Dentistry 3000

Vol 13, No 1 (2025)
ISSN 2167-8677 (online)
DOI 10.5195/d3000.2025.1001

Shear Bond Strength and SEM/EDX of Two Bioactive Bulk Fill Resin Base Composite Restorations

Rana Fahad Ahmed Al Azawee, Nadia Modhafer Ahmed Al Shakir

College of Dentistry, University of Mosul, Iraq

Abstract

Objective: To compare the shear bond strength of two bioactive bulk-fill dental restorative materials to sound dentin after aging in artificial saliva and to evaluate the mode of failure and elemental analysis by SEM/EDX**. Material and Methods**: Forty maxillary premolars extracted due to orthodontic reasons from subjects with ages between 14 and 30 years were used in this study. The teeth were divided into two groups according to the type of composite, Predicta Bioactive or Cention Forte. Each group had samples stored or not stored in artificial saliva. Occlusal dentin of all teeth was exposed by cutting 2mm below the deepest point on the occlusal surface of each tooth's crown. Each restorative material was applied according to the corresponding manufacture instructions. The shear bond strength was measured between the composite and dentin and by using SEM-EDX to evaluate the mode of failure and element analysis. **Results**: The study revealed that Cention Forte, after storage, exhibited the highest shear bond strength (10.13 MPa), followed by its non-stored samples (8.38 MPa), while Predicta Bioactive showed the lowest values, especially in the non-stored samples (6.38 MPa). **Conclusion**: Cention Forte had stronger bonding than Predicta Bioactive.

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Citation: Al Azawee RFA, et al. (2025) Shear Bond Strength and SEM/EDX of Two Bioactive Bulk Fill Resin Base Composite Restorations. Dentistry 3000. 1:a001 doi:10.5195/d3000.2025.1001
Received: July 27, 2025
Accepted: August 4, 2025
Published: September 9, 2025

Email: Nadia.Mudhafar@uomosul.edu.iq

Introduction

Composite restorations are widely used in direct esthetic dentistry due to their ability to mimic various shades and translucencies, providing high-quality aesthetic outcomes. Additionally, the incremental placement of conventional composite materials can introduce voids and interfacial bonding failures, leading the remaining tooth structure and restoration interface to greater stress [1,2].

A main affecting factor for maintaining a restoration chemically bonded to the tooth structure is its ability to withstand shear stresses (that can be defined as the stresses created at the interface between restoration and tooth surface) which are caused by perpendicular or parallel forces acting on the tooth surface [3].

The oral environment is dynamic and complex, exposing restorative materials to variations in pH, temperature, microbial activity, and mechanical stress, dental tissues are in constant ionic exchange with fluoride, calcium, and phosphate, regulated by saliva, which plays a crucial role in maintaining a balanced mineral environment [4]. With this understanding, the focus of restorative dentistry has gradually shifted from biocompatibility to bioactivity, emphasizing materials that can interact with the oral environment to promote remineralization and inhibit bacterial growth [5]. Bioactive resin composites are designed to discharging remineralizing ions, elevating pH levels, and generating hydroxyapatite, thereby enhancing the longevity and effectiveness of restorations [6].

Several bioactive composites have been developed with properties aimed to combating caries while maintaining their stability under occlusal forces, temperature changes, and enzymatic degradation [7,8]. Among these materials and for direct restorations, Predicta Bioactive which is a bulk-fill resin composite was designed. According to the manufacturer, it demonstrates high compressive, tensile, and flexural strengths while also releasing fluoride, calcium, and phosphate ions to facilitate the formation and mineral apatite remineralization at the tooth-restoration interface [9].

Similarly, Cention Forte (Ivoclar Vivadent) belongs to the Alkasite material family and is characterized by its high flexural strength and esthetic properties. It contains an alkaline filler that releases hydroxide ions to

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regulate pH during acid attacks, preventing demineralization and promoting remineralization through the release of calcium and fluoride ions. Additionally, it exhibits moderate viscosity and strong mechanical properties [10].
The aim of this study was to evaluate and compare shear bond strength of two bioactive bulk-fill dental restorative materials to sound dentin after aging in artificial saliva, and to evaluate their mode of failure and element analysis by SEM/EDX.

Material and Methods

An ethical approval was received from the "Research Ethics Committee" of the College of Dentistry, University of Mosul (no. UoM. Dent.25/1013) Because the research study used extracted human teeth.
Forty maxillary premolar teeth extracted for orthodontic purposes from patients aged 14 to 30 years were collected for this study from different health center in Mosul, each tooth was examined using a stereomicroscope at10X magnification. Only teeth that were free of caries, cracks, attrition, abrasion, or restorations were included. The teeth cleaned to remove debris using an ultrasonic scaler, then were immersed in a 0.1% thymol solution (DBH, England) for 48 hours to ensure disinfection and subsequently stored in deionized water (Almansur factory, Iraq) until the time of the study, for the next mounting step the teeth samples were prepared [11-13].
For mounting the teeth a retentive tube polyvinyl chloride (PVC), 2cm in diameter and 2cm in length, was used as a mold, each tooth was positioned with the aid of a dental surveyor and a sticky wax (Hoppegarten, Germany) was used to attach the tooth to the rod of the surveyor, the selected teeth with their roots were embedded at the tube center and parallel to its long axis to a level of 2 mm below the cement-enamel junction simulate the position of the tooth in the alveolar bone [14,15].
Forty teeth were divided randomly into two groups according to the type of composite restorative materials (20 teeth for each group). Then, each group was subdivided into two subgroups. The first 10 teeth were not stored in artificial saliva, while the second 10 teeth were stored for 30 days in artificial saliva. All teeth were tested for shear bond strength using a universal testing machine (Hongjin HAS-UT-5PC, China) and for mode of failure and element analysis using SEM/EDX (Axia ChemiSEM, Holland).
The occlusal dentin of each tooth was exposed by cutting 2 mm below the deepest point on the occlusal surface of each tooth's crown. this cut was made using a diamond coated separating disc attached to a slow-speed hand piece with water coolant. perpendicular to the long axis of the tooth the cutting was made. Occlusal dentin surface of all specimens was polished using 600-grit Wet Silicon Carbide abrasive papers for ten time in a circular motion under running tap water to obtain flat dentin bonding surface [12,16].
For Predicta Bioactive (Parkell, USA) bulk fill resin composite, an etch and rinse protocol was used following the manufacturer's recommendations, where the dentin surface was exposed, 37% phosphoric acid gel (Spident, Korea) was used for 15 seconds and then rinsed with water for 15 seconds. The teeth were dried with oil-free air for 5 seconds [17]. According to the manufacturer's instructions, one layer of G-Premio Bond (Universal Bond Quick, GC, Japan) was rubbed onto the dentin surface for 20 seconds with a microbrush, the bond remained on the dentin surface for 10 seconds and was dried at the maximum airflow rate for 5 seconds. Finally, the adhesive layer was light cured at a light intensity of 1000 mW/cm2 for 10 seconds using light emitting diode light curing unit (Rogin Dental, China) at a 1mm distance [18]. For a standardized distance we used celluloid crown which cut 1mm from cervical margin so the distance between occlusal surface of celluloid crown and occlusal surface of coronal dentin is 1mm.
A custom-made Teflon mold was designed to standardize bulk fill composite application on bonded dentin to produce a 4mm diameter and 2mm height [16]. Each restorative material was applied according to the corresponding manufacture instructions utilizing the bulk placement technique. For the Cention Forte (Ivoclar Vivadent, Schaan, Liechtenstein), the primer was dispensed as one drop in a dish and mixed with the applicator for 5 seconds, applying on dentin for 10 s, blowing by air pressure for 5 seconds (self-cure) [19]. Cention Forte capsules were activated by pressing the plunger on a flat surface to allow mixing of the powder and liquid. Immediately after activation, the capsule was inserted in the amalgamator (capsule mixer) (Softly8, Italy) and mixed for 17 seconds at room temperature (21°C) (low temperature led to delay setting of material). After mixing, immediately the capsule was placed into the applicator, clicked 3 clicks and dispensed to fill the mold, keeping the tip immersed in the material to prevent the formation of air bubbles [20].
The Predicta Bulk Bioactive (Parkell, USA) composite was delivered on bonded dentin by a spiral nozzle to fill the mold and slowly express the composite as we withdraw the tip, keeping the tip immersed in the material to eliminate air entrapment.
A celluloid strip was placed over the restoration to achieve a smooth, flat surface to prevent oxygen inhibition surface layer, then the material was light-cured using a light-emitting diode (LED) curing unit with an intensity of 1000 mW/cm² (Rogin Dental, China) in a one step for 20 seconds. The tip of light-curing was positioned perpendicularly to the occlusal surface of the restorative material, maintaining a standardized distance of 1 mm (equal to the thickness of a metal plate from a custom-made Teflon mold). Additional curing was performed for 20 seconds on each side of the restorative material to optimize polymerization [21,22].
Each group were subdivided into two subgroups. The first subgroup was not stored in artificial saliva. All samples were stored in plastic container containing 30 ml of deionized water in an incubator (Binder, Germany) at 37± 2°C in 95% humidity for 48 hours until subjected to shear bond strength test [13,23]. The second subgroup was stored in artificial saliva. Samples were placed upright inside a plastic container. Each container was then filled with 30 ml of artificial saliva (pH 7) and securely covered. The containers were incubated at 37 ± 2 °C and 95 % humidity for 30 days, and they were replenished every week until the end-point time (30 days) [24,25].
An universal testing machine (Hongjin HAS-UT-5PC, China) was used to measure the shear bond strength between the composite and dentin. All samples were loaded at a speed of 0.5 mm per minute until de-bonding occurred with a maximum failure load that was recorded automatically in Newton (N) by a computer connected to the testing machine [16]. To calculate shear bond strength in MPa, the maximum force was divided by the surface area of the sample.
Shear bond strength = (**F**) Fracture Load (N)/ (**A**) Surface area (mm2)
The surface area (A) was calculated from the following equation:
$A = \pi r^2$ Where $\pi = 3.14$ r = Radius of each specimen (2mm),
$A = 3.14 * 2^2 = 12.56 mm^2$.
To identify the mode of failure, all samples (de-bonded dentin surfaces) following shear bond strength test were examined using a stereromicroscope at 40X magnification [27]. Also, Scanning Electron Microscopy (SEM) of three de-bonded surface samples were done to determine the micromorphological topography at 5000X and the mode of failure at 1300X [26].
The mode of failure was categorized as adhesive (failure at the adhesive-substrate interface), cohesive (within the material mass), or

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mixed (a combination of adhesive and cohesive) [23].
All corresponding samples (de-bonded surfaces and fractured composite stubs) were collected after shear bond strength testing to prepared for SEM/EDX (Axia ChemiSEM, Holland), the teeth samples from each subgroup (non-storage and storage in artificial saliva for 30 days) were subjected to longitudinal sectioning (sectioning at the middle of mesio-distal direction) to the surface of acrylic base and then by using slow speed hand piece with cutting diamond disc each half sectioned horizontally below cement-enamel junction near the acrylic base, then one of the obtained sections from each tooth sample was randomly selected and cleaned in an ultrasonic water bath (Granbo, China) for 3 min, in order to remove the debris and left to dry for 24 hours [28]. The corresponding samples were secured to the aluminum stubs using carbon double-sided tape. After that, a thin gold coating (15nm) was spattered on the surface for 20 seconds to determine the mode of failure and micromorphological topography. The SEM system was adjusted to a 30 kV accelerating voltage for this purpose [29]. The weight percentages of chemical elements in the de-bonded dentin and restorative material core for each tooth have been identified by analyzing the chemical composition using EDX data. In addition, the calcium-to-phosphorus (Ca/P) ratio was determined for both the storage and non-storage groups [30].
The statistical analysis included the following tests: “two-way analysis of variance” (ANOVA) was analyzed the results for shear bond strength and “Duncan's multiple range” to identify the significance among groups at P≤0.05; independent sample t-test was used to compare different groups to assess the effect of storage in artificial saliva on the shear bond strength. Statistical significance was set at p<0.05; and chi-square test to compare different groups to assess the effect of storage in artificial saliva on the mode of failure.

Results

Shapiro Wilks test showed that the data were normally distributed (p<0.05). in most of the groups,
The results of the descriptive statistics that included the minimum, maximum, mean, and standard deviation value of shear bond strength of all groups of the study as shown in Table 1.
The highest shear bond strength was observed for Cention Forte, whereas the lowest shear bond strength was observed for Predicta Bioactive.

Mode of failure

De-bonded tooth samples were inspected using a steromicroscope at 40X magnification as shown in Figure 1.
The most common failure pattern was mixed except for Cention Forte stored in artificial saliva, which had a cohesive failure.

SEM\EDX analysis

SEM imaging was performed at 5000X magnification to examine the de-bonded areas of all restorative material core and the coronal dentin samples stored or not in artificial saliva. The de-bonded samples were analyzed to assess morphological changes at these critical regions (Figures 2 and 3).
SEM for Predicta bioactive showed a slightly precipitation of hydroxyapatite (HA) at the de-bonded area of composite for both groups (Figure 4).
SEM for Cention Forte showed precipitation of hydroxyapatite (HA) at the de-bonded area of composite for both the samples not stored in artificial saliva (B1) and stored (B2) (Figure 5).
SEM images showed the presence of "crystal-like" deposits at de-bonded coronal dentin areas in the Predicta Bioactive samples in both stored and not stored in artificial saliva (Figure 6).
SEM images of Cention Forte samples, both not stored and stored in artificial saliva showed that de-bonded coronal dentin areas had “crystal-like” depositions (Figure 7).

EDX analysis

EDX analysis was conducted to evaluate the elemental composition of the de-bonded areas of all restorative materials core and the coronal dentin samples. This analysis aimed to detect compositional changes associated with aging in artificial saliva. The weight percentage of calcium and phosphorus elements were used to calculate the calcium/phosphorus ratios as illustrated in the following equation:

$$Ca/P = \frac{\text{Calcium element weight percentage}}{\text{Phosphorus element weight percentage}}$$

The EDX spectra showed low concentration of phosphorous (P) for samples not stored in artificial saliva (Figure 8).
The EDX spectra showed low concentration of phosphorous (P) Centrion Forte samples (Figure 9).
The EDX spectra of Predicta Bioactive for both not stored and stored in artificial saliva showed a change in the spectra for both phosphorous (P) and calcium (Ca). Predicta Bioactive samples showed increase in the spectra of calcium (Ca) with slightly decrease in the spectra of phosphorous (P) (Figure 10).
The EDX spectra for Cention Forte both samples showed a change in the spectra for both phosphorous (P) and calcium (Ca). Cention Forte samples stored in artificial saliva showed an increase in the spectra of calcium (Ca) that was higher than Predicta Bioactive with slightly decrease in the spectra of phosphorous (P) (Figure 11).
The mean Ca/P ratios was (1.84±0.71) in Predicta Bioactive samples stored in artificial saliva, which is higher than the ratio of natural HA (1.67). This indicated apatite deposition, which confirms the SEM analysis that showed apatite deposition at the restoration surface.
The mean Ca/P ratio for Cention Forte samples stored in artificial saliva was (2.63±0.62), which was higher than the ratio for natural HA (1.67). This indicated apatite deposition, which would support the SEM analysis that showed apatite deposition at the restoration surface.
For Predicta Bioactive samples not stored in artificial saliva exhibited a mean Ca/P ratio of (1.81±0.45), significantly above the natural hydroxyapatite (HA) ratio of dentin (1.67), suggesting a composition like native minerals. In samples stored in artificial saliva, the Ca/P ratio at the de-bonded area of the coronal dentin samples increased to (2.71 ± 0.38). The rising ratio, significantly above that of natural dentin hydroxyapatite, indicates the development and deposition of a hydroxyapatite-like crystals.
The EDX elements for samples not stored in artificial saliva of Cention Forte detected the mean Ca/P ratio (1.97±0.38) slightly higher than natural HA ratio (1.67) for dentin. While after storage in artificial saliva, the coronal dentin area of Cention Forte showed Ca/P ratio about (4.01±1.98), which is higher than that for natural HA ratio for dentin. This indicates HA precipitation.

Discussion

Alkasite-based tooth-colored restorative material is a hybrid that releases calcium, fluoride, and hydroxyl ions, which exhibit effective anti-cariogenic properties. This novel material combines the advantages of glass ionomer cements (GICs) and resin-based composites. Dual-cure capability allows bulk placement with or without adhesive [31].
Shear bond strength test was selected to evaluate bonding strength of the two different restorative materials when applied to flat (mid-coronal) dentin. The clinical importance of this test because of it closely simulates the shearing forces found at the tooth–restoration interface [32].
The aging procedure is essential in determining the bond durability, one of the factors which affect the properties of restorative material is Saliva which is a slightly acidic body fluid having a pH scale of 6–7 and whose main ingredient is water (99%).

Artificial saliva was used to simulate the wet oral environment. The teeth were stored in artificial for a period as the restoration remains in continuous contact with saliva in the oral cavity [33].
In the current study, teeth were stored in artificial saliva in an incubator at (37 °C ± 1) for 30days at pH 7. This was performed to mimic the oral cavity's environment for assessing the behavior of restorative materials [34].
In this vitro study, bonding effectiveness and bond durability after artificial aging (30 days in artificial saliva) were investigated through evaluate 1) shear bond strength of these tested material according to storage condition 2) micromorphological analysis of these materials at debonded dentin area by SEM identify the main failure modes 3)elementary analysis of both core of materials and debonded dentin area by SEM/EDX analysis. The null hypothesis was rejected since there were significant differences of two bioactive bulk-fill dental restorative materials in values of bonding to sound dentin and failure patterns after aging in artificial saliva
In the current study, Cention Forte showed the greatest value of shear bond strength. The highest mean in the Cention Forte with storage group (10.13 MPa), followed by Cention Forte without storage (8.38 MPa). These values were significantly higher compared to Predicta Bioactive, indicating that Cention Forte exhibited superior bonding performance, particularly after being stored in artificial saliva.
This could be due to the strong mechanical properties of Cention Forte which is due to its chemical composition as its monomer matrix consists of a mixture of urethane dimethacrylates (UDMA), tricyclodecan-dimethanol dimethacrylate (DCP), tetramethyl-xylylendiurethane dimethacrylate (aromatic aliphatic-UDMA) and polyethylene glycol 400 dimethacrylate (PEG-400 DMA), which interconnects (cross-links) during the process of polymerization leading to stronger mechanical properties. This agrees with Bassiouny *et.al*. (2024) [23], who valuate the shear bond strength (SBS) of Cention Forte and Tetric N-Ceram Bulk Fill composite
Methacrylate-Modified Polyacrylic Acid present in the primer offering dual adhesion mechanisms (mechanical and chemical) by forming micro-mechanical interlocking provided by the surface roughness, most likely combined with chemical interaction through its acrylic / itaconic acid copolymers. Polyacrylic acid can remove the smear layer and leave the smear plug, producing a partial demineralization of the dentin, leaving hydroxyapatite around the collagen fibers, allowing the chemical interaction of the carboxylic groups with dentin hydroxyapatite [35]. These results agree with previous work [36], who determine and compare flexural strength and microhardness of Cention N with other materials at a distinctive period in artificial saliva.
The current study also agrees with previous work [37,38] that compared the bonding efficacy of three bioactive self-adhesive restorative systems to dentin after storage in artificial saliva revealed that using of primers prior to application of alkasite-based restorative material is highly recommended and show high microshear bond strength values for both immediate and storage groups of Cention Forte with primer among other tested groups.
The contrary, Predict Bioactive showed a lower SBS (with p value less, than or equal 0.05) which could be attributed to its unique monomer composition , a novel monomer (Poly-2-HEMA), which has been advocated to reduce the risk of potentially toxic effects of BisGMA-based compounds that cause increased solubility[39] by promotes the formation of an unstable aqueous gel that is susceptible to hydrolytic degradation ,HEMA has a negative interaction with 10-MDP in the adhesive agent (G-Premio BOND) used in this study, which significantly reduces the demineralization of hydroxyapatite. This decreases the formation of MDP Ca salts and partially inhibits the deposition of the nanolayers, which are necessary to obtain an adequate chemical interaction with the dentin substrate [40].
This may be explained the low viscosity of Predicta bulk bioactive composite (as the Predicta Bioactive type used in the current study is low viscosity type as claimed by manufacture). the morphology of the filler particles and the amount of filler loading that improved the mechanical and physical properties of the resin-based composites. Reduced filler content of bulk-fill composites results in an increase in the polymerization shrinkage and shrinkage stresses to potentially debond the material from dentin during polymerization [41,43].This come in agreement with the study conducted by Hegde et al., 2023) [44] , who compare microtensile bond strength of high-viscosity bulk-fill composites and low-viscosity bulk-fill composites revealed that the high-viscosity bulk-fill composites exhibited the highest microtensile bond strength in comparison to other resin-based composites, nanocomposites, and bulk-fill flowable composites.
Although aging in artificial saliva did not yield a statistically significant overall effect. Cention Forte demonstrated the highest SBS after storage, Predicta Bioactive also exhibited improved SBS following storage. The use of ion-releasing materials in restorative dentistry may contribute to the reduced activity of proteases such as metalloproteinases (MMPs) and cathepsins involved in collagen degradation. Such enzymes are considered one of the main causes for reduction of bonding longevity when simplified bonding systems are applied in dentine with self-etching or etch-and-rinse protocols [45], thus reducing the enzymatic degradation at the bonding interface. It may be also possible that in the case of diffusion of calcium and phosphate ions through permeable hybrid layers, these may precipitate and crystallize in complex calcium-phosphates and inhibit MMPs through the formation of a Ca-PO/MMP complex [46].
Cention Forte storage group had the mean value (10.13±2.11) in which the increase was statistically significant in comparison to the non-storage group of the same material (8.38 ± 1.12)
After storage, Predicta Bioactive showed some improvement, but not nearly as much as Cention Forte, according to the results. For Predicta Bioactive, its capacity to release ions that promote bond stability implies it can aid in remineralisation and improve the connection. This material demonstrates great potential as a bioactive bulk-fill composite, which can release F, Ca, and P ions, for long-term restoration treatments. This bulk-fill resin-based composite is simple to apply, cures in two stages, and has optical properties [47].
presence of "HEMA" in Predicta bioactive, a hydrophilic monomer with enhanced solubility which may explain its ability to release more ions and enhance its bioactivity[48] also downsizing bioactive glass particles to nano-size improves the alkalizing and hydroxyapatite-forming Based on the information provided by the manufacturer and validated by the EDX study, one of the unique compositions of Predicta Bioactive as manufacture claimed is titanium dioxide (TiO_2) TiO_2 nanofller incorporation has no effect on the shear bond strength of the flowable composites[49] however ,may enhance the bioactivity and hydroxyapatite (HA) formation of this material. This in agreement with (Witkowska *et al.,* 2024) [50], Who indicates the good bioactivity of titanium oxide layers. Under simulated biological conditions at pH 7.4 found that TiO_2 surface shows predominantly negative properties, creating an electrostatic environment that attracts oppositely charged ions, such as calcium. Subsequently, the positively charged Ca^{2+} undergoes a reaction with negatively charged PO_4^{3-} and CO_3^{2-}, leading to the formation of a surface layer containing Ca–P. Over time, this layer may crystallize into hydroxyapatite [51,52].
The current study found that the storage group of Cention Forte exhibited the highest

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shear bond strength, having a low incidence of mixed failure and a higher incidence of cohesive failure. On the other hand, the non-storage group of Predicta Bioactive revealed the lowest shear bond strength, associated with a greater rate of adhesive failure. These findings align with Sabatini's work, [53] which demonstrated a link between the highest bond strength and mixed failure, while the lowest strength corresponded to adhesive failure. Evidence from several studies supports the hypothesis that cohesive failure in dentin is related to high bond strength [54] perhaps because of the strong bonding to dentin developed by universal adhesives [55].

Scanning electron microscopy (SEM) is often used for the morphological analysis of adhesive-dentin interfaces. Many studies have investigated the relationship between bonding performance and interfacial properties [56], (SEM) analysis for Cention Forte showed the interface was completely sealed as an acid-resistant, resin-dentin interdiffusion zone with resin tags extending into the dentinal tubules. close the dentinal tubules in storage group while partially cover dentinal tubules in non-storage group, indicating that the primer effectively demineralized the dentin surface and facilitated the diffusion of resin into exposed collagen fibrils. Both Cention Forte and Predicta Bioactive demonstrated the ion releasing property, which supported the formation of a hybrid layer and improved bond strength after storage in artificial saliva. this finding supported by (SEM) analysis that show mixed failure in storage group of Predicta Bioactive.

The combined Scanning Electron Microscopy (SEM) and Energy-Dispersive X-ray (EDX) analytical technique was utilized to assess both the morphology and mineral content of dentin surfaces. This technique allowed for qualitative evaluation of surface morphology and quantitative assessment of mineral changes resulting from experimental interventions. The preservation of the crystalline structure and the measured Ca/P ratio were used to determine the extent of remineralization, reflecting the effectiveness of the applied treatments. SEM, with magnification ranging from 50X to over 10,000X, is a valuable tool in dental research, especially when paired with EDX, which detects elemental composition and can indicate the presence of hydroxyapatite through calcium phosphate analysis [57,58].

The definition of "bioactive" will vary based on the application. In restorative dentistry, the capacity of materials to release ions like as calcium, phosphorus, and fluoride, hence facilitating remineralisation and the formation of hydroxyapatite crystals upon contact with physiological fluids, is referred to as bioactivity [59,60]. An increase in the Ca/P ratio is a critical indicator of remineralisation, which allows for the assessment of a material's suitability for use on demineralised dental tissue. The typical Ca/P ratio in natural dentition is 1.67, when the Ca/P ratio is less than 1.67, it can be classified as a non-stoichiometric crystal that is deficient in calcium [61,62].

The study found that under controlled storage conditions, SEM/EDX revealed differences in both the restorative materials surface and the area of the coronal dentin-restoration interface on tooth structure in Cention Forte and Predicta Bioactive indicated their bioactivity by increase the Ca/P ratio of restorative materials surface for Predicta Bioactive and Cention Forte (1.21±0.20 to 1.84±0.71, 1.31±0.12to2.63±0.63, respectively) and also for coronal dentin-restoration interface area (1.75±0.35 to 2.71±0.34, 1.97±0.38 to 4.01±1.98, respectively) after storage in artificial saliva at pH =7 in 37 C temperature for 30 days. this finding agreement with (Di Lauro *et al.*, 2023) [63], in which this study evaluated the effect of pH and temperature on the ion release of a resin-based material containing alkaline fillers and a self-setting high-viscous glass ionomer cement the highest amount detected at pH = 6.8 was at 37 °C after 28 days which is comparable to the condition of our study. In neutral and alkaline solutions, Ca and PO_4 ions can be precipitated as apatite [64,65].

In this study SEM images supported these findings which showed apatite-like crystal in both the restorative materials surface and the area of the coronal dentin-restoration interface on tooth structure in Cention Forte and Predicta Bioactive.

The Results of the present study showed no significant different in Ca/P ratio in both the restorative materials surface and the area of the coronal dentin-restoration interface on tooth structure in Cention Forte and Predicta Bioactive in non-storage and storage groups.

In non-storage groups of this study Cention Forte and Predicta Bioactive showed no significant different in Ca/P ratio and less than storage groups .For Cention Forte this can be due to the fillers in which three inorganic glasses: barium alumino-silicate glass, calcium barium alumino-fluoro-silicate and a basic calcium fluoro-silicate glass referred to as an “Alkasite”fille [66] that lead to the formation of a superficial layer of calcium fluoride and calcium phosphate with 0.5 mm thickness on the surface of Cention Forte at the initiation of setting reactions, which resists dissolution with deionized water for some time[67] . Previous work [68] showed that the phosphate ion release from Cention N bioactive materials significantly increased as the storage time increased 24 h to 4 h, and 6 months in distilled water.

Cention Forte primer and Predicta Bioactive contain HEMA, which is hydrophilic monomer, high hydrophilicity promotes increased water acceptance that results in the hydrolytic degradation [69], with increased their solubility, that explain their ability to release more ions and enhance their bioactivity. In this study, the storage media was artificial saliva with pH=7 at 37°C temperature for 30 days that is preferable environment for Cention®Forte to release Ca, F, and P ions, which results in the formation of apatite on its surface, like previous results [7,70].

In the current study, even there is no significant different in Ca/P ratio in both Cention Forte and Predicta Bioactive in non-storage and storage groups. However, in storage groups, the Ca/P ratio of Cention Forte is higher in both the restorative materials surface and the area of the coronal dentin-restoration interface on tooth structure (2.63±0.63), (4.01±1.98) respectively than Predicta Bioactive (1.84±0.71, 2.71±0.34, respectively). This can be attributed to the fact that it contains alkaline fillers with a strong affinity for water [71] .When alkaline fillers come in contact with the saliva three salts are connected (Na_2O, CaO, CaF_2) in SiO_2 are dissolved and released Ca, F and OH ions depend on the pH, forming apatite in vitro on dentine at pH 7 if phosphate available Combine with a specific primer [72].

Clinical trials are essential to assess the durability of these bioactive restorative materials, their performance in patients, and the mechanisms by which these hybrid materials release ions and interact with decayed dentin. Cention Forte appears suitable for durable restorations, however Predicta Bioactive may still be advantageous in cases where bioactivity and remineralisation properties are required.

Conclusion

This study showed that employing ion-releasing restorative material, specifically alkasite-based primers before applying it, is highly suggested because this method seems to be the best way to get a stronger binding with dentin. As a result, this study's finding that applying primer can improve bonding to dentin goes against the manufacturer's initial advice and classification of this type of restoration as self-adhesive. Compared to Predicta Bioactive, Cention Forte had a stronger connection and was better at generating apatite. Higher Ca/P ratios and SEM images suggest that storing in artificial saliva made mineral deposition at the contact better. These results support the idea that Cention Forte can interact with living things, especially when it is moist and old.

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Table 1. Shear bond strength values in all groups of the study in Mpa.

Restorative materials	Saliva Artificial	N	Minimum	Maximum	Mean	Std. Deviation
Predicta Bioactive	Non-stored	10	5.10	8.52	6.38	1.15
	Stored	10	4.57	8.99	7.18	1.31
Cention Forte	Non-stored	10	7.05	10.31	8.38	1.12
	Stored	10	7.22	13.42	10.13	2.11

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Figure 1. The mode of failure: (a) adhesive failure, (b) mixed failure, and (c) cohesive failure.
Then, Scanning Electron Microscopy (SEM) at 1300X was used in three samples from each subgroup to determine the mode of failure of each de–bonded coronal dentin samples (Figures 2 and 3).

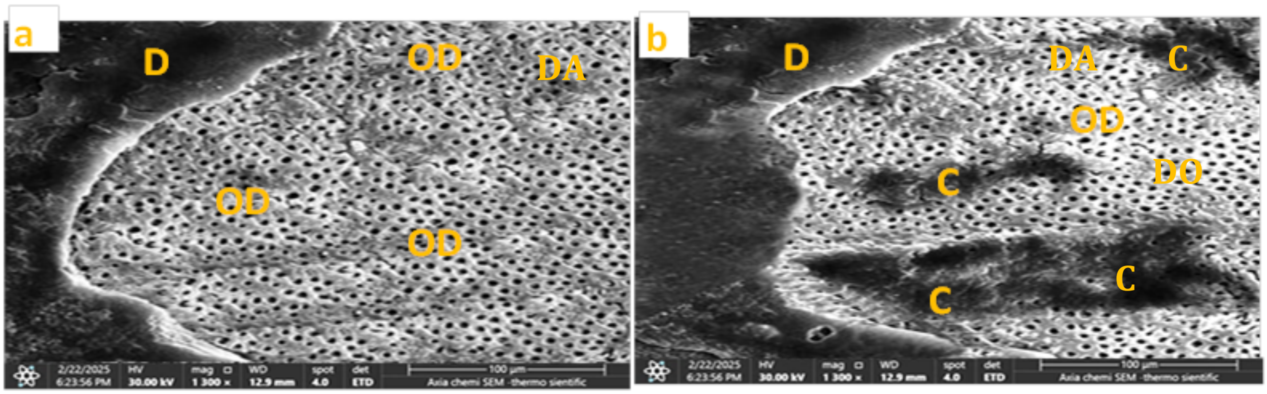

Figure 2. SEM images at 1300x magnification for Predicta Bioactive representing the mode of failure of de-bonded coronal dentin samples; adhesive failure of a sample not stored in artificial saliva (a); mixed failure of a sample stored in artificial saliva (b). Notes: OD = open dentinal tubules, C = composite material, D = dentin, DA = de-bonded area.

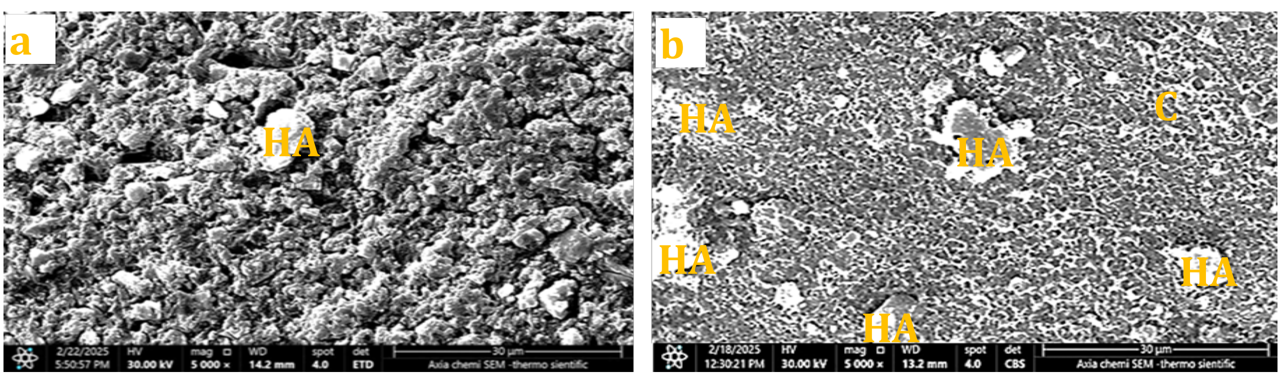

Figure 3. SEM images at 1300x magnification for Cention Forte representing the mode of failure of de-bonded coronal dentin sample; mixed failure of a sample not stored in artificial saliva (a); cohesive failure of a sample stored in artificial saliva (b). Notes: CD = closed dentinal tubules, OD = open dentinal tubule, D = dentin, DA = de-bonded area.

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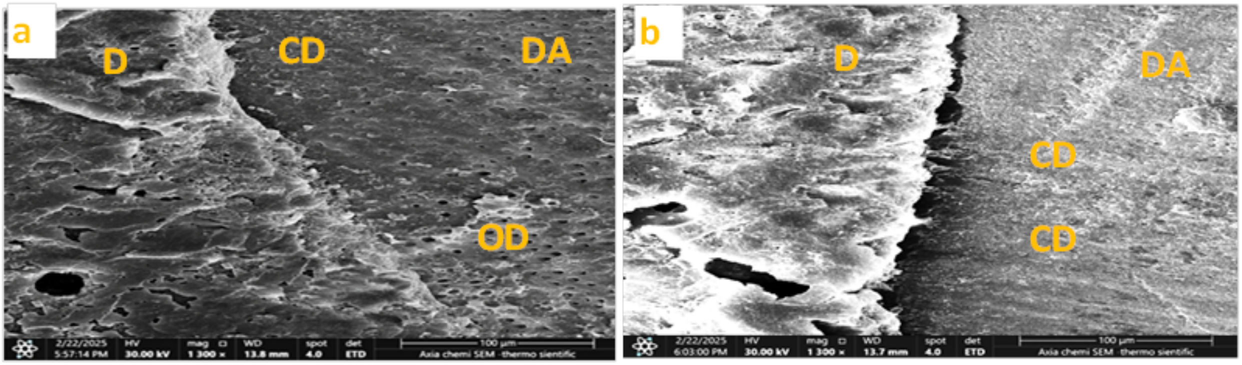

Figure 4. The SEM image at 5000X magnification for:(a); Predicta Bioactive composite not stored in artificial saliva (A1) showed slightly precipitation of hydroxyapatite (b); after storage in artificial saliva (A2) showed hydroxyapatite deposition (HA) at the de bonded area of composite (C).

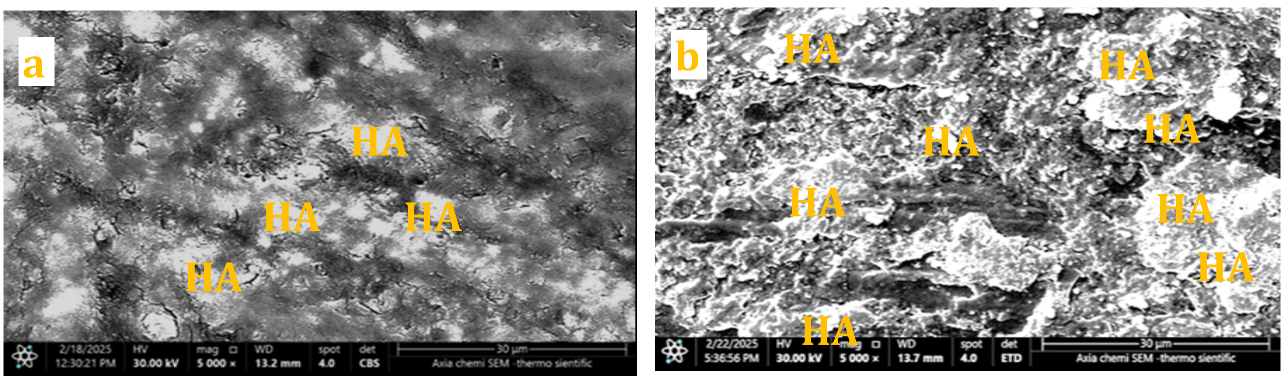

Figure 5. The SEM image at 5000X magnification for (a) Cention Forte composite not stored in artificial saliva and (b) after storing in artificial saliva showed hydroxyapatite (HA) deposition at the de-bonded area of the composite (C).

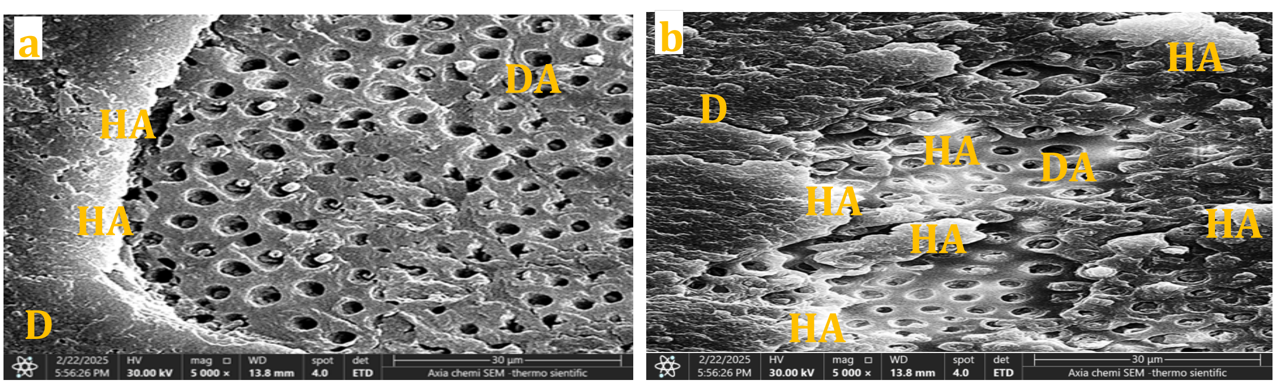

Figure 6. SEM image at 5000X magnification for de-bonded areas of coronal dentin samples: (a) Predicta Bioactive not stored in artificial saliva and (b) after storage in artificial saliva. Both images showed crystal-like deposition of hydroxyapatite (HA) at the de-bonded coronal dentin area. D = dentin, DA = de-bonded area.

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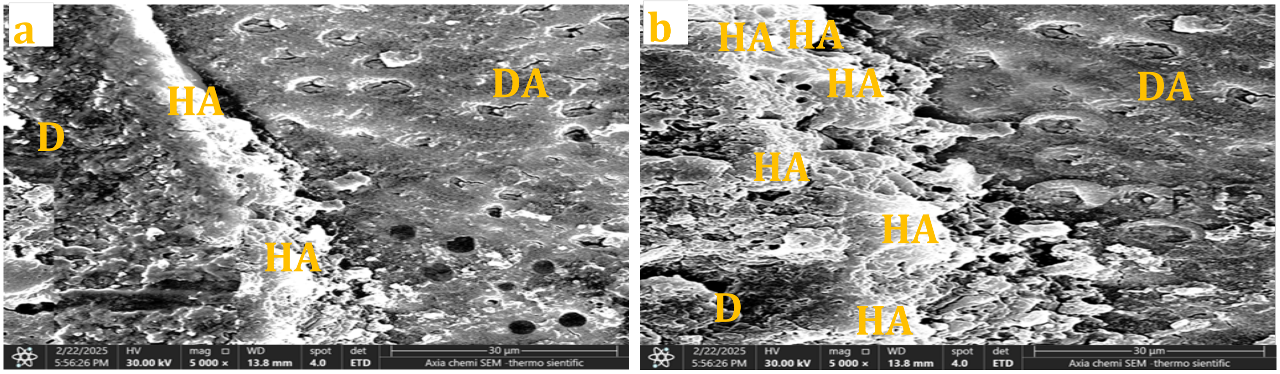

Figure 7. SEM images at 5000X magnification for de-bonded areas of coronal dentin samples: (a) Cention Forte not stored in artificial saliva and (b) after storage in artificial saliva. Both images showed crystal-like hydroxyapatite (HA) depositions at the de-bonded coronal dentin areas. D = dentin, DA = de-bonded area.

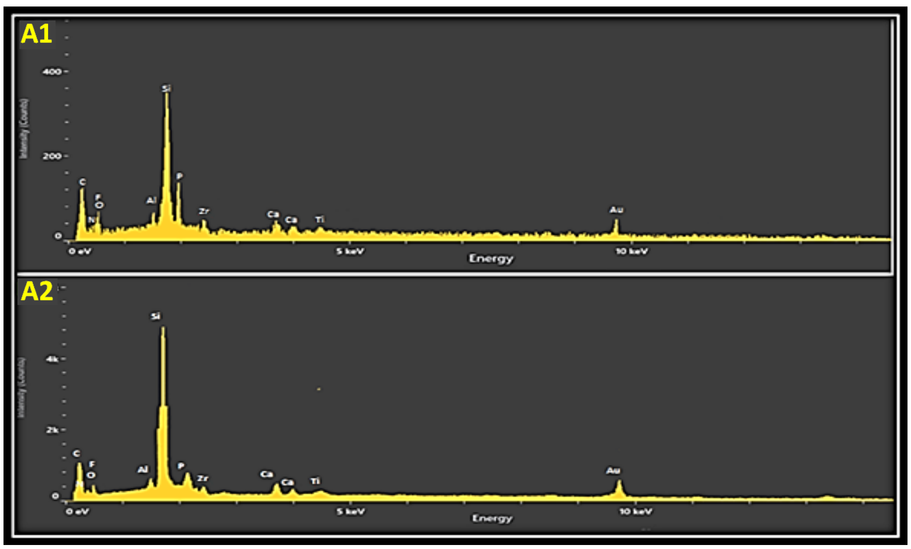

Figure 8. The EDX spectra for one core sample of Predicta Bioactive not stored in artificial saliva (A1) and after storage for 30 days in artificial saliva (A2) at pH=7 showing the identification of the phosphorus peak after storage.

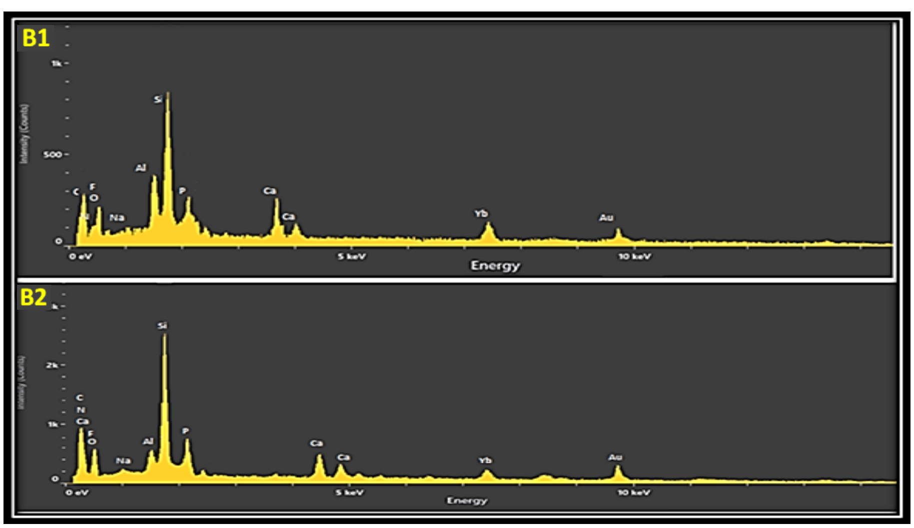

Figure 9. The EDX spectra for one core sample of Cention Forte not stored in artificial saliva (B1) and after storage for 30 days in artificial saliva at pH=7 (B2) showing the phosphorus peak identification after storage.

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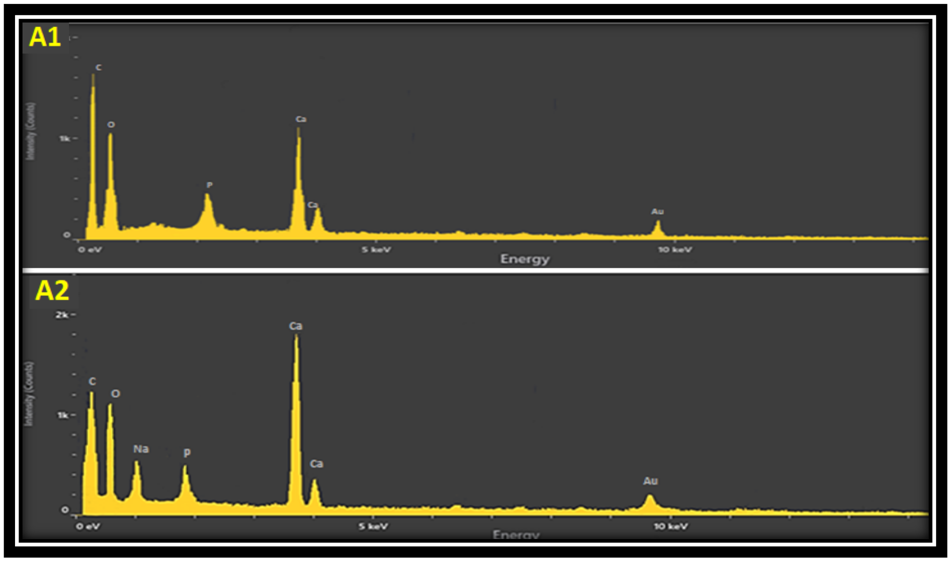

Figure 10. The EDX spectra of one tooth sample (de-bonded coronal dentin) for Predicta Bioactive not stored in artificial saliva (A1) and after storage for 30 days in artificial saliva (A2) showing a change in the spectra for both phosphorous (P) and calcium (Ca).

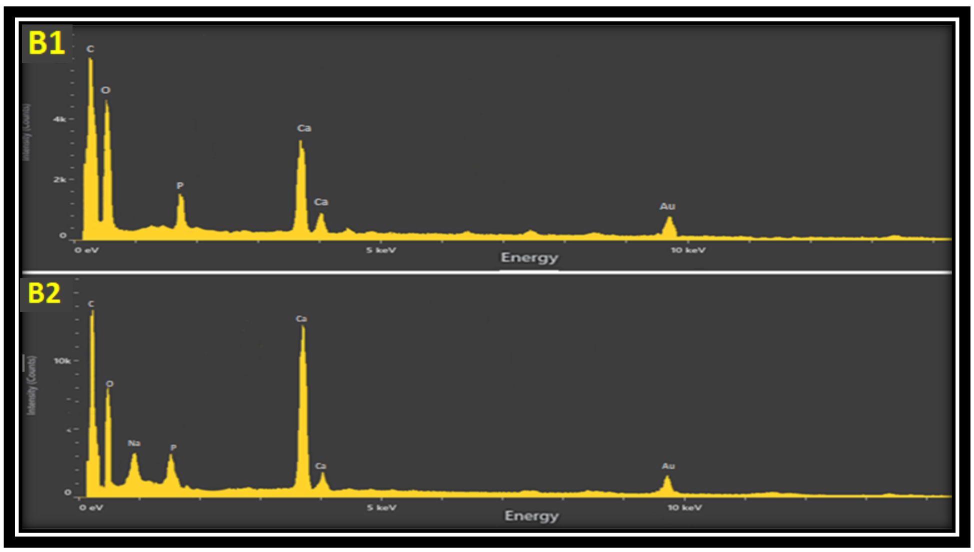

Figure 11. The EDX spectra of one tooth sample (de-bonded coronal dentin area) for Cention Forte not stored in artificial saliva (B1) and after storage for 30 days in artificial saliva (B2) showing a change in the spectra for both phosphorous (P) and calcium (Ca).