the specimens were secured to the universal testing machine⁴ by means of their metallic molds (Figs. 5 and 6).

The specimens were oriented so that the stainless steel knife of the universal testing machine was

---

⁴ Model Instron, comten industries inc., St. Petersburg Florida, USA.

3 Ivoclar Vivadent AG, Liechtenstein.
perpendicular to the interface between the tooth surface and the tested material (Fig. 6). The knife was used to apply load at a cross head speed of 0.5 mm/ min until debonding [27] (Fig. 7).

The fracture load was recorded and the shear bond strength values were calculated according to the following equation:

$$\text{Shear bond strength} = \frac{\text{Fractured load (Kg)}}{\text{Surface area (Cm}^2)}.$$  

The surface area ($A$) was calculated from the following equation:

$$A = \pi r^2$$

where $\pi = 3.14$ and $r = \text{Radius of each specimen} = 0.2 \text{ Cm}$.

So $A = 0.1256 \text{ Cm}^2$.

The shear bond strength values were converted into MPa according to the following equation [28]:

$$\text{MPa} = \frac{\text{Kg/ Cm}^2 (\text{the obtained results})}{0.098067}.$$  

The recorded data related to each group was collected, tabulated and subjected to statistical analysis using One Way ANOVA and HSD Tukey's tests. Spearman's correlation test was performed to detect the presence of a relationship between the shear bond strength and mode of failure.

2.2. Mode of failure

All the deboned surfaces of the specimens were examined by four different examiners (dentists) under a stereomicroscope at magnification 40× to record the mode of failure.

Failure was identified as “adhesive” if no observable glass ionomer remained on the enamel surface and it had a polished appearance. “Cohesive within ionomer” if a visible thin coating or bulk amounts of glass ionomer remained on the enamel surface or “mixed type” of failure if a mixture of both modes of failure was observed [29].

For verification of the true mode of failure, all specimens were inspected by Scanning Electron Microscope (SEM) at 200× fold magnification. Two examiners determined the SEM failure patterns together without knowing the patterns given at the preceding microscopic inspections. Therefore, the previous tabulated mode of failure was confirmed.

---

5 SZ-CTY Olympus, Japan.
6 JSM-5300 scanning microscope, JEOL, Peabody, MA, USA.
Data were then collected & statistically analyzed to find out the relation between the mode of failure of the different groups and subgroups.

3. Results

Using One Way ANOVA test; a statistical significant difference was recorded between the three groups concerning each tested storage period. After 24 h; there was a highly statistical significant difference ($p < 0.0001$) which was more obvious after storage for 3 months ($p < 0.0001$). However, this statistical significance was diminished after 6 months storage although it was present since $p$-value recorded (0.04). This was clarified in Table 2.

3.1. Shear bond strength results

Concerning each tested group or material, as shown in Table 2, Ketac Nano (group II) revealed the highest shear bond strength values along the different storage periods followed by group I (Ionofil Molar) while the lowest shear bond strength values were found in group III (Glass Carbomer) (Fig. 8).

The statistical analysis of Variance (using One way ANOVA test) considering the storage time as a factor revealed a highly significant difference in shear bond strength values among the three different storage periods in all the tested materials (group I, II and III) recording $P$ values of 0.0005, <0.0001 and <0.0001 respectively as shown in Table 2.

So, further statistical analysis using (Tukey’s HSD test) was performed to fetch out which period of storage was responsible for the observed statistical significance.

Tukey’s HSD test recorded that the shear bond strength in subgroup A was statistically different from both subgroups B & C which were not significantly different compared to each other assuming that the first interval (24 h) was the cause of the recorded significance.

3.2. Mode of failure results

Percentages of mode of failure in the different subgroups are shown in Table 3.

Representative stereo microscope and scanning electron microscopic pictures of each type of failure (cohesive, adhesive & mixed) are shown in Figs. 9–11.

Regardless the different subgroups, Chi-square test revealed the presence of a statistical significant difference related to both the adhesive and mixed modes of failure ($p = 0.005$ and $p = 0.033$ respectively) while the cohesive failure mode wasn’t significantly different ($p = 8.7$) among the three tested groups. This is obvious in Table 3.

Finally, Spearman’s correlation test (at a significance level $p \leq 0.05$) was performed to find out the presence or absence of a relationship between the shear

| Subgroup group       | Subgroup A (24 h) mean ± SD | Subgroup B (3 months) mean ± SD | Subgroup C (6 months) mean ± SD | F-test (P-value) |
|----------------------|------------------------------|---------------------------------|---------------------------------|------------------|
| Group I (Ionofil Molar) | 5.25 ± 0.62                  | 7.82 ± 1.42                     | 5.91 ± 0.87                     | 13.7 (0.0005)**  |
| Group II (Ketac Nano)  | 9.30 ± 0.67                  | 12.07 ± 0.76                    | 6.7 ± 0.73                      | 86.07 (<0.0001)**|
| Group III (Glass Carbomer) | 2.17 ± 0.63                  | 6.66 ± 0.68                     | 5.72 ± 0.79                     | 52.2 (<0.0001)** |
| F-test (P-value)        | 273.84                       | 66.27                           | 3.79                            |
| (*<0.0001)**            | (<0.0001)**                   | (0.04)*                         |

F = One Way ANOVA.
*Significant at ($P < 0.05$).
**Significant at ($P < 0.001$).
bond strength values recorded and the mode of failure obtained.

One of the main results from the analytic investigation was that a strong correlation had been found between the mean bond strength values and the observed failure mode.

A positive correlation was obtained between the adhesive mode of failure and shear bond strength values where $R$ value recorded (9.3). The increase in shear bond strength values was accompanied by an increase in the adhesive percentage of failure mode as found in group II.

Table 3
Mode of failure of the tested materials.

| Mode of failure | Subgroup | Adhesive | Cohesive | Mixed | Chi-square test $\chi^2$ (p) |
|-----------------|----------|----------|----------|-------|-----------------------------|
| (Group I)       | Subgroup A (24 h) | 0        | 50%      | 50%   | 56.4(0)*                   |
| Ionofil Molar   | Subgroup B (3 months) | 0        | 25%      | 75%   |                             |
|                 | Subgroup C (6 months) | 12.5%    | 12.5%    | 75%   |                             |
| (Group II)      | Subgroup A | 12.5%    | 25%      | 62.5% | 32.7(0.0000013)**          |
| Ketac Nano      | Subgroup B | 12.5%    | 12.5%    | 75%   |                             |
|                 | Subgroup C | 25%      | 0        | 75%   |                             |
| (Group III)     | Subgroup A | 0        | 66.7%    | 33.3% | 52.5(0)*                   |
| Glass Carbomer  | Subgroup B | 0        | 16.7%    | 83.3% |                             |
|                 | Subgroup C | 0        | 50%      | 50%   |                             |

$\chi^2 = $ Chi-square test.
*Significant at $p < 0.05$ level.
**Highly significant at $p < 0.001$ level.

Fig. 9. a & b: Stereo microscope and SEM images of enamel side of debonded specimen (Ionofil Molar) showing cohesive mode of failure (complete attachment of glass ionomer to enamel).

Fig. 10. a & b: Stereo microscope and SEM images of enamel side of debonded specimen (Ketac Nano) showing adhesive mode of failure (complete detachment of glass ionomer from enamel surface).
In addition, a negative correlation was obtained between the cohesive mode of failure and shear bond strength values where \( R = 4.5 \). A clear example is group II which recorded the least percentage of cohesive failure and the highest shear bond strength values.

Also, this was seen in group III recording the least shear bond strength values and the highest percentage of cohesive failure, however, no percentage of adhesive failure was recorded and this was true for all tested storage periods.

On the other hand, no correlation was found between the shear bond strength values and the mixed mode of failure regarding any tested group or storage period.

4. Discussion

This in vitro study was performed to evaluate the bond strength since it was reported that, bond strength testing can reveal valuable clinical information, when gathered in a well-controlled design. In vitro studies testing GI cements typically have large standard deviations in bond strength values which make inter-study evaluation and comparison difficult [30].

Shear bond strength is a simple and widely used test to assess the bonding performance of restorative materials [31], particularly regarding the GICs, which present low bond strength, other tests may offer great difficulty to be applicable [29].

Currently, the tested material was (Glass Carbomer) which is a modified glass ionomer cement used as a restorative filling material or fissure sealant. It has the ability to bond adequately and remineralize the tooth surface since it contains nanocrystals of calcium fluoro-apatite particles as claimed by the manufacturer. Therefore, bonding procedures to enamel was currently investigated.

By introducing an aging factor into the study design, one can assess the durability of adhesion. Different artificial aging techniques can be used, depending on the specific types of bond degradation that are being investigated [32].

The most commonly used artificial aging technique is long-term water storage. In a trial to mimic the clinical situation more closely, artificial saliva can be used but bond strength reductions obtained were similar to those obtained with pure water degradation [33]. Even enzymes can be added to the storage media. For example, esterases that can be produced by bacteria in vivo which are able to catalyze the breakdown of resin components [34]. In addition, load and thermal cycling was performed to mimic the clinical conditions [25].

Thus, in the current in vitro study, shear bond strength was recorded after three different storage periods (24 h, 3 months and 6 months) in order to evaluate the effect of time on bond strength of glass ionomer materials since the timeframe within which GIC shear bond strength is measured is an important consideration [35].

For all the tested groups, it was found that; shear bond strength increased after three months compared to the baseline data (24 h).

A suitable explanation of this result is that after 24 h of storage, maturation of the glass ionomer cement is incomplete causing the lowest values of bond strength. The improvement of the bond strength after three months storage was due to aging which allow sufficient time for complete cement maturation [36]. This was also supported by Choudhari et al. [37] who reported that adhesion between glass ionomer and tooth structure is based initially on hydrogen bonding and over time matures and evolves into a stronger chemical bond.

Confirming the previous explanations, it was found that, the recorded shear bond strength values after 6
months storage period in groups I & III still showed an increase compared to the base line data. However, bond strength values declined after prolonged storage period (6 months) regarding group II which might be explained by hydrolysis of the bond or dimensional shrinkage of the cement due to aging caused by modification of the area of stress concentration thus consequently weakened the bond in glass ionomer–tooth interface [36].

Another theory might present, variations in bond strength of glass ionomer cements under the storage time to be related to an equilibrium of factors that increase strength, such as hydration of metal-carboxylate links and maturation of polysalt matrix, and those that decrease them, such as polymer matrix hydrolysis, which occur at different times explaining the importance of water sorption in the aging process [38].

On the same time, the current results showed that, Ketac Nano (RMGIC) recorded the highest shear bond strength values compared to Ionofil Molar (control group) and Glass Carbomer which agreed with those of Arora et al. [39] who had stated that RMGIC demonstrated improved mechanical and physical properties than that of the conventional GIC.

The pre-conditioning step with the nano-primer was supposed to improve the shear bond strength of nano-filled resin modified glass ionomer (Ketac Nano) to the tooth structure. Furthermore, conventional glass ionomers bond to tooth substrate by ion-exchange while RMGIs bond to tooth substrate through both ion-exchange and micromechanical interlock (dual mechanism of adhesion), as presented before by several authors [40–44].

These results were also in agreement with Sur-yakumari Nujella et al. [45] who found that mean shear bond strengths of light-cured RMGIC materials (9.71 Mpa) are significantly higher than that of conventional glass ionomer materials (3.81 Mpa), which might be due to the presence of light-activated resin component hydroxyethyl methacrylate (HEMA) with its superior wetting ability. The improved adhesion values might be also due to the slowness of acid—base reaction, which makes the polyacid available for longer periods thus resulting in higher bond strengths.

The composition of the restorative materials can interfere with their bond strength [38]. Materials containing resin components in their composition have an improved performance, with evidence of mechanical interlocking. Some studies have demonstrated that the 2-hydroxylethylmetacrylate (HEMA) concentration in the GIC formulation may contribute to the different bonding abilities of such material [29].

On the contrary, Breschi et al. [46] reported that the presence of high concentrations of hydrophilic monomers in primers may produce incomplete polymerization and water permeability, causing elution and degradation of resin components, such as some self-etch adhesive systems. Due to both water attractions by HEMA and oxygen inhibition, hydrolytic degradation of Ketac Nano-tooth bond might be expected to occur [47]. On the other hand, alterations in viscosity are frequent and can interfere severely with the bond strength of these cements [48]. When P/L ratio is reduced, there is a decrease in the ion release process and consequently, the cross links present lower strengths which can be reduced more rapidly. However, at high P/L ratios, unreacted particles can act as stress concentration points and also reduce strength [49]. Therefore, this may explain that the currently conventional type of glass ionomer recorded lower shear bond strength values than those of Ketac Nano group.

An experimental technique which is not yet fully established by which the cements hardening is substantially accelerated with ultrasonic or heat treatment was reported to solve many problems related to the slow setting of glass ionomers and enhance their mechanical properties [4].

The manufacturer recommended the use of further heat treatment on the tested material (Glass Carbomer) which was not followed in the current research assuming that this might be a reasonable explanation of the low shear bond strength values recorded for group III.

In addition, the results of Glavina et al. [50] study reported that enamel shear bond strength of Glass Carbomer (13.7 MPa) was significantly higher than that of conventional GIC (6.7 MPa) after being heated with three different LED polymerization units (1000 mW/cm², 1200 mW/cm², 1600 mW/cm²) and no statistically significant differences in the shear bond strength of Glass Carbomer material regarding the use of different polymerization units were observed. Accordingly, in the current study, LED curing unit (1000 mW/cm²) was chosen for curing of the tested material (Glass Carbomer).

Confirming the bond strength results, examination of fracture mode of the tested specimens showed that mixed and cohesive modes of failure were predominant among the Ionofil Molar and Glass Carbomer tested groups while adhesive mode of failure had the least
incidence however in Ketac Nano group the adhesive failure was greater than the cohesive one.

Explaining the mixed mode of failure of the tested Ionomil Molar and Glass Carbomer specimens might be due to the low resistance of GIC to early wear and the formation of glass ionomer matrix. Therefore, part of the glass ionomer remained adhered to the tooth structures, while part was broken at the interface GIC-tooth. Another explanation might be due to the chemical bonding of GICs to the tooth structure [51].

This was confirmed by explaining that, the longer term studies have shown an ion exchange process at the interface between the GIC cements and the calcified structures consisting of a complex of carboxyl, calcium and phosphate ions, derived from the cement, the enamel and dentine. This ion exchange layer is a few micrometers thick, and is extremely strong and results in the formation of a distinct interfacial region with time [52].

Another point of view presented that, during bond strength testing, the glass-ionomer fails cohesively before the ion-exchange layer fails i.e. cohesive failure in the cement rather than adhesive failure at the interface with tooth structure, and this complicates the interpretation of bond strength testing in the laboratory [53].

The current results were in agreement with some previous studies [54–57] and disagreed with Wang et al. [29] who revealed predominant adhesive failure mode.

It was also reported that, the presence of cohesive and mixed failures for GIs means that bond represent only the tensile bond strength of the cement rather than the strength of the tooth—cement interface [12,43,58] which may indicate that the interfacial strength of the bond is actually higher than the inherent strength of the material [59]. Another explanation was that, the degradation process of glass ionomer with prolonged time was due to decreased material properties rather than to decreased bonding potential [32]. However the formation of this ion exchange layer has been claimed to be the reason for the long term durability of the adhesive bond formed by these cements [60].

This explanation was also confirmed by several authors [61–64] who considered that no relation exists between fracture type and adhesion strength which was not true currently except for the mixed type of failure mode.

The tested Glass Carbomer material showed only cohesive and mixed failure modes while no adhesive mode of failure had been detected. This might be due to the findings of Koenraads et al. [17] who stated that all glass ionomer restorations showed fracture lines within the material (cohesive failure).

In addition, it was recorded that, the higher the bond strength of group II, the higher the rate of adhesive failure compared to groups I and III. However, this was only in 12.5% of samples while the mixed mode of failure was nearly in 75% of samples. This low percentage can explain the strong disagreement shown in the researches done by Leloup et al. [65] and Braga et al. [66] who found a significant positive correlation between shear bond strength and the rate of cohesive failure. Currently, we emphasized that under higher magnifications, the incidence of mixed & cohesive failure modes might have been even higher. However, it still remains speculative how failure site descriptions should be valued.

In contrast, Furuse et al. [67] concluded different results, showing lower bond strength values which were significantly correlated with mainly adhesive fractures.

Also the current statistical correlation indicated that, the increase in cohesive failure of group III was associated with decrease in the shear bond strength compared to group I and/or II at 24 h or 6 months storage period which was recorded as 66.7% and 50% respectively. This agreed with Leloup et al. [65] and Kanehira et al. [68] but it was an interesting summarizing observation which indicated an increase in the bond strength related to the higher rate of cohesive failure however, it still leaves the question open, which factors might be responsible for the high bond strength and the associated cohesive failure mode. Currently, the storage time was one effective factor.

As shown in this part, commonly reported failure modes from deboned macro shear tested specimens, as assessed by low magnification microscopy, are investigator — dependent and thus not very useful to shed light on the efficacy of the tested variables on the bonding quality acquired. SEM inspection, which was used in the current research, is surely advisable but costly and time consuming. An alternative simple method to improve detection of characterization of the failure pattern by light microscopy was recorded by Kanehira et al. [68] who pretreated the fracture site with a droplet of acid followed by slight rinsing to enhance differentiation between the non-affected resin surface and the acid etched enamel or dentin area exposed.

5. Conclusions

In conclusion: under the limitations of this study,
i. The durability or the maturation of the three tested glass ionomer materials was acceptable.
ii. Maturation of bond strength was related to the mode of failure.
iii. In addition to the difficult practical manipulation and application, the shear bond strength of Glass Carbomer was low compared to the conventional glass ionomer in contrast to the claims of the manufacturer.
iv. In spite of recording low shear bond strength, Glass Carbomer tested material exhibited no adhesive mode of failure which is considered an advantage in the routine clinical practice of restorative materials.

References

[1] Khattab NM, Omar OM. Papain—based gel for chemomechanical caries removal: influence on microleakage and microshear bond strength of esthetic restorative materials. J Am Sci 2012;8:391—9.
[2] Matis BA, Cochran MJ, Carlson TJ, Guba C, Eckert G. A 3 years clinical evaluation of two dentin bonding agents. J Am Dent Assoc 2004;135:451—7.
[3] Bernabe PFE, Holland R, Morandi R, Souze V, Nery MJ, OtoboniFilho Ja, et al. Comparative study of MTA and other materials in retrofitting of pulpless dogs’ teeth. Braz Dent J 2005;16:149—55.
[4] Davidson CL. Advances in glass-ionomer cements. J Appl Oral Sci 2006;14:3—9.
[5] DaSilva RC, Zuanon AC, Spolidorio DM, Campos LA. Antibacterial activity of four glass ionomer cements used in atraumatic restorative treatment. J Mater Sci Mater Med 2007;18:1859—62.
[6] Yan Z, Sidhu SK, McCabe JF. The influence of microstructure on thermal response of glass ionomers. J Mater Sci Mater Med 2007;18:1163—7.
[7] Gjorgjevska E, Nicholson JW, Gjorgovski I, Iliovska S. Aluminum and fluoride release into artificial saliva from dental restorative placed in teeth. J Mater Sci Mater Med 2008;19(3):i63—i3167.
[8] Farrell CV, Johnson GH, Oswald MT, Tucker RD. Effect of cement selection and finishing technique on marginal opening of cast gold inlays. J Prosthett Dent 2008;99:287—92.
[9] Kleverlaan CJ, Van Duinen RN, Feilzer AJ. Mechanical properties of glass ionomer cements affected by curing methods. Dent Mater 2004;20:45—50.
[10] Dowling AH, Fleming JP. Are encapsulated anterior glass-ionomer restoratives better than their hand-mixed equivalents? J Dent 2009;37:133—40.
[11] Talal A, Tanner KE, Billington R, Pearson GJ. Effect of ultrasound on the setting characteristics of glass ionomer cements studied by Fourier Transform Infrared Spectroscopy. J Mater Sci Mater Med 2009;20:405—11.
[12] Pereira PN, Yamada T, Inokoshi S, Burrow MF, Sano H, Tagami J. Adhesion of resin-modified glass ionomer cements using resin bonding systems. J Dent 1998;26:479—85.
[13] Wilder AD, Swift EJ, Thompson JY, McDougal RA. Effect of finishing technique on the microleakage and surface texture of resin modified glass ionomer restorative materials. J Dent 2000;28:367—73.
[14] Yamazaki T, Schricker SR, Brantley WA, Culbertson BM, Johnston V. Viscoelastic behavior and fracture toughness of six glass-ionomer cements. J Prosthet Dent 2006;96:266—72.
[15] Pikethy M. Nano particles as building blocks. Mater Today 2003;6:36—42.
[16] Duinen Van. Clinical effects at the glass ionomer |tooth |saliva interface. J Dent 2006;34:614—22.
[17] Koenraads H, Van der Kroon G, Frencken JE. Compressive strength of two newly developed glass-ionomer materials for use with the Atraumatic Restorative Treatment (ART) approach in class II cavities. Dent Mater 2009;25:551—6.
[18] Sudsangiam S, van Noort R. Do dentin bond strength tests serve a useful purpose? J Adhes Dent 1999;1:57—67.
[19] Lührs AK, Guhr S, Gänay H, Geurtsen W. Shear bond strength of self-adhesive resins compared to resin cements with etch and rinse adhesives to enamel and dentin in vitro. Clin Oral Invest 2010;14:193—9.
[20] Guimaraes GS, de Morais LS, Elias CN, Pérez CA, Bolognese AM. Chemical and morphological analysis of the human dental enamel treated with argon laser during orthodontic bonding. Dental Press J Orthod 2011;16:100—7.
[21] Geerts SO, Seidel L, Albers AI, Gueders AM. Microleakage after thermocycling of three self-etch adhesives under resin modified glass ionomer cement restorations. Inter J Dent 2010;2010:1—6.
[22] Abdalla AI, El Zohairy AA, Abdel Mohsen MM, Feilzer AJ. Bond efficacy and interface morphology of self-etching adhesives to ground enamel. J Adhes Dent 2010;12:19—25.
[23] Ansari ZJ, Sadr A, Moezizadeh M, Aminian R, Ghasemi A, Shimada Y, et al. Effects of one-yearstorage in water on bond strength of self-etching adhesives to enameland dentin. Dent Mater 2008;27:266—72.
[24] Lim HN, Kim SH, Yu B, Lee YK. Influence of HEMA content on mechanical and bonding properties of experimental HEMA added glass ionomer cements. J Appl Oral Sci 2009;17:340—9.
[25] Sibel A, Jason W, Donald E. Surface protection for newly erupting first molar. Compend Contin Educ Dent 2006;27:46—52.
[26] Eckert GJ, Platt JA. A statistical evaluation of microtensile bond strength methodology for dental adhesives. Dent Mater 2007;23:385—91.
[27] Mauro SJ, Sundfeld RH, Bedran-Russa AKB, FragaBriso ALF. Bond strength of resin modified glass ionomer to dentin, the effect of dentin surface treatment. J Minim Interv Dent 2009;2:45—53.
[28] Phillips RW. Skinner’s science of dental materials. 9th ed. Philadelphia: Saunders; 1991. p. 339—76 [Chapter 12].
[29] Wang L, Sakai VT, Kawai ES, Buzalaf MA, Atta MT. Effect of adhesive systems associated with resin-modified glass ionomer cements. J Oral Rehabil 2006;33:110—6.
[30] Emily Y. Evaluation of shear bond strength of two resin-modified glass ionomer cements. A thesis submitted in partial fulfillment of the requirements for the degree of Master of Science in Dentistry at Virginia Commonwealth University; 2003.
[31] Cheng JT, Itoh K, Kusunoki M, Hasegawa T, Wakumoto S, Hisamitsu H. Effect of dentin conditioners on the bonding efficacy of one-bottle adhesives. J Oral Rehabil 2005;32:28—33.
[32] De Munck J, Van Landuyt K, Peumans M, Poitevin A, Lambrechts P, Bruun M, et al. A critical review of the durability of adhesion to tooth tissue: methods and results. J Dent Res 2005;84:118–32.

[33] Kitasako Y, Burrow MF, Nikaido T, Tagami J. The influence of storage solution on dentin bond durability of resin cement. Dent Mater 2000;16:1–6.

[34] Finer Y, Santerre JP. Salivary esterase activity and its association with the biodegradation of dental composites. J Dent Res 2004;83:22–6.

[35] Yesilyurt C, Bulucu B, Sezen O, Buluti G, Celik D. Bond strengths of two conventional glass-ionomer cements to irradiated and non-irradiated dentin. Dent Mater 2008;27:695–701.

[36] Mesquita MF, Domitti SS, Consani S, de Goes MF. Effect of storage and acid etching on the tensile bond strength of composite resins to glass ionomer cement. Braz Dent J 1999;10:1–60.

[37] Choudhari S, Sudha P, Mopagar V. Clinical evaluation of efficacy of three restorative materials in primary teeth-six month follow up study. Int J Clin Dent Sci 2010;1:45–52.

[38] Aratani M, Pereira AC, Sobrinho LC, Sinhoreti MA, Consani S. Compressive strength of resin modified glass ionomer restorative material: effect of P/L ratio and storage time. J Appl Oral Sci 2005;13:356–9.

[39] Arora V, Kundabala M, Parolia A, Thomas MS, Pai V. Comparison of the shear bond strength of RMGIC to a resin composite using different adhesive systems: an in vitro study. J Conserv Dent 2010;13:80–3.

[40] Glasspoole EA, Erickson RL, Davidson CL. Effect of surface treatments on the bond strength of glass ionomers to enamel. Dent Mater 2002;18:454–62.

[41] Yap S, Yap A, Teo C, Ng J. Polish retention of new aesthetic restorative materials over time. Singapore Dent J 2004;26:39–43.

[42] Coutinho E, Van Landuyt K, De Munck J, Poitevin A, Yoshida Y, Inoue S, et al. Development of a self-etch adhesive for resin-modified glass ionomers. J Dent Res 2006;85:349–53.

[43] Coutinho E, Cardoso MV, De Munck J, Neves AA, Van Landuyt KL, Poitevin A, et al. Bonding effectiveness and interfacial characterization of a nano-filled resin-modified glass-ionomer. Dent Mater 2009;25:1347–57.

[44] El- Askarya PS, Nassifb MS. The effect of pre-conditioning step on the shear bond strength of nano-filled resin modified glass ionomer to dentin. Eur J Dent 2011;5:150–60.

[45] Suryakumari Nujella BP, Choudary MT, Reddy SP, Kumar MK, Gopal T. Comparison of shear bond strength of aesthetic restorative materials. Contemp Clin Dent 2012;3:22–6.

[46] Breschi L, Mazzoni A, Ruggeri A, Cadenaro M, Di Lenarda R, Dorigo E. Dental adhesion review: aging and stability of the bonded interface. Dent Mater 2008;24:90–101.

[47] Van Landuyt KL, Snuwaert J, DeMunck J, Peumans P, Lambrechts P, Van Meerbeek B. The role of HEMA in 1-step self-etch adhesives. Dent Mater 2008;24:1412–9.

[48] Mitsushashi A, Hanaoaka K, Teranaka T. Fracture toughness of resin modified glass-ionomer restorative materials: effect of powder/liquid ratio and powder particle size reduction on fracture toughness. Dent Mater 2003;19:747–57.

[49] Yap AUJ, Mudambi S, Chew CL, Neo JCL. Mechanical properties of an improved visible light-cured resin-modified glass ionomer cement. Oper Dent 2001;26:295–301.

[50] Glaiva D, Gornata K, Skrinjaric I. Retention of glass carboxer fissure sealant after 12 months of clinical trial. Inter J Pediat Dent 2011;21:104.

[51] Carvalho TS, van Amerongen WE, de Gee A, Bönecko M, Sampaio FC. Shear bond strengths of three glass ionomer cements to enamel and dentine. Med Oral Patol Oral Cir Bucal 2011;16:406–10.

[52] Lucas ME, Arita K, Nishino M. Toughness, bonding and fluoride release properties of hydroxyapatite-added glass ionomer cement. Biomaterials 2003;24:3787–94.

[53] Tyas MJ. Clinical evaluation of glass ionomer cement restorations. J Appl Oral Sci 2006;14:10–3.

[54] Suwatviroj P, Louise B, Joseph E. Micro tensile bond strength of tooth-coloured materials to primary tooth dentin. Pediatr Dent 2004;26:67–74.

[55] Knight GM, McIntyre JM, Mulyani. Bond strengths between composite resin and auto cure glass ionomer cement using the co-cure technique. Aust Dent J 2006;51:175–9.

[56] Cho E, Oshida Y, Platt JA, Cochran MA, Matis BA. Microtensile bond strength of glass ionomer cements to artificially created carious dentin. Oper Dent 2006;31:590–7.

[57] Lenzi TL, Cadioli JC, Imparato JC, Raggio DP. Bond strength of high viscous glass ionomer cement to primary dentin. Int J Dent 2011;10:5–9.

[58] Pereira LCG, Nunes MCP, Dibb RGP, Powers JM, Roulet JF, Navarro MF. Mechanical properties and bond strength of glass ionomer cements. J Adhes Dent 2002;4:73–80.

[59] Fritz UB, Finger WJ, Uno S. Resin-modified glass ionomer cements: bonding to enamel and dentin. Dent Mater 1996;12:161–6.

[60] Nicholson JW. Glass ionomer dental cements: update. Mater Tech 2010;25:8–13.

[61] Mazzeo N, Hondrum S. Resin bonding to primary teeth using 3 adhesive systems. J Pediat Dent 1995;17:112.

[62] Rosa BT, Perdigao J. Bond strengths of nonrinsing adhesives. Quint Int 2000;31:353.

[63] Hosoya Y, Kawashita Y, Marshall GW, Goto G. Influence of carisolv for resin adhesion to sound human primary dentin and young permanent dentin. J Dent 2001;29:163.

[64] Sattabanasuk V, Shimada Y, Tagami J. The bond of resin to different dentin surface characteristics. Oper Dent 2004;29:331.

[65] Leclerq G, D’Hoore W, Bouter D, Degrande M, Vreen J. Meta-analytical review of factors involved in dentin adherence. J Dent Res 2001;80:1605–14.

[66] Braga RR, Meira JB, Boaro LC, Xavier TA. Adhesion to tooth structure: a critical review of “macro” test methods. Dent Mater 2010;26:38–49.

[67] Furuse AY, Cunha LFD, Benetti AR, Mondelli J. Bond strength of resin-resin interface contaminated with saliva and submitted to different surface treatments. J Appl Oral Sci 2007;15:501–5.