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Comparative Evaluation of Bond Strength, Micro hardness and Fluoride Release of Two Bioactive Restorative Materials

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Comparative Evaluation of Bond Strength, Fluoride Release, and Microhardness of Two Bioactive Restorative Materials

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Abstract

Purpose: This study aimed to evaluate and compare bond strength, microhardness, and fluoride release properties of two bioactive restorative materials: Zirconomer and Cention N. **Materials and methods:** A total of 120 specimens of two bioactive restorative materials were used for this in-vitro study. Based on the bioactive material used, these specimens were divided into two main groups ($n = 60$): group 1 for Cention N material and group 2 for Zirconomer materials. Then, each group was further subdivided into three subgroups ($n = 20$) according to the type of the performed test. Subgroup A: specimens for the shear bond strength (SBS) test, subgroup B: specimens for microhardness test, and subgroup C: specimens for fluoride ion release (FIR) test. **Results:** Regarding the SBS, the results showed that there were statistically significant differences between the two tested groups ($P \leq 0.001$). Cention N recorded a statistically higher mean SBS value (13.40 ± 1.92 MPa) than Zirconomer (6.91 ± 0.67 MPa). While regarding the microhardness test, the results showed that the Zirconomer recorded a statistically higher mean Vickers hardness number value than Cention N (63.49 ± 2.07, 46.04 ± 5.54 Vickers hardness number, respectively). Regarding FIR, the Cention N group, FIR mean value was 6.86 ± 0.66 mg/l after 1 day, while after 7 days the mean value was 3.54 ± 0.98 mg/l, and after 14 days the mean value was 0.49 ± 0.20 mg/l. **Conclusion:** It was concluded that Cention N had improved bond strength to enamel compared to Zirconomer restorative material. Zirconomer had better microhardness than Cention N. Zirconomer has shown promising results owing to its high fluoride release compared to Cention N, which might contribute to its anticariogenic property. Zirconomer could be the material of choice over Cention N in clinical situations where fluoride release is required in greater amounts.

Keywords: Bioactive, Fluoride ion release, Ion chromatography, Vickers microhardness

1. Introduction

These days, resin-based restorations are in high demand because of their superior adhesion, excellent esthetics, and ease of handling. Good mechanical strength, resistance to wear, tooth-restoration, and interface, esthetics, bond strength, roughness, and surface hardness are the primary requirements for any good material [1]. Dental professionals now have access to a variety of materials due to the rapid advancement of dental medicine in general and restorative dentistry in particular [2].

“Cention N,” is a newly introduced restorative material, it is a modified resin composite with the ability to release hydroxyl, fluoride, and calcium ions, possessing possible anticariogenic qualities. Glass hybrids are a significant category of dental restorative materials that have sufficiently improved their mechanical and wear-resistant qualities in their most recent iteration to be used in stress-bearing areas [3].

In place of amalgam, new technology for restorative materials has been developed. In place of amalgam, a Zirconia reinforced glass-ionomer cement, “Zirconia-improved,” has been developed. It is a modification of glass-ionomer cement that adds more Zirconia to enhance the material's mechanical qualities. Amalgam and resin composite

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have both been proposed as amalgam substitutes [4]. Bulk-filled restorative material that can be applied in a single incremental layer and has a fluoride release profile was developed in response to the need for faster and easier restorative therapy [5]. The release of different remineralizing ions from bioactive restorative materials into the oral cavity and at the tooth–restoration interface improves the longevity of restorations and decreases the rate of recurrent caries. Therefore, this study was performed to compare and evaluate the bond strength, microhardness, and fluoride release of these two bioactive materials, Cention N, and Zirconomer improved.

2. Materials and methods

2.1. Specimens' grouping

A total of 120 specimens of the two bioactive restorative materials were used for this in-vitro study. These samples were divided into two main groups ($n = 60$) based on the bioactive restorative material used:

Group 1: Cention N material.
Group 2: Zirconomer material.

After that, each group was further divided into three subgroups ($n = 20$) based on the type of test.

Subgroup A: specimens for bond strength test.
Subgroup B: specimens for microhardness test.
Subgroup C: specimens for fluoride release test.

2.2. Specimens' preparation

2.2.1. Specimens' preparation for shear bond strength

Forty cleaned extracted bovine incisor teeth were used in the study. The ethical approval for this research was taken from the Research Ethics Committee (REC) (P-MA-21-01) at Al-Azhar University, Faculty of Dental Medicine for Girls'. Separation of the crown of the teeth was made using an Isomet (Isomet Linear Precision Saw; Buehler, Lake Bluff, Illinois, USA) at 0.5 mm apical to cement–enamel junction, underwater coolant. Then, it was rinsed with distilled water and stored in 0.5 % chloramine T solution at 4 °C until use. At the end of the study, these teeth were disposed of in a medical waste container [6]. The crowns were embedded lingually with the labial surface facing upward in acrylic resin blocks [7] fixed in a specially fabricated plastic mold with a diameter of 1 cm height and 2 cm internal diameter [8].

(1) Cention N specimens: the enamel surface of the specimens was conditioned by acid etch for 30 s, rinsed, and then the bond (Tetric N − Ivoclar-Vivadent) was applied to the enamel surface for at least 10 s. The Tetric N bond was light-cured for 20 s using an LED light-curing device (Elipar S10, 3 M, and ESPE, USA) with a 470 wavelength. While Cention N was manipulated (one scoop of powder/drop of liquid resin). Half of the powder was added to the liquid and thoroughly mixed before the remaining powder was added gradually (total mixing time 60 s). A mixture was hand-mixed with a plastic spatula on a mixing pad until it reached a smooth consistency [9].
(2) Zirconomer specimens: Zirconomer was manipulated as per the manufacturer's instructions (two scoops of powder/one drop of liquid). Mixing was done on a mixing pad using a plastic spatula. The dispensed powder was separated into two halves; the first half was introduced to the dispensed liquid and mixed for 5–10 s using a plastic spatula. The other half of the proportioned powder was added and stirred until the mixture became the consistency of thick putty (total mixing time 30 s).
(3) Then each separate mix of both materials was carried to the mold with nonstick aluminum instrument [10] in a specifically made split Teflon mold that was put on the enamel. The split mold was removed after a setting period of 5-min, and the excess material was scraped with a fine-grit diamond (TF-12EF; MANI Dia Burs Inc. Zurich, Switzerland) and polished using a Shofu kit [11].

2.2.2. Specimens' preparation for microhardness test and fluoride ion release test

Disc-shaped specimens were constructed using split Teflon mold with dimensions (6 mm diameter × 2 mm height) [12]. The mold was placed over a glass slab, and both tested materials were manipulated according to the manufacturer's instructions, as previously mentioned.

2.3. Testing procedures

2.3.1. Shear bond strength test assessment

The shear bond strength (SBS) was assessed by placing each specimen in the universal testing machine (Z020; Zwick, Ulm, Germany). It is important to ensure that the enamel surface remained parallel to the machine's path, a steel knife edge movies with a speed of 0.5 mm/min was used to apply a shearing force, the maximum/load in Newton (N) that must be applied to start debonding and converted into Megapascals using the software (the material's load divided by its surface area) according to the following equation [7].

$$\text{Shear bond strength} = \frac{\text{Load}}{\text{Area}} \text{Mpa}$$

2.3.2. Microhardness assessment

The microhardness tests were conducted using a digital Vickers microhardness tester (SCTMC, 1000Z, China) using a 300 g load for 15 s dwell time. Three points were made on each specimen's top surface to measure the Vickers hardness number (VHN). The disc distances from the borders' indentation points must be at least 1 mm, so the mean value was computed, and the VHN was established. Vickers hardness test was measured through the formula:

$$HV = 1.854(F / D2).$$

Where D^2 is the indentation's area (measured in square millimeters) and F is the applied load (kilograms-force). The applied load is usually mentioned along with HV [13].

2.3.3. Fluoride release assessment

Ion chromatography (IC) (ICS-5000 DC; Dionex, Camberley, UK) with suppressed conductivity was used for determination of free fluoride ions. Each specimen was placed in a sealed sterile polypropylene tube with 10 ml purified water and kept in a 37 °C incubator prior to testing. Before measurement, each tube had been properly shaken, and then the samples were taken out and placed in fresh tubes with 10 ml of freshly purified water. They were then cleaned with distilled water, dried with absorbent paper, and incubated. Using IC, the amount of fluoride released from each restoration specimen was measured at different time intervals: 24 h, 7 days, and 14 days. Each specimen's fluoride output was measured in milliliters of distilled water (mg/l) [3]. An IC was fitted with an analytical column and guard column. One milliliter of each solution was put into the instrument's injection loop. The loop was set up so that 250 μl was transferred to the column for analysis (flow rate of 0.5 ml/min). From the chromatogram, the peak associated with fluoride could be easily identified. Fluoride concentrations were calculated using the peak area [14] (Fig. 1).

2.4. Statistical analysis

Data on SBS, VHNN, and fluoride ion release (FIR) were presented as mean and SD values. Statistical analysis was then performed using a commercially available software program (SPSS 19; SPSS, Chicago, IL, USA). Data was evaluated using a one-way analysis of variance test, followed by Tukey's post hoc test when analysis of variance indicated a significant difference [15].

3. Results

3.1. Shear bond strength results

The SBS of the two tested restorative materials (Cention N and Zirconomer improved) used in this in-vitro study was measured using the universal testing machine (Table 1 and Fig. 2).

As a result of the findings, there was a substantial variation in the mean value of the Cention N group as the mean value of the shear bond was (13.40 ± 1.92 MPa) while the mean value for the Zirconomer group was (6.91 ± 0.67 MPa). The result

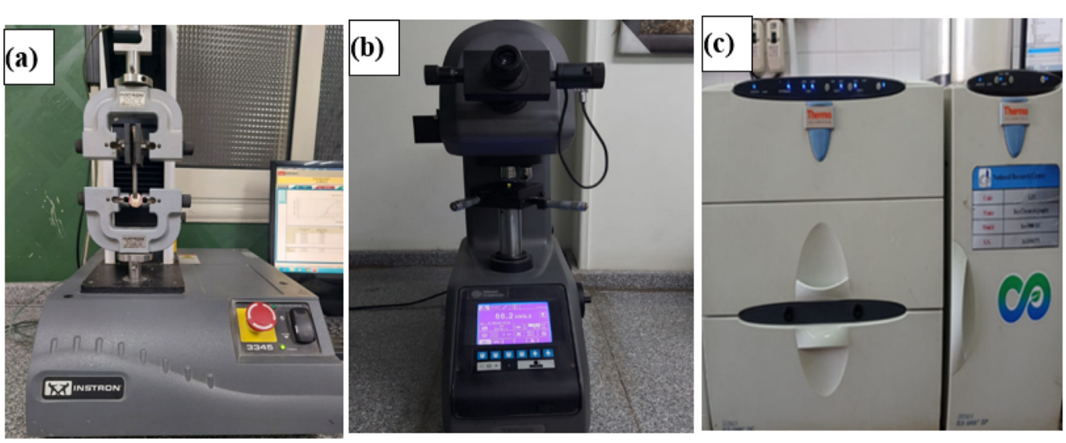

Fig. 1. (a) Universal testing machine. (b) Vickers microhardness tester. (c) Ion chromatography device.

Table 1. The mean and SD of shear bond strength (MPa) for Cention N and Zirconomer improved groups.

Groups	Mean ± SD	*P* value
Cention N	13.40 ± 1.92	0.000^{HS}
Zirconomer	6.91 ± 0.67	

HS, highly significant.

Table 2. The mean and SD Vickers hardness number for Cention N and Zirconomer groups.

Groups	Mean ± SD	*P* value
Cention N	46.04 ± 5.54	0.000^{HS}
Zirconomer	63.49 ± 2.07	

HS, highly significant.

indicates that there was a statistically highly significant difference in the mean SBS value between ($P \leq 0.001$ and confidence 99 %).

3.2. Microhardness test results

The microhardness of the two tested restorative materials (Cention N and Zirconomer improved) was measured in this in-vitro study using a digital Vickers microhardness tester. Independent *t* test indicated that there was statistically highly significant difference in the mean value of hardness between Cention and Zirconomer improved group at the 0.001 level ($P \leq 0.001$ and confidence 99 %). The result showed a higher significant mean value in Zirconomer improved group as the mean value was 63.49 ± 2.07 VHNN while in Cention N group a mean value was 46.04 ± 5.54 VHNN (Table 2 and Fig. 3).

3.3. Fluoride ion release test results

Regarding the Cention N group, FIR mean value was 6.86 ± 0.66 mg/l after 1 day, while after 7 days the mean value was 3.54 ± 0.98 mg/l, and after 14 days the mean value was 0.49 ± 0.20 mg/l. While regarding Zirconomer group, FIR mean value was 37.91 ± 2.18 mg/l after 1 day ranged, while after 7 days, the mean value was 17.98 ± 1.68 mg/l, and after 14 days, the mean value was 5.01 ± 2.02 mg/l (Fig. 4).

4. Discussion

In modern dentistry, the use of "bioactive" materials for reconstructive and restorative operations purposes. It must be noted that depending on the application, the perception of what is actually considered "bioactive" differs. In restorative dentistry, the term "bioactive" usually describes a material's ability to produce hydroxyapatite crystals on its surface. As well as the term "bioactive" in restorative dentistry typically refers to a material's capacity to crystallize hydroxyapatite on its surface [5].

Fluoride-releasing restorative materials, such as glass-ionomer, might be considered among the earliest definitions of bioactive material in the field of restorative dentistry if we accept adhesion to tooth tissues and fluoride release as the basis for bioactivity. Calcium can form on the surface of dental materials because of the repairs' capacity for biomineralization. Since bioactive materials would prevent matrix metalloproteinase enzyme activity and enhance the hybrid layer, this feature might benefit the dental tissue underneath [15].

The current study used two recent bioactive materials, Cention N and Zirconomer. These materials were chosen mainly for their ease of handling. Their mechanical characteristics and manipulative variables were also taken into account. As a novel class of restorative material, Cention N is basically a subset of the category of resin composite

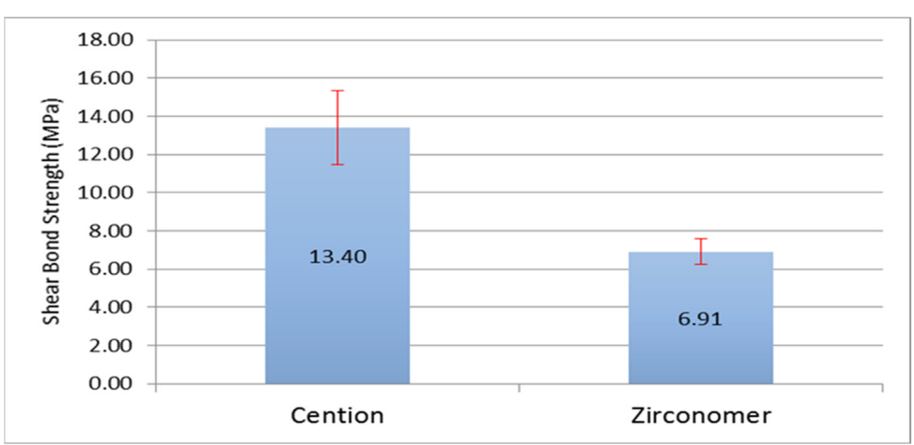

Fig. 2. Bar chart showing mean shear bond strength (MPa) for Cention and Zirconomer improved groups.

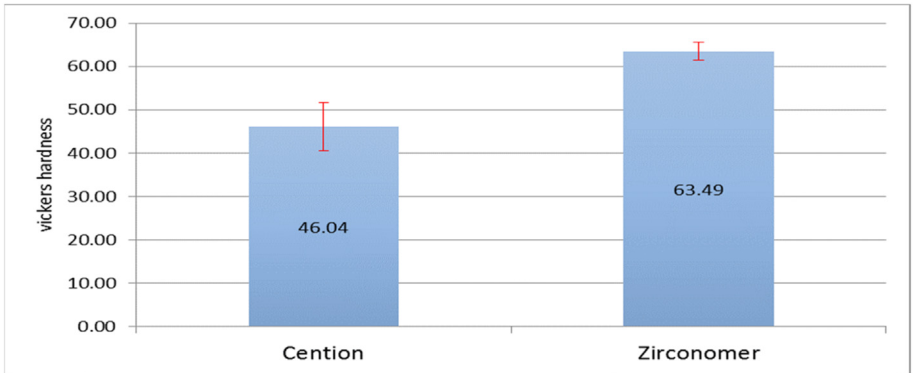

Fig. 3. Bar chart showing mean values of VHN for Cention N and Zirconomer improved groups. VHN, Vickers hardness number.

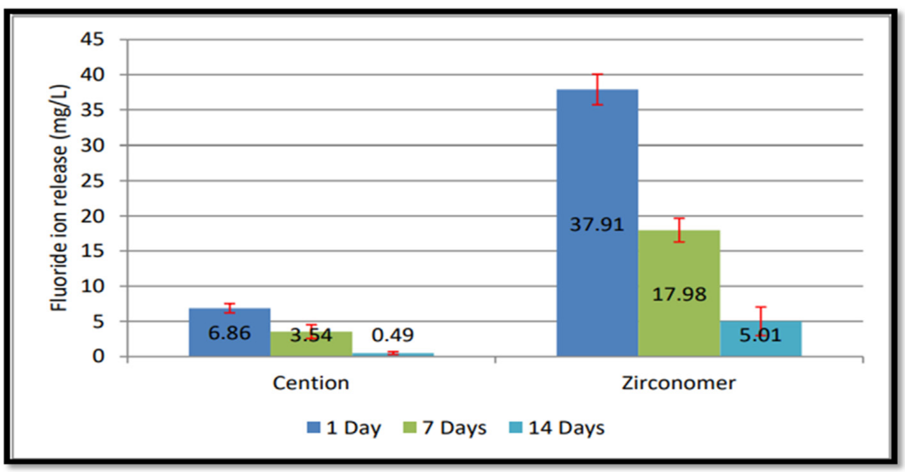

Fig. 4. Bar chart showing the mean of fluoride ion releases (mg/l) for Cention and Zirconomer groups at different time intervals (intragroup comparison).

materials. An alkaline filler in this new class can release ions that neutralize acid [3]. Zirconomer is a strong and reliable alternative that combines amalgam's strength with glass-ionomer's advantages [10]. Both materials, Cention N and Zirconomer, have been chosen for the current study to give a chance to substitute the classic direct restorative materials resin composite and amalgam restoration with new tooth-colored restorative materials with fluoride release profile [3]. The purpose of the current study was to compare these two newly introduced bioactive restorative materials regarding their bond strength, microhardness, and fluoride release.

SBS has been used in the current study as it is fast, easy to perform and also reflects the clinical situation, and is accurate qualitatively when comparing various materials regarding their bond strength value [16]. Bovine teeth specimens were used instead of human teeth for SBS assessment as they are easy to obtain, resemble human dentition, and have a more homogenous composition [8]. Bonding to dentin is a clinical challenge. The tested material in the current study was constructed onto the enamel substrate as it is homogeneous, and that permitted the control of the parameters of bonding variables, which may inadvertently affect the final results [17]. To ensure improved adhesion, an adhesive system is strongly recommended with Cention N as it is explicitly described in the Cention N recommendations of the manufacture directions to "follow the preparation guidelines for the placement of amalgam restoration when using Cention N without an adhesive" [18]. Therefore, in the current study Cention N specimens were bonded to the enamel substrate using Tetric Bond Universal in total-etch mode [19].

In this study, the specimens were fabricated in the shape of discs with 2 mm thickness [19] as in most studies related to tooth-colored restorative materials placement techniques. The thickness of the increments was 2 mm thickness as it allowed proper polymerization, preventing air bubble incorporation, and it is also more time and effort consuming.

Hardness is one of the physical surface properties as it is the resistance of the material to indentation or penetration. Vickers hardness tester has been selected to measure the microhardness due to the ease of specimen preparation, simplicity of the test method, and availability of the equipment. Hardness has been widely used as a method of investigating factors that influence the degree of conversion of resins and for characterization of the mechanical quality of a polymer. It is also a nondestructive testing method so that the test specimen can be reused [10].

The fluoride release from most of the bioactive restorative materials has three possible explanations: bulk diffusion, diffusion via pores and cracks, and surface loss. It is also related to the experimental procedures, specimen size, storage medium, frequency of storage environment changes, and quantity of media used to measure fluoride levels [20]. In this study FIR test was performed, and the storage media was selected to be distilled water in an attempt to replicate the saliva's moist oral environment. Since synthetic saliva fails to exhibit a clinically significant degree of microbial activity, the flushing effect of salivary flow was replicated using distilled water; it is also preferred compared to artificial saliva because of the latter's high viscosity and use of ions as a storage medium. These ions may affect the release of fluoride ions from restorative materials. As a result, they cause an incorrect evaluation of the fluoride ions released [11].

IC was utilized in this investigation to accurately measure the fluoride release concentration up to 0.001 ppm. IC has an advantage over the commonly used ion-selective electrode. It can differentiate between fluoride ions and fluoride complexes released from the materials [3]. An additional benefit of IC is that it usually takes only 10 min or less to analyze a specimen [21]. Based on many comparable investigations, the time intervals of 1, 7, and 14 days after setting have been chosen for the analysis of the fluoride ion concentration [20]. It appears that 34 ISO 4049's suggested 24-h exposure to water is insufficient to gauge a material's behavior in a therapeutic context [22].

Regarding the results of the SBS test, the findings of the present study indicated that Cention N recorded a higher mean SBS value (13.40 $\pm$ 1.92 MPa) compared to Zirconomer that recorded 6.91 $\pm$ 0.67 MPa. This might be due to the composition of Cention N, which contains a strong polymer structure and a stable self-cure initiator. The organic monomer component of four distinct dimethacrylate together make up 21.6 % of the product's final weight. In addition to the cross-linking components, UDMA, PEG-400 DMA, DCP, and an aromatic aliphatic-UDMA combine the polymerization reaction. Moreover, apart from the inorganic fillers is the glass filler made of barium aluminum silicate that also contains ytterbium trifluoride, which act to relieve the shrinkage stress along with the silanes that link the filler particles together and strengthens the bond by creating a chemical bond between the matrix and the glass surface [17].

This outcome aligned with a prior study that showed that Cention N had a stronger shear bond than Zirconomer, despite the latter's mechanical properties being improved by the addition of Zirconia filler particles. This is because the latter has a higher tendency to fail early due to moisture sensitivity, as it does not affect Zirconomer moisture sensitivity or early bond failure tendency [23]. The current study SBS result was also in accordance with a previous study which investigated and discovered that better adherence of Cention N to the tooth structure is implied by greater SBS, and explained that the results were due to etching of enamel, bonding agent penetration and creation of a hybrid layer to create a microchemical bond beside the (Aromatic aliphatic-UDMA) present in the Cention N that cross-link with tooth structure [24].

Regarding the results of a microhardness test, the current study's findings revealed that the Zirconomer improved group had a higher significant mean VHN value than Cention N (63.49 $\pm$ 2.07 VHN) (46.04 $\pm$ 5.54 VHN), respectively, this could be explained by the fact that the material's excellent tolerance to occlusal loads and toughness is further enhanced by the uniform integration of Zirconia particles into the glass portion. Additionally, polycrystalline material is present in Zirconia-ceramic fillers. The filler/volume ratio of the material was improved using Zirconomer. The restorative materials' surface characteristics were found to be influenced by the following factors: resin type, particle size, filler/volume ratio, material content, and filler size, which explain that the resin coating in Cention N reduced the surface hardness [2]. Another explanation for the higher mean VHN values in Zirconomer improved is that the Zirconomer matrix has a good balance of large and small-sized particles, suggesting that the bonds are comparatively strong between the particles and the hydrogen salt matrix. Moreover, the Zirconia has superior strength, toughness, hardness, and resistance to corrosion because of a process known as transformation toughening. Crucially, Zirconomer is made with a high-strength manufacturing process that uses cold isostatic pressing to shape Y-TZP (Yttrium Tetragonal Zirconia Particles), producing stable, nonsintered objects in the green stage with high primary density that resemble chalk [25].

The results of this study were consistent with those of a previous study, which showed that the surface microhardness appeared to be significantly influenced by the polarity of the matrix, the degree of cross-linking of the continuous matrix, the kind and size of filler particles, and the number of photoinitiators, all influenced the behavior of restorative materials. The lower filler amount in Cention N (57.6 vol%) and the greater inorganic filler particle size (0.1−35 μm) were responsible for the lower values of VHN of Cention N [12]. However, this current investigation was in disagreement with a previous study, which revealed that compared to Zirconomer, Cention N displayed greater microhardness values. This could be because Cention N is related to the size of the inorganic filler nanoparticles. It made Cention N's microhardness better and enhanced, making it a more clinically appropriate option for clinical treatments [2].

Regarding the FIR results, the current investigation indicated that Zirconomer recorded a higher significant mean FIR value either after 1 day, 7 days, or 14 days than Cention N. The Zirconomer-improved specimens' exciting turn: after 24 h, the fluoride release value was low, but after seven and 14 days, it increased. This could be due to the chemical makeup of the Zirconomer, its physical characteristics, and its mixing consistency (an 8: 1 powder-to-liquid ratio) all of which might contribute to its high fluoride release. Moreover, the fine glass particles in the Zirconomer could also be the cause of the fluoride's fast-release pattern.

According to the results of the current study, it was reported that Zirconomer demonstrated a fluoride discharge that was more explosive than Cention N. The Cention N might include fillers such as barium, aluminum, silicate glass, ytterbium trifluoride, calcium fluorosilicate (alkaline) glass, Tetric N-Ceram technology isofiller, and calcium fluorosilicate glass. Fluoride ions are released by 78.4 wt% of the filler material and only 24.6 wt% of the final restorative material out of all of this. Furthermore, Cention contains surface-modified filler materials that are resistant to deterioration and may release trace amounts of fluoride ions [3].

Another explanation, is that the fluoride released from the Zirconomer group, which absorbs water and releases fluoride. The manufacturer claims that the glass-ionomer particles were finely micronized, and this result was consistent with reports because the surface area of smaller glass particles is greater, increasing the acid−base's reactivity and ability to release fluoride [26]. This outcome was also in agreement with another study that discovered the maximum mean fluoride release occurred on the first and third days. This was ascribed by the study to the first "burst effect," which can result in high initial fluoride content due to surface wash-off and explosive fluoride release. This effect is clinically referred to as the "burst effect," which reduces the amount of residual bacteria in the restored cavities and improves dentin and enamel remineralization. Following that, there is a sharp fall over the next few days, which is probably merely the result of mass diffusion and gradual, continuous diffusion via the material's pores [3]. This investigation, however, disagreed with a prior study, which found that the amounts of fluoride ions in both the Cention N and Zirconomer materials increase gradually over time [27]. This result may be explained by the optimum molecular weight (Mw) of the polyacrylic acid, which encourages the formation of polysalt bridges and cross-linking in the set cement's structure. The availability of acidic groups for better acid−base reactions and carboxylic acid groups for increased acid−base reactions would both increase with an optimum Mw [28].

4.1. Conclusion

Within the scope of the current study's limitations and findings, it was possible to conclude that:

(1) Cention N had improved bond strength to enamel compared to Zirconomer restorative material.
(2) Zirconomer had better microhardness than Cention N.
(3) Zirconomer has shown promising results owing to its high fluoride release compared to Cention N, which may contribute to its anticariogenic property and could be the material of choice over Cention N in clinical situations where fluoride release is required in greater amounts.

4.2. Recommendation

Further, in-vivo studies are warranted to gain a more clinical close insight into the properties of these promising restorative materials. Moreover, further mechanical properties should be evaluated as modulus of elasticity and flexural strength to assess the clinical performance of these newly introduced bioactive materials.

Ethical approval

The ethical approval for this research was taken from the Research Ethics Committee (REC) (P-MA-21-01) at Al-Azhar University, Faculty of Dental Medicine for Girls' and this investigation was not funded by any agency.

Conflict of interest

There are no conflicts of interest.

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Paper extracted from Master thesis entitled "Comparative Evaluation of Bond Strength, Microhardness and Fluoride Release of Two Bioactive Restorative Materials."

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