

MICROANALYSIS FOR IONS RELEASE OF RESTORATIVE MATERIALS USED AS POSTERIOR RESTORATIONS

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ABSTRACT

Aim: Recently, new hybrid bioactive restorative materials with the dual advantages of avoiding cavities and encouraging remineralisation have entered the market. Important determinants of the bioactive material's antibacterial qualities are the kind of ions produced and how quickly they are released. The study's objective was to quantify the quantity of ions produced by bioactive materials used in posterior restorations in order to lower the risk of recurrent caries.

Materials and methods: For restorative materials utilised in the study, 45 cylindrical specimens were made (nine samples, each group G, measuring 4 mm in thickness and 6 mm in diameter). Giomer (G I), Alkaside Cention (G II), Ketac Molar (G III); Ketac Silver (G IV); and Zirconomer (G V). Following sample preparation as directed by the manufacturer, the specimens were immersed in demineralising and remineralising media (artificial saliva and Coca-Cola) for pH cycling. At 24h, 7, and 28 days (T1, T2, and T3), the number of ions released from each material was measured using energy dispersive X-ray (EDX) analysis.

Results: Comparison between groups was performed by using One Way ANOVA test followed by Tukey's Post Hoc test for multiple comparisons, while comparison between different intervals was performed by using Repeated Measures ANOV followed by Tukey's Post Hoc test for multiple comparisons. The significant level was set at $P \leq 0.05$.

There was a significant difference between all groups regarding all elements and at all times of investigation $P \leq 0.05$.

Conclusions: The ions release from bioactive restorative materials is time and composition dependent. With similar releases of cariostatic ions, all studied restorative materials can be regarded as promising bioactive materials employed as posterior restorations.

KEY WORDS: Ions release, bioactive materials, posterior restorations, Giomer, Cention forte, Ketac molar, Ketac silver, Zirconomer.

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INTRODUCTION

Dental caries is a complicated infectious condition characterised by the slow disintegration of hard tooth components and brought on by microbes. The main pathogen is *Streptococcus mutans*. Caries is caused by a complex interaction between fermenting food carbohydrates and cariogenic oral bacteria, or biofilm. However, a person's susceptibility to caries will depend on a number of factors, including their immune system, genetic susceptibility, diet, tooth structure, pH of biofilm (or dental plaque), oral hygiene, and socioeconomic status. Lactic acid is a byproduct of the biofilm's bacteria's energy-producing conversion of carbohydrates into sugars. By removing calcium and phosphate from the dental structures, this begins the demineralisation process, which eventually leads to the collapse and breakdown of the tooth structures, resulting in a cavity. It also gradually lowers the pH of plaque to dangerously low levels (below 5.5).

Remineralisation takes place when equilibrium is reached and these minerals are then reconstituted into the enamel. A carious cavity is created when demineralisation exceeds remineralisation, a process that could take months or years. Saliva is essential for the demineralisation process because it buffers plaque acid, eliminates substrates, and supplies calcium and phosphate. Remineralisation occurs after enough time has elapsed since an acid attack, rendering the "caries inactive."¹

Even now, the most common reason for restoration failure and replacement is recurrent caries. Although recurrent caries is produced by the local acidic development of bacterial biofilms at tooth structural sites, the exact mechanism by which marginal and/or wall recurrent caries start is not fully known².

Research is conducted on the creation of restorative materials that have the ability to suppress bacterial biofilms. In addition to preventing or reducing the risk of caries recurrence,

the goal is to obstruct biofilm acidity and preserve biocompatibility, non-toxicity, and acceptable strength and aesthetic qualities. To ensure long-term efficacy and therapeutic applicability without negatively impacting the material's mechanical and aesthetic qualities, further advancements are still required².

It has been noted that new hybrid restorative materials are being developed that combine the benefits of glass ionomer cements (GICs) (self-adhesive qualities and ion release) and resin composites (mechanical strength, aesthetics, and high bond strength). In addition to the resinous self-adhesive form of conventional GICs known as resin-modified glass ionomer cements (RM-GICs), this almost resulted in the invention of ion-releasing, non-adhesive compomers and giomers. Surefil One (Dentsply-Sirona, Konstanz, Germany), Cention N (Ivoclar-Vivadent, AG, Schaan, Liechtenstein), and Activa BioActive Restorative (Pulpdent Corporation, Watertown, MA, USA) are three new hybrid composites that have recently entered the market. Fluoride ions are said to be released by all three. These substances are called bioactive due to their ionic release³.

"Giomers," a class of bioactive materials with novel technology, was unveiled. The surface pre-reacted glass ionomer (S-PRG) particle, which is created when fluoroboroaluminosilicate glass is pre-reacted with polyacid to create a glass-ionomer matrix structure, serves as the foundation for their composition. This matrix is then blended with the resin matrix. This particle shields the structure's core from the damaging effects of moisture while allowing the release of fluoride and other ions through its surface layer. Additionally, lab tests show that this technology inhibits cariogenic bacteria, stops demineralisation, and facilitates remineralisation. Giomers are hence materials that have the S-PRG particle in their composition rather than glass ionomers³.

The alkasite material represented by Cention Forte and Cention N (Ivoclar-Vivadent, AG, Schaan, Liechtenstein) has an alkaline filler that can release ions that neutralise acids and aid in preventing demineralisation. Additionally, significant amounts of calcium ions and fluoride are released, allowing for the remineralisation of enamel³.

The following are some adjustments made to standard glass ionomer to make it suitable for use as posterior restorations with sufficient mechanical properties: Zirconomer, Ketac Silver, and Ketac Molar.

Ketac Molar was found to have a statistically significant protective effect against caries when compared to resin composite, and it was also able to suppress bacterial development and demineralise enamel in trials involving fake caries^{4,5,6}.

Regarding Ketac Silver, earlier research showed that sintered silver GICs had potent antibiofilm properties⁷.

New to the glass-ionomer family is zirconia-infused GIC (zirconomer). Zirconia (ZrO_2) is a ceramic material with excellent mechanical properties⁸, and better biocompatibility in contrast to standard glass ionomers⁹.

Restorative dentistry materials are increasingly being referred to as “bioactive” in scientific literature and ads. Bioactive materials are substances that have the capacity to interact with biological systems, act as biological agents, and establish a connection with tissues^{10,11}.

Restorative materials with bioactive nanoparticle-based platforms and calcium phosphate (CaP) interactions are discovered to be promising for slowing mineral loss, reducing caries-related biofilms, and increasing remineralisation. A hybrid of nanotechnology and remineralisation potentials is utilised. Ion release from CaP-containing composites is dependent on several critical parameters, such as pH level and CaP particle size, with nanosized

particles, the volume fraction of CaP, and the total content causing a larger release of ions.

The industry offers a variety of fluoride-containing hybrid restorative materials, such as bioactive resin composites, Compomers, Gionomers, and modified glass ionomers. In order to preserve the clinical benefits of standard glass ionomers and composite resins while resolving a problem with them, hybrid materials were developed¹².

In addition to their capacity for fluoride release and recharge, glass ionomers exhibit a strong chemical link with the tooth's structure and exhibit high biocompatibility. Reinforced glass ionomers were developed as a result of efforts to overcome their brittle character, early moisture sensitivity, and slower strength development¹³.

The conservative dentistry movement has given rise to novel restorations and bioactive materials that are said to have the dual benefits of preventing cavities and promoting remineralisation while also protecting tooth tissues. In fact, the goal of the modern therapeutic gradient is to preserve dental tissues to the greatest extent feasible, so prolonging their longevity and enabling the possibility of re-intervention. The most aesthetically pleasing materials for direct procedures utilised in restorative dentistry today are composite resins. However, research has revealed that some of these materials promoted the growth of bacteria and the development of plaque, which led to the recurring caries occurrence and the final failure of the restoration.

Within this framework, a set of novel materials was created that had the ability to release ions and remineralise; these materials have been proposed as possible substitutes for traditional restorative materials. The advantages of pH increase and ion release qualities on remineralisation and tooth caries prevention have been examined in a number of research. It was expected that they would prevent the demineralisation of dentin and enamel and lessen the chance of subsequent caries¹⁴.

About the fact that caries is more common in older people's approximal surfaces of posterior teeth than on occlusal surfaces, namely in the occlusal surfaces¹⁵. The most morphologically vulnerable tooth types in the oral cavity to dental caries are molars¹⁶.

The type of ions released and their rate of release are important factors for the bioactive material's antibacterial properties. In this regard, it was interesting to investigate the restorative materials that are designated to be used as posterior restorations (due to their documented high strength properties, durability, and high performance in areas with heavy mastication forces). To evaluate the

extended bioactive effect of these materials inside the oral cavity to reduce the likelihood of recurrent caries, it is equally crucial to analyse the discharge pattern and the rate of fluoride release beyond the initial burst.

Therefore, the purpose of this study was to measure the amount of ions released from bioactive restorative materials utilised in posterior restorations. The current study's null hypothesis was that, over the course of the investigation, there would be no differences in the ions released from Giomer (Beautiful II) with Surface pre-reacted glass particles when compared to other types of examined materials.

MATERIALS

The materials used in this study, The composition, manufacturers, concentration, and pH were listed in Tables (1) and (2):

Material	Specification	Composition	Manufacturer
Surface pre-reacted glass (Beautiful II) restoration Shade (A3)	Nano hybrid radiopaque bioactive composite	Bis-GMA, UDMA, BisMPEPP, TEGDMA. 83.3 wt% Fluorosilicate glass	SHOFU Dental GmbH, Japan. www.shofu.com
Alkasite Cention forte self cured Shade (A2)	Radio opaque bioactive self-cured bulkfill-RBC, with a light-curing option.	Matrix : UDMA- DCP, Aromatic aliphatic-UDMA (Tetramethyl-xyllylen-diurethane dimethacrylate), PEG-400 DMA Fillers: Ca-fluorosilicate glass, Ba-Al silicate glass, copolymer, Ca-Ba-Al fluorosilicate glass, (alkaline) glass filler, ytterbium. Filler loading 75% wt%_ 61 vol% particle size range (0.1–35 µm)	Ivoclar Vivadent Inc., NY, USA www.Ivoclar.com
Ketac molar	Glass Ionomer Cement	Liquid: Polyalkenoic acid, tartaric acid and water Powder: Fluorosilicate glass Al-Ca-La copolymer, 5% acrylic acid and maleic acid	3M ESPE Dental Products, St.Paul. U.S.A
Ketac silver	Silver Reinforced Glass Ionomer Cement	Liquid:Polyalkenoic acid 30 - 50 % by Wt, tartaric acid 5 - 15 % by Wt and water 40 - 60 % by Wt Powder: Fluorosilicate glass Al-Ca-La copolymer 40 - 50 % by Wt, Silver 45 - 55 % by wt Titanium dioxide 1 - 5 % by Wt	3M ESPE Dental Products, St.Paul. U.S.A
Zircono mer® Improved	Nano-sized zirconia Reinforced Glass Ionomer Cement known as white amalgam	Powder: fuoroaluminosilicate glass, zirconium oxide Liquid: polyacrylic acid solution and tartaric acid	Shofu Inc. Kyoto, Japan

Storage media	Composition	Concentration	pH
Artificial saliva	Na ₃ PO ₄	3.90 mM	7.2
	NaCl ₂	4.29 mM	
	KCl	17.98 mM	
	CaCl ₂	1.10 mM	
	MgCl ₂	0.08 mM	
	H ₂ SO ₄	0.50 mM	
	NaHCO ₃	3.27 mM	
	Distilled water	2.87 mM	
	Phosphate (Pi)	5.43mM	
Calcium (Ca ²⁺)	0.84 mM		
Coca-Cola	Titratable acid	40.0 mmol/L OH to pH 5.5 and 83.6 mmol/L OH)	2.7
	Phosphoric acid	11 g	
	Carbonated water	2.400 g	
	Sugars	10.7 g	
	Sodium	0.01g	
	Caffeine	3.1 g	
	Decocainized coca leaf	1.1 g	

METHODS

This study was carried out to evaluate microanalysis for ions release of S-PRG restorative material versus four bioactive restorative materials

Ethical approval:

The protocol of this study was approved by the Council of Conservative

Dentistry Department – Faculty of Dentistry – October 6 University and the ethical issues were reviewed and revised by the Research Ethics Committee – Faculty of Dentistry – October 6 University on December 2021 (Approval No.RECO6U/19-2021 at meeting 1st December 2021.

Sample size calculation:

Sample size calculated depending on a previous study¹⁷, the minimally accepted sample size was 9 per group, when mean \pm standard deviation of Ca Ion Release in group I was 30.06 ± 0.98 while estimated mean difference with Group II was 1.5, when the power was 85 % & type I error probability was 0.05. The Independent t test was performed by using P.S.Power 3.1.6.

Micro analysis for ions release

1) Grouping of the specimens

According to the material utilised, 45 cylindrical-shaped specimens representing each restorative material employed in the investigation were created (nine samples per group G). The investigated materials were: Giomer; (Beautiful II) (Shofu Inc. Kyoto, Japan) (GI), Alkasite Cention forte (Ivoclar Vivadent Inc., NY, USA) (GII), Ketac Molar (Glass ionomer) (3M ESPE, St. Paul, MN, USA) (GIII), Ketac Silver (Glass ionomer) (3M ESPE, St. Paul, MN, USA) (G IV), and Zirconomer (Shofu Inc. Kyoto, Japan) (G V). Energy Dispersive X-ray (EDX) analysis was used to measure the amount of ions released from each material at 24, 7, and 28 days (T1, T2 and T3) respectively.

To ensure that every tested item had the same dimensions, split Teflon moulds were made. The mold's measurements were 4 mm thick and 6 mm in diameter. For each material under test, a single mould was employed. A glass slide and a Mylar strip were positioned on top of each Teflon ring (Polydentia, Switzerland)¹⁸.

For the Giomer (GI); (Beautiful II) discs, a single mould was utilised. They were completed by using a composite applicator (a German stainless steel dental composite nonstick filling tool) to pack a sequence of wedge-shaped oblique increments. The giomer was progressively packed so that each increment's average thickness ranged from 0.1 to 2 mm. Using a light cure unit (Guilin Woodpecker Medical Instrument Co., Ltd., 541004 P.R. China), each increment of the inserted material was photo-

polymerized for 20 seconds at zero distance from the glass slide at a wavelength of 385nm ~ 515nm and an output light intensity of 1000 mW/cm²-1200mW/cm².

A second glass slide was placed with gentle pressure on the disc to extrude the excess material and cause photo-polymerization after the final increment of material was packed into the mould and a second Mylar strip was used to cover the top surface of the mould. After that, the cylindrical specimens were carefully taken out of the moulds and their diameter and thickness were measured again with a digital calliper (Bacolis, Egypt).

To prepare Alkaside Cention forte (Ivoclar Vivadent Inc., NY, USA) (GII) discs, another mould was used. First, each capsule was shaken and put into an amalgamator (Linea Tac Mixer, ex Kent Express, UK) and mixed at high speed (± 4000 rpm) for 10 seconds. Next, the capsule was forcefully applied to the mould using a special applicator (gun for glass ionomer capsules, manufactured in China, brand: Generic). To ensure the absence of air bubbles, the capsule was applied to the mould using a special applicator (glass ionomer capsule gun), which was used to ensure there were no air bubbles. The samples were then polymerised in the self-curing mode, which was left for 4-5 minutes as directed by the manufacturer. The same procedure was repeated with Ketac Molar (Glass ionomer) (3M ESPE, St. Paul, MN, USA) (GIII) and Ketac silver (Glass ionomer) (3M ESPE)(G IV)

For Zircomer (Shofu Inc., Japan, Kyoto) (G V). A different mould was used to create specimens. The specimens were prepared with a glass slap that was wrapped in a Mylar strip. The glass ionomer liquid and powder were combined until a uniform mixture was achieved, following the manufacturer's instructions for proportioning. A condenser of an appropriate size was used to pack the materials into the mould in one step. After that, a glass slide and another Mylar strip were placed on top, and the mould was squeezed for a short while to remove any

remaining material and create a smooth, uniform surface. The glass ionomer mixture was kept in the mould until it solidified completely.

Every group's specimens underwent pH cycling, which involved immersing them in demineralising and remineralising medium (artificial saliva such as Coca-Cola). Every sample was submerged for six hours at 37 OC in Coca-Cola with a pH of 2, 6. After that, they were rinsed with distilled water, dried using absorbent paper, and submerged for eighteen hours in fake saliva. Each sample was stored in 10 ml of the above specified solutions individually in a graduated plastic container until the two immersion periods, 7 and 28 days. Every day, the solutions were modified¹⁹.

Microanalysis for ions release was measured after at 24 hours (T1), 7 days (T2), 28 days (T3), respectively, using Energy Dispersive X-ray (EDX), (AMETEK, Made in USA), at National Research Center, EDX works by striking an atom's inner shell with an electron beam, which removes an electron from the shell and leaves behind a positively charged electron-hole. Another electron from an outer shell is drawn to the displaced electron in order to fill the vacuum. An X-ray can be produced when an electron transitions from the atom's outer, higher-energy shell to its inner, lower-energy shell. The energy of this X-ray is unique to the specific element²⁰. A representative figure of Energy dispersive X-ray spectrum of Giomer (GI) (Beautiful II). fig 1

Statistical analysis:

Statistical analysis was performed with SPSS 20®, Graph Pad Prism® and Microsoft Excel 2016. All data were explored for normality by using Shapiro Wilk and Kolmogorov Normality test which revealed that all data originated from normal distribution, accordingly comparison between groups was performed by using One Way ANOVA test followed by Tukey's Post Hoc test for multiple comparisons, while comparison between different intervals was performed by using Repeated Measures ANOV followed by Tukey's Post Hoc test for multiple comparisons. The significant level was set at $P \leq 0.05$.

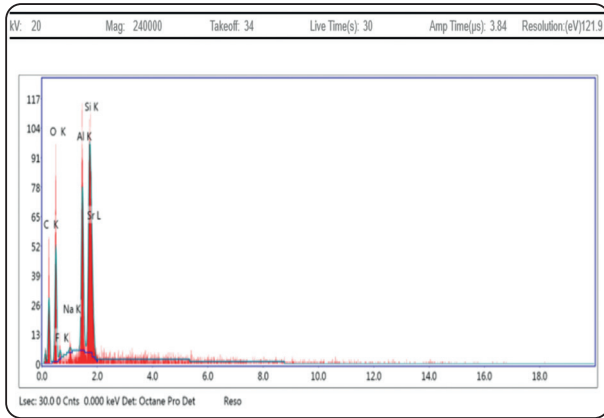


Fig. (1) Energy dispersive X-ray spectrum of Giomer (GI); (Beautiful II) at T1

- ¹ Statistical Package for Social Science, IBM, USA.
- ² Graph Pad Technologies, USA
- ³ Microsoft Co-operation, USA.

RESULTS

Intergroup comparison (Comparison between groups)

Comparison between groups at T1:

There was a significant difference between groups regarding all elements at T1 as $P < 0.0001$, the highest concentration was in GI regarding Al^{3+} , SiO_3^{2-} , Sr^{2+} , and BO_3^{3-} . The highest concentration was in GII regarding C, N, Y and Ba, while it revealed the least concentration regarding O, F-, and Na+. The highest concentration in GIII was regarding P, and K, while the least concentration regarding Ca. In Group IV, the highest concentration was in O, F-, Ca, Ag, Ti, and B, while the least concentration was demonstrated in C, Al^{3+} , SiO_3^{2-} , Sr^{2+} , P, and K. In Group V, the highest concentration was demonstrated in Na+, and Zr. As presented in table 3 and figure 2.

TABLE (3) Comparison between all groups regarding different elements at T1:

T1	GI		GII		GIII		GIV		GV		P value
	M	SD	M	SD	M	SD	M	SD	M	SD	
C	31.60 a	6.47	38.69 b	4.18	27.26 ac	0.55	17.81 d	0.76	25.18 c	0.98	0.0001*
O	32.1 a	1.59	30.69 a	5.49	42.16 b	0.71	47.74 b	3.72	44.60 b	0.80	0.0001*
F-	4.27 a	1.25	3.07 b	1.02	3.92 a	0.32	4.98 a	0.33	3.99 a	0.30	0.0002*
Na+	1.85 a	0.46	0.90 b	0.47	1.51 a	0.26	1.17 a	0.99	3.39 c	0.21	0.0001*
Al^{3+}	9.13 a	0.94	3.99 b	0.90	7.20 c	0.34	5.91 d	0.48	8.02 c	0.26	0.0001*
SiO_3^{2-}	9.52 a	0.78	8.57 a	1.49	7.43 b	0.44	5.46 c	0.28	9.06 a	0.55	0.0001*
Sr^{2+}	9.77 a	0.58	0.0 b	0.00	4.48 c	0.40	0.05 b	0.04	0.00 b	0.00	0.0001*
Ca	0.0 a	0.00	3.85 b	0.99	2.37 c	0.57	11.43 d	0.60	3.98 b	0.16	0.0001*
P	0.0 a	0.00	0.0 a	0.00	2.24 b	0.08	0.65 c	0.57	0.87 c	0.10	0.0001*
K	0.0 a	0.00	0.0 a	0.00	1.11 b	0.18	0.13 c	0.11	0.0 a	0.00	0.0001*
Ag	0.0 a	0.00	0.0 a	0.00	0.0 a	0.00	1.94 b	1.08	0.0 a	0.00	0.0001*
Ti	0.0 a	0.00	0.0 a	0.00	0.0 a	0.00	2.74 b	0.83	0.0 a	0.00	0.0001*
BO_3^{3-}	7.55a	0.88	0.0 b	0.00	0.0 a	0.00	0.0 a	0.00	0.0 a	0.00	0.0001*
N	0.0 a	0.00	3.71 b	1.67	0.0 a	0.00	0.0 a	0.00	0.0 a	0.00	0.0001*
Y	0.0 a	0.00	0.30	0.08	0.0 a	0.00	0.0 a	0.00	0.0 a	0.00	0.0001*
Ba	0.0 a	0.00	5.84 b	0.85	0.0 a	0.00	0.0 a	0.00	0.0 a	0.00	0.0001*
B	0.0 a	0.00	0.0 a	0.00	0.70 b	0.44	0.0 a	0.00	0.0 a	0.00	0.0001*
Zr	0.0 a	0.00	0.0 a	0.00	0.0 a	0.00	0.0 a	0.00	0.91 b	0.12	0.0001*

*Significant difference as $P < 0.05$.

Means with different superscript letters per raw were significantly different as $P < 0.05$.

Means with the same superscript letters per raw were insignificantly different as $P > 0.05$.

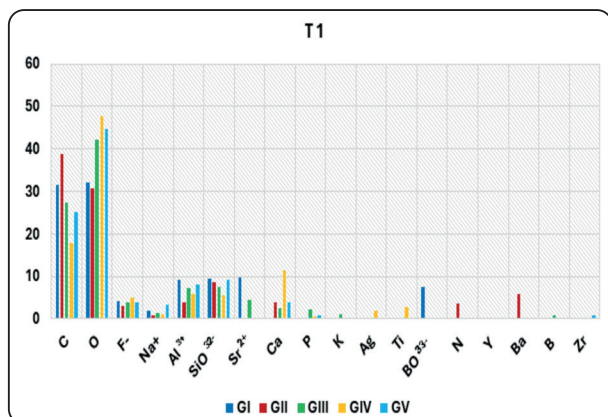


Fig. (2) Bar chart representing all elements in T1 regarding all groups.

Comparison between groups at T2:

There was a significant difference between groups regarding all elements at T2 as $P < 0.05$, the highest concentration was in GI regarding C, Sr^{2+} , and BO_3^{3-} , while the least concentration was in Na^+ . The highest concentration was in GII regarding SiO_3^{2-} , N, Y, and Ba, while it revealed the least concentration regarding O, F^- , and Al^{3+} . The highest concentration in GIII was regarding O, and P, while the least concentration was regarding C, SiO_3^{2-} , Ca, and K. In Group IV, the highest concentration was in O, K, and Ag, while the least concentration was demonstrated in Sr^{2+} . In Group V, the highest concentration was demonstrated in F^- , Na^+ , Al^{3+} , Ca, and Zr while the least concentration was in P. As presented in table 4 and figure 3.

TABLE (4) Comparison between all groups regarding different elements at T2:

T2	GI		GII		GIII		GIV		GV		P value
	M	Sd	M	SD	M	SD	M	SD	M	SD	
C	35.81 a	1.78	30.96 b	5.11	18.81 c	3.13	19.22 c	2.96	20.84 c	0.77	0.0001*
O	27.34 a	0.83	25.42 b	2.02	40.89 c	1.65	40.27 c	1.11	39.43 c	0.28	0.0001*
F^-	1.62 a	0.28	1.37 a	0.53	3.15 bc	0.69	2.60 b	0.21	3.36 c	0.33	0.0001*
Na^+	1.41 a	0.10	0.0 b	0.00	1.56 a	0.18	1.92 c	0.32	3.74 d	0.17	0.0001*
Al^{3+}	10.16 a	0.24	5.55 b	0.40	10.44 a	1.48	10.06 a	1.28	11.04 a	0.11	0.0001*
SiO_3^{2-}	8.35 a	0.17	12.75 b	2.50	7.32 a	1.00	8.69 a	1.00	12.58 b	0.48	0.0001*
Sr^{2+}	15.31 a	0.46	0.0 b	0.00	9.76 c	1.28	2.99 d	0.38	0.0 b	0.00	0.0001*
Ca	0.0 a	0.00	5.26 b	2.31	3.87 b	0.53	0.0 a	0.00	6.06 c	0.24	0.0001*
P	0.0 a	0.00	0.0 a	0.00	4.22 b	1.15	2.70 c	0.65	0.91 d	0.14	0.0001*
K	0.00 a	0.00	0.00 a	0.00	1.82 b	0.25	5.08 c	7.22	0.00 a	0.00	0.006*
Ag	0.00 a	0.00	0.00 a	0.00	0.00 a	0.00	5.72 b	1.02	0.00 a	0.00	0.0001*
BO_3^{3-}	4.70 a	0.84	0.00 b	0.00	0.00 b	0.00	0.00 b	0.00	0.00 b	0.00	0.0001*
N	0.00 a	0.00	9.03 b	2.10	0.00 a	0.00	0.00 a	0.00	0.00 a	0.00	0.0001*
Y	0.00 a	0.00	0.70 b	0.19	0.00 a	0.00	0.00 a	0.00	0.00 a	0.00	0.0001*
Ba	0.00 a	0.00	9.59 b	1.43	0.00 a	0.00	0.00 a	0.00	0.00 a	0.00	0.0001*
Zr	0.00 a	0.00	0.00 a	0.00	0.00 a	0.00	0.00 a	0.00	2.02 b	0.07	0.0001*

*Significant difference as $P < 0.05$.

Means with different superscript letters per row were significantly different as $P < 0.05$.

Means with the same superscript letters per row were insignificantly different as $P > 0.05$.

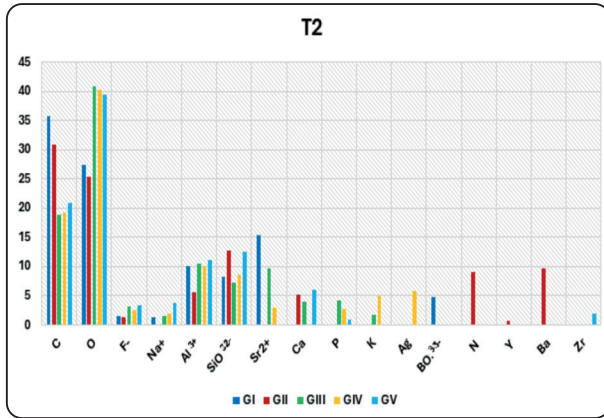


Fig. (3): Bar chart representing all elements in T2 regarding all groups.

Comparison between groups at T3:

There was a significant difference between groups regarding all elements at T3 as $P < 0.05$, the highest concentration was in GI regarding C, Sr^{2+} , and BO_3^{3-} . The highest concentration was in GII regarding N, Y, and Ba, while it revealed the least concentration regarding O, F-, and Al^{3+} . The highest concentration in GIII was regarding P, while the least concentration was regarding Na^+ , SiO_3^{2-} , Ca, and K. In Group IV, the highest concentration was in O, Al^{3+} , K, and Ag, while the least concentration was demonstrated in C and Sr^{2+} . In Group V, the highest concentration was demonstrated in F-, Na^+ , SiO_3^{2-} and Ca while the least concentration was in P. As presented in table 5 and figure 4.

TABLE (5) Comparison between all groups regarding different elements at T3:

T3	GI		GII		GIII		GIV		GV		P value
	M	SD	M	SD	M	SD	M	SD	M	SD	
C	35.49 a	1.32	33.48 a	2.01	18.96 b	2.02	16.11 b	6.44	19.05 b	1.33	0.0001*
O	28.50 a	0.91	25.26 b	1.67	39.58 c	0.71	40.21 c	0.94	39.12 c	0.33	0.0001*
F-	1.50 a	0.23	1.17 a	0.18	2.77 b	0.15	3.59 c	1.37	4.17 c	0.32	0.0001*
Na+	1.49 a	0.29	0.0 b	0.00	1.12 c	0.11	1.82 d	0.28	3.19 e	0.30	0.0001*
Al ³⁺	9.89 a	0.39	5.54 b	0.44	10.03 a	0.30	11.72 c	0.93	11.60 c	0.64	0.0001*
SiO ₃ ²⁻	8.14 a	0.24	11.34 b	0.85	7.41 a	0.55	9.57 c	1.06	12.39 d	0.28	0.0001*
Sr ²⁺	14.98 a	0.66	0.0 b	0.00	9.97 c	0.24	3.41 d	0.46	0.0 b	0.00	0.0001*
Ca	0.0 a	0.00	3.68 b	1.12	3.41 b	0.08	0.0 a	0.00	6.33 c	0.41	0.0001*
P	0.0 a	0.00	0.0 a	0.00	4.65 b	0.67	3.92 b	1.19	1.87 c	0.49	0.0001*
K	0.0 a	0.00	0.0 a	0.00	2.09 b	0.20	2.21 b	0.26	0.0 a	0.00	0.0001*
Ag	0.0 a	0.00	0.0 a	0.00	0.0 a	0.00	7.45 b	2.25	0.0 a	0.00	0.0001*
BO ₃ ³⁻	3.702 a	0.94	0.0 b	0.00	0.0 b	0.00	0.0 b	0.00	0.0 b	0.00	0.0001*
N	0.0 a	0.00	9.72 b	1.08	0.0 a	0.00	0.0 a	0.00	0.0 a	0.00	0.0001*
Y	0.0 a	0.00	0.83 b	0.27	0.0 a	0.00	0.0 a	0.00	0.0 a	0.00	0.0001*
Ba	0.0 a	0.00	8.97 b	0.95	0.0 a	0.00	0.0 a	0.00	0.0 a	0.00	0.0001*

*Significant difference as $P < 0.05$.

Means with different superscript letters per row were significantly different as $P < 0.05$.

Means with the same superscript letters per row were insignificantly different as $P > 0.05$.

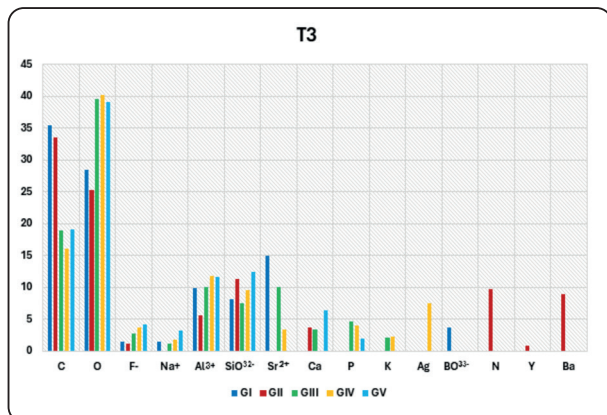


Fig. (4) Bar chart representing all elements in T3 regarding all groups.

Intragroup comparison:

Group I:

There was a significant change by time in all elements except C (P=0.06), there was a significant decrease in O (P=0.001), F- (P=0.0001), Na+ (P=0.01), SiO₃²⁻ (p=0.0001), and BO₃³⁻ (P=0.0001) the significance started at T2, while there was a significant increase in Al³⁺ (p=0.003), and Sr²⁺ (P=0.000) the significance started at T2, as presented in table 6 and figure 5.

TABLE (6) Comparison between T1, T2, and T3 regarding all elements in Group I:

GI	T1		T2		T3		
	M	SD	M	Sd	M	SD	
C	31.60 a	6.47	35.81 a	1.78	35.49 a	1.32	0.06
O	32.10 a	1.59	27.34 b	0.83	28.50 b	0.91	0.001*
F-	4.27 a	1.25	1.62 b	0.28	1.50 b	0.23	0.0001*
Na+	1.85 a	0.46	1.41 b	0.10	1.49 b	0.29	0.01*
Al ³⁺	9.13 a	0.94	10.16 b	0.24	9.89 b	0.39	0.003*
SiO ₃ ²⁻	9.52 a	0.78	8.35 b	0.17	8.14 b	0.24	0.0001*
Sr ²⁺	9.77 a	0.58	15.31 b	0.46	14.98 b	0.66	0.0001*
BO ₃ ³⁻	7.55 a	0.88	4.70 b	0.84	3.70 b	0.94	0.0001*

*Significant difference as P<0.05.

Means with different superscript letters per raw were significantly different as P<0.05.

Means with the same superscript letters per raw were insignificantly different as P>0.05.

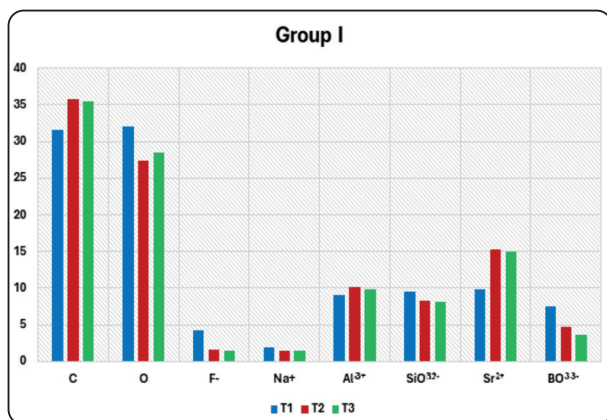


Fig. (5) Bar chart representing Comparison between T1, T2, and T3 regarding all elements in Group I.

Group II:

There was a significant change by time in all elements except Ca (P=0.06), there was a significant decrease in C (P=0.001), O (P=0.004), F- (P=0.0001), Na+ (P=0.01) while there was a significant increase in Al³⁺ (p=0.0001), Sr²⁺ (P=0.000), N (0.0001), Y (0.0001) and Ba (0.0001). In all elements the significance started at T2, as presented in table 7 and figure 6.

TABLE (7) Comparison between T1, T2, and T3 regarding all elements in Group II:

GII	T1		T2		T3		
	M	SD	M	SD	M	SD	
C	38.69 a	4.18	30.96 b	5.11	33.48 b	2.01	0.001*
O	30.69 a	5.49	25.42 b	2.02	25.26 b	1.67	0.004*
F-	3.07 a	1.02	1.37 b	0.53	1.17 b	0.18	0.0001*
N+	0.90 a	0.47	0.00 b	0.00	0.00 b	0.00	0.0001*
Al ³⁺	3.99 a	0.90	5.55 b	0.40	5.54 b	0.44	0.0001*
SiO ₃ ²⁻	8.57 a	1.49	12.75 b	2.50	11.34 b	0.85	0.0001*
Ca	3.85 a	0.99	5.26 a	2.31	3.68 a	1.12	0.08
N	3.71 a	1.67	9.03 b	2.10	9.72 b	1.08	0.0001*
Y	0.30 a	0.08	0.70 b	0.19	0.83 b	0.27	0.0001*
Ba	5.84 a	0.85	9.59 b	1.43	8.97b	0.95	0.0001*

*Significant difference as P<0.05.

Means with different superscript letters per row were significantly different as P<0.05.

Means with the same superscript letters per row were insignificantly different as P>0.05.

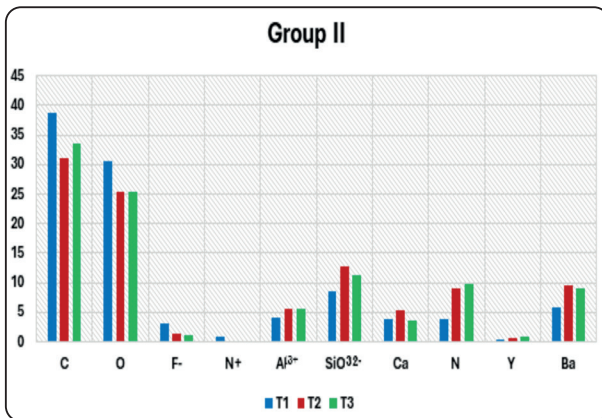


Fig. (6) Bar chart representing Comparison between T1, T2, and T3 regarding all elements in Group II.

Group III:

There was a significant change by time in all elements except SiO32 (P=0.9), there was a significant decrease in C, O, F-, Na+ and B (P=0.0001) while there was a significant increase in Al3+, Sr2+ , Ca, P, K N (P=0.0001) . In all the elements the significance started at T2 except O as the significance started at T3, as presented in table 8 and figure 7.

TABLE (8) Comparison between T1, T2, and T3 regarding all elements in Group III:

GIII	T1		T2		T3		
	M	SD	M	SD	M	SD	
C	27.26 a	0.55	18.81 b	3.13	18.96 b	2.02	0.0001*
O	42.16 a	0.71	40.89 ab	1.65	39.58b	0.71	0.0001*
F-	3.92 a	0.32	3.15 b	0.69	2.77 b	0.15	0.0001*
N+	1.51 a	0.26	1.56 a	0.18	1.12 b	0.11	0.0001*
I3+	7.20 a	0.34	10.44 b	1.48	10.03 b	0.30	0.0001*
SiO ₃ ²⁻	7.43 a	0.44	7.32 a	1.00	7.41 a	0.55	0.90
Sr ²⁺	4.48 a	0.40	9.76 b	1.28	9.97 b	0.24	0.0001*
Ca	2.37 a	0.57	3.87 b	0.53	3.41 b	0.08	0.0001*
P	2.24 a	0.08	4.22 b	1.15	4.65 b	0.67	0.0001*
K	1.11 a	0.18	1.82 b	0.25	2.09 c	0.20	0.0001*
B	0.70 a	0.44	0.00 b	0.00	0.00 b	0.00	0.0001*

*Significant difference as P<0.05.

Means with different superscript letters per row were significantly different as P<0.05.

Means with the same superscript letters per row were insignificantly different as P>0.05.

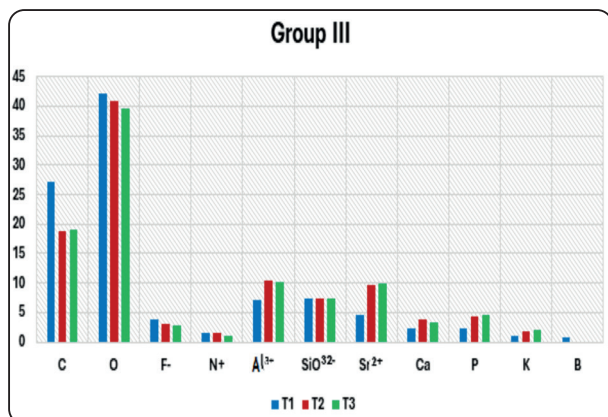


Fig. (7): Bar chart representing Comparison between T1, T2, and T3 regarding all elements in Group III.

TABLE (9) Comparison between T1, T2, and T3 regarding all elements in Group IV:

G IV	T1		T2		T3		P value
	M	SD	M	SD	M	SD	
C	17.81 a	0.76	19.22 a	2.96	16.11 a	6.44	0.29
O	47.74 a	3.72	40.27 b	1.11	40.21 b	0.94	0.0001*
F-	4.98 a	0.33	2.60 b	0.21	3.59 c	1.37	0.0001*
N+	1.17 a	0.99	1.92 b	0.32	1.82 ab	0.28	0.03*
Al ³⁺	5.91 a	0.48	10.06 b	1.28	11.72 b	0.93	0.0001*
SiO ₃ ²⁻	5.46 a	0.28	8.69 b	1.00	9.57 b	1.06	0.0001*
Sr ²⁺	0.05 a	0.04	2.99 b	0.38	3.41 b	0.46	0.0001*
Ca	11.43 a	0.60	0.00 b	0.00	0.00 b	0.00	0.0001*
P	0.65 a	0.57	2.70 b	0.65	3.92 c	1.19	0.0001*
K	0.13 a	0.11	5.08 a	7.22	2.21 a	0.26	0.06
Ag	1.94 a	1.08	5.72 b	1.02	7.45 b	2.25	0.0001*
Ti	2.74 a	0.83	0.00 b	0.00	0.00 b	0.00	0.0001*

*Significant difference as $P < 0.05$.

Means with different superscript letters per row were significantly different as $P < 0.05$.

Means with the same superscript letters per row were insignificantly different as $P > 0.05$.

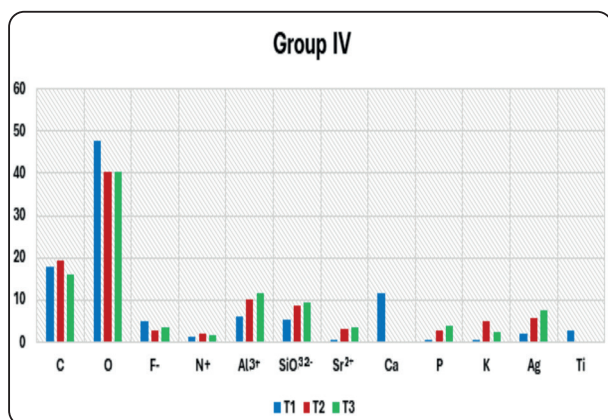


Fig. (8): Bar chart representing Comparison between T1, T2, and T3 regarding all elements in Group IV.

Group IV:

There was a significant change by time in all elements except C ($P=0.29$) and K ($P=0.06$), there was a significant decrease in O, F-, SiO₃²⁻, Ca, and Ti ($P=0.0001$) while there was a significant increase in N +, Al³⁺, Sr²⁺, P, Ag ($P=0.03, 0.0001$). In all the elements the significance started at T2 except O as the significance started at T3, as presented in table 9 and figure 8.

Group V:

There was a significant change by time in all elements as $P=0.0001$, there was a significant decrease in C, O, F-, and N+ while there was a significant increase in Al³⁺, SiO₃²⁻, Ca, and P. ($P=0.03, 0.0001$). In all the elements the significance started at T, as presented in table 10 and figure 9.

TABLE (10) Comparison between T1, T2, and T3 regarding all elements in Group V:

G V	T1		T2		T3		P value
	M	SD	M	SD	M	SD	
C	25.18 a	0.98	20.84 b	0.77	19.05 b	1.33	0.0001*
O	44.60 a	0.80	39.43 b	0.28	39.12 b	0.33	0.0001*
F-	3.99 a	0.30	3.36 b	0.33	4.17 a	0.32	0.0001*
N+	3.39 a	0.21	3.74 b	0.17	3.19 a	0.30	0.0001*
Al ³⁺	8.02 a	0.26	11.04 b	0.11	11.60 b	0.64	0.0001*
SiO ₃ ²⁻	9.06 a	0.55	12.58 b	0.48	12.39 b	0.28	0.0001*
Ca	3.98 a	0.16	6.06 b	0.24	6.33 b	0.41	0.0001*
P	0.87 a	0.10	0.91 a	0.14	1.87 b	0.49	0.0001*

*Significant difference as P<0.05.

Means with different superscript letters per raw were significantly different as P<0.05.

Means with the same superscript letters per raw were insignificantly different as P>0.05.

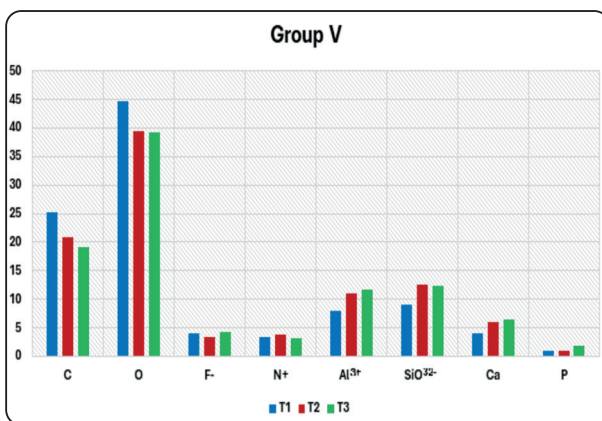


Fig. (9) Bar chart representing Comparison between T1, T2, and T3 regarding all elements in Group V.

DISCUSSION

Major improvements in restorative materials show a paradigm change from a “passive” material that might restore the form and function of a tooth to an “active” material that promotes tooth remineralisation and improves the marginal integrity of a restoration, which are bioactive materials. A restorative material’s bioactivity is determined by a number of characteristics, including its capacity to remineralise tooth structure, promote the production of hydroxyapatite, bind chemically to the tooth surface through ion exchange, have antimicrobial qualities, and be biocompatible. To be genuinely

bioactive, a restorative material needs to show signs of hydroxyapatite production. The release of calcium, phosphate, and fluoride ions in vitro can be used to evaluate this indirectly¹³.

In a healthy environment, the released calcium can hasten the mending of hard tissues by combining with phosphate ions found in the saliva or in the restorative material itself. When low pH and moisture are present, the Si-O-Si bonds that are naturally present in bioactive glass hydrolyse, causing fluoride, calcium, silicon, and hydroxyl ions to be released quickly into the oral environment. Ion deposition to the tooth structure and the prevention of bacterial growth are made possible by the alkaline environment that is consequently formed by the release of hydroxyl ions¹³. Clinically speaking, the pH of the oral environment can rise due to the release of calcium and phosphate ions, which can result in the deposition of an apatite-like material.

Existing literature suggests that the release of calcium and phosphate from bioactive materials has positive effects in caries-prone areas. Nevertheless, the bioactivity of these materials directly depends on the amount of ion release, which hasn’t been well documented.

Researches had shown that metallic nanoparticles have antibacterial and remineralising qualities whether used alone or in composite materials. They could also be applied to enhance the mechanical and cosmetic qualities of dental materials. Ions and proteins found in saliva can combine with nanoparticles to form ion-protein complexes, which then precipitate onto tooth surfaces²¹.

The intent of this study was to quantify the release of ions from bioactive restorations in standard solutions, such as artificial saliva (positive control) and distilled water (negative control), as well as in a pH-cycling model (demineralising and remineralising solutions) to mimic oral conditions and dynamic scenarios. In addition to having a low pH, the cola beverage was chosen as a demineralising solution because of its low levels of calcium and fluoride ions. Every day, the solution was replaced to prevent fluoride from building up in the immersion solutions containing the specimens and to guarantee that the undersaturation state remained constant¹⁹.

The bioactive restorative materials used in the current study were: Giomer (Gi); (Beautiful II), this is a new class of composite resins that have just been created for direct adhesive restorations. They offer the benefits of composite resins (easier to polish, biocompatible, and fluoride release) as well as glass-ionomers (recharging capacity and fluoride release). Bulk-fill giomer was created with the promise of anticariogenic characteristics, aesthetics, durability, and comfort rapid treatment in both high and low viscosity forms³.

Giomer is based on pre-reacted glass (PRG) technology, in which fluorine including glass particles and polymer-containing acid react acid-base in the presence of water to produce a glass-ionomer phase before being dispersed into the resin. Giomer is known for its "bioactive effects by sustained release of (6 kinds of) multi-ion." The fragile glass-ionomer phase, which is fully reacted with polyacrylic acid, comprises the filler's core in F-PRG fillers. Only

the glass filler's surface experiences the glass-acid reaction in S-PRG fillers, leaving the core completely intact.

As a result S-PRG fillings are considered stronger due to their increased resistance to deterioration. The giomer materials' S-PRG fillers provide prolonged fluoride release and rechargeability, providing the tooth with ongoing guard against the recurrence of caries. Gionomers offer the advantages of amalgam and composite resin materials since they maintain the tooth structure while offering long-term durability and aesthetics together with ion release. The traditional version, Beautiful II (Shofu Inc.), is a nano-hybrid composite that combines the qualities of glass ionomers and composite resins. With a total filler content of 83.3 wt% (68.6 vol%) and distinct nanofillers (10–20 nm), it is clinically appropriate for all kinds of restorations in the anterior and posterior regions³.

Giomer is a resin-based dental adhesive that releases fluoride and is made of glass fillers that have already undergone a reaction. Fluoroaluminosilicate glass (FASG) and polyalkenoic acid (PAA) react acid-base in the presence of water to generate a wet siliceous hydrogel, which is how PRG fillers are made. The desiccated xerogel was further ground and silanized after the freeze-drying procedure to create PRG fillers with a particular size range. The surface reaction between the glass ionomer and the acid determines the PRG-fillers that are a part of the Giomer product (surface reaction type, S-PRG fillers).

The surface reaction is comprised of three layers: an initial glass core made of multifunctional fluoro-boro-aluminosilicate glass, a pre-reacted glass-ionomer phase on the glass core's surface, and an outer SiO₂ coating layer, a reinforced modified layer, covering the pre-reacted glass-ionomer phase's surface. The primary prerequisite for enhancing the apatite production capacity and dentin mineralisation in the presence of phosphate-

containing solutions is the availability of mineral ions (calcium and fluoride) from restorative materials during the remineralising process^{3,39}.

Recently, Ivoclar Vivadent (Cention forte) (G II) was released as a tooth-colored restorative filling material that may be placed in large quantities in retentive preparations with or without the use of an adhesive. Like compomer, it belongs to a new class of filling material called "alkasite" restoratives, which are essentially a subgroup of composite resin. Cention forte is a self-curing restorative based on urethane dimethacrylate (UDMA) that has the option of extra light curing. Dimethacrylates and initiators make up the liquid, and different glass fillers, colours, and initiators are present in the powder. It is radio opaque and has glass fillers that are alkaline and can release hydroxide, calcium, and fluoride ions. Because Cention forte uses only cross-linking methacrylate monomers along with a stable, effective self-cure initiator, it exhibits a high degree of polymerisation and polymer network density over the entire depth of the restoration.

Advertised as an alkasite (a RBC with alkaline fillings), cent forte (CF; Ivoclar Vivadent, Schaan, Liechtenstein) boasts an improved polymerisation method and extra bioactive or ion-releasing qualities. Because of the release of hydroxide (OH⁻) ions, they have alkalisising capabilities, which is how they get their name, alkasite. Its wear patterns and mechanical and micromechanical characteristics make it suitable for use in posterior restorations. Because of its bioactivity, CF exhibits higher monomer conversion than typical RBCs and can reduce dentin demineralization³.

Ketac Molar (Glass ionomer) (3M ESPE, St. Paul, MN, USA) (GIII), the powder part of glass ionomer cements is made up of calcium fluoride-rich crystals. The negative fluoride ions are released from the powder together with the positive Ca²⁺, Al³⁺, and Na⁺ ions once the liquid and powder have been mixed and the acid/base reaction is

started. These ions then accumulate as ions, salt compounds, or complex compounds in the cement matrix. These compounds supply the majority of the fluoride released from the set cement. Glass ionomer cements have a high degree of fluoride discharge at first because the filling's surface stores the majority of the fluoride that is emitted. Over the course of several months, the rate of fluoride emission decreases and eventually stabilises at a steady state. From the filling's core to its surface, where it enters solution, the released fluoride moves. The refluoridization of glass ionomer cement fillings can be improved by using fluoride-containing toothpaste, gels, or solutions.

Ketac silver (Glass ionomer) (3M ESPE, St. Paul, MN, USA) (G4), showed good antibiofilm effects, sintered silver GICs created a burst effect.

Zirconomer®Improved (Shofu Inc. Kyoto, Japan) (G5) material is made up of tooth-colored restorative materials and ceramic and zirconia reinforced glass ionomer cements, which may be able to solve amalgam's shortcomings. Its manufacturers stated that it maintains the ability of GIC to release fluoride while displaying the strength of amalgam. It was reported to have good antibacterial activity against cariogenic bacteria and is considered as a promising biomaterials⁹.

Reviewing the recent in vitro and in vivo studies, we can summarize the action of different ions as follows:

Fluoride release has a key role in the oral cavity in preventing caries by reducing the demineralization and enhancing the remineralization of dental hard tissues, plus restraining growth of the microbial flora.

The attainment of this antibacterial and cariostatic activity is contingent upon the quantity of fluoride emitted. The fluoride elution process is not simple and is influenced by a number of variables, including the composition of the storage

media, flow, plaque formation, pH of the oral cavity, ratio of powder to liquid, curing time, and exposed surface area. It is required to characterise any newly developed material or new construction made of an existing material for which the maker claims to have improved its mechanical or physical attributes in order to fully release its rights²². Sodium fluoride (NaF) may be effective in slowing the advancement of dental cavities and encouraging the remineralisation of enamel²³.

It was noted that the combination of silver nanoparticles and sodium fluoride demonstrated synergistic potential for caries prevention and remineralisation. Silver nanoparticles with sodium fluoride solution have been shown in a clinical investigation to prevent dentine caries without staining²¹.

Though it can also function as a radical scavenger, alumina creates stable nanoparticles (NPs) that are anti-oxidants and antimicrobials by breaking down the bacterial cell wall through the production of Reactive Oxygen Species (ROS). Furthermore, there is no toxicity to human cells from these NPs. This nanoparticle works best when combined with other oxides, such as TiO₂, which have the ability to scavenge radicals and function as an antibacterial. Thus, there are two purposes for using Al-Ti bimetal oxide nanoparticles²⁴.

According to reports, silica gel enhanced apatite nucleation on its surface, suggesting that silicon may facilitate the production of hydroxyapatite. The hydrated silica gel surface silanol groups reacted with the surrounding calcium and phosphorus ions to form physiologically active apatite on the silica gel surface. On the other hand, it was documented that the surface of enamel was adsorbed with Si ions that were liberated from bioactive glass particles, hence offering locations for the nucleation of heterogeneous apatite. Once nucleated, it will proliferate on its own to create an apatite layer that resembles bone. It was reported that when sodium

silicate was used as a catalyst for apatite nucleation, an apatite layer was formed for bonelike apatite coating on materials with complex shapes. The majority of the apatite nucleation was attributed to specific silicate oligomers with structures like dimer, linear trimer, and cyclic tetramer. According to reports, silicon (Si) aided in the mineralisation of dentin by a process that involved silicic acid condensation into oligomers. When enough Si was adsorbed on dentin containing anionic groups (SiO⁻), it served as a site of nucleation for the following, enhanced production of apatite²⁵.

To improve the bonding capabilities of bioactive glass, strontium can also be added. The addition of fluoride and strontium to bioactive glass can prevent the acid produced by cariogenic bacteria from dissolving hydroxyapatite. Strontium has a synergistic caries-inhibiting action with fluoride and can take the role of calcium in precipitate formation. Consequently, strontium can stop caries lesions because of its ability to remineralise tooth hard tissues^{26, 27, 28}. It was reported that when combined with fluoride, strontium ions produced by S-PRG filler seems to have the ability to improve enamel remineralisation. It was anticipated that strontium and fluorine ion release would encourage the development of strontium fluorapatite on dentinal surfaces. Because of their structural closeness, strontium can substitute calcium on dentinal surfaces²⁹. Sr has the potential to improve enamel remineralisation when combined with F30, according to a newly published study. It has also been observed that elevated concentrations of strontium ion (Sr²⁺) have anticariogenic properties. Because of its affinity for exchanging calcium for hydroxyapatite crystals, teeth are somewhat protected against caries³¹.

Regarding calcium ions, their release is necessary for a restorative material to be able to cause the production of hydroxyapatite in a physiological setting. Hard tissue repair may be aided by the

released calcium's reaction with phosphate ions found in saliva or the restorative substance itself¹³. Additionally, it has been demonstrated that CaO nanoparticles work well against both Gram-positive and Gram-negative bacteria. Their ability to break the cell membrane, which leads to the leakage of intracellular contents and ultimately to cell death, is what gives them their antibacterial activity³². Over the past few decades, the clinical application of calcium and phosphate ions has not proven to be successful. It is difficult to use insoluble calcium phosphates in dental procedures.

Because calcium phosphates are intrinsically insoluble, soluble calcium and phosphate ions are only useful at extremely low concentrations. During the remineralisation process, the bioavailability of calcium and phosphate ions is always constrained.

Fluoride can prevent enamel and dentine from demineralising and speed up the remineralisation of carious lesions. Because calcium fluoride nanoparticles have a higher degree of labile fluoride concentration in oral fluid, they were developed as anti-caries agents. Calcium fluoride nanoparticles can limit the formation of biofilms and significantly lower the synthesis of exopolysaccharides²¹.

Utilising bioactive chemicals is a viable way to guarantee the release of phosphate ions, maximise the alkalinising potential of bioactive dental materials, and promote tooth remineralisation in the long run³³.

By encouraging the synthesis of fluorapatite in the presence of calcium and phosphate ions created during enamel demineralisation by plaque bacterial organic acids, fluoride plays a significant role in the remineralisation and repair of early lesions. Fluoride deposition is made possible by tiny porosities on the enamel's surface³⁴. Fluoride-containing dental materials show distinct variations in their fluoride release and absorption properties. They can also operate as a reservoir for fluoride, raising the concentration of fluoride in hard oral tissues like saliva and teeth,

or they can prevent or lessen secondary caries³⁵. In addition to inhibiting bacterial and fungal quorum sensing, boron is known to have antibacterial activity in cutaneous disorders and periodontitis. Since quorum sensing is essential for the production of biofilms, it stands to reason that S-PRG acts by inhibiting this function in streptococci²⁵.

The literature has also revealed boron's cariostatic effects. Teeth that have accumulated boron in the form of calcium borate rather than calcium phosphate are more resistant to dental caries³¹. Sr and B possess remineralising, acid-resistant, and antibacterial qualities³⁶. It has also been found that barium substitutes calcium in dentin hydroxyapatite³¹.

Ag⁺ ions discharged (leached) from the surface exert the well-known silver ions, which are extensively studied for their bactericidal action. It has been discovered that AgNPs can limit the growth of *P.aeruginosa* in dental plaque biofilms. AgNPs exhibit strong antibacterial action against a range of pathogenic bacteria, including oral pathogens, according to several investigations. When used as an antibacterial therapy, AgNPs demonstrated tremendous potential in both preventing dental cavities and controlling strains of *S. sobrinus* and *Streptococcus mutans*.

It is hypothesised that when a bacterial cell comes into contact with silver nanoparticles, it will absorb silver ions, which could lead to the production of free radicals and subsequent damage to the cell membrane caused by free radicals³².

AgNPs have the ability to prevent cariogenic pathogens—particularly *Streptococcus mutans*—from growing and adhering. AgNPs possess antifungal and antiviral properties as well. Research has indicated that AgNPs possess broad-spectrum antibacterial capabilities in vitro. AgNPs based on anticaries agents exhibit a useful bactericidal action against the biofilm of *Streptococcus mutans*. Furthermore, AgNPs can prevent the formation of

lactic acid and collagen degradation by biofilm, which can slow down the demineralisation of teeth. Researchers have attempted to mix AgNPs with fluoride to give them mineralising qualities. AgNPs and sodium fluoride together demonstrated a higher remineralisation effectiveness than sodium fluoride, according to an *in vitro* study²¹.

According to a randomised clinical experiment, 66.7% of dentine caries in primary teeth may be stopped after 12 months with an annual application of nano-silver fluoride³⁷.

According to current reports, TiO₂ nanoparticles function as anti-microbial agents against a broad range of bacteria and are incredibly durable. When TiO₂ absorbs a high energy photon, its electrons become excited and bounce from the valence band to the conduction band, creating an energy hole (h⁺) and a free electron (e⁻), which together with an oxidant reduce the product. This is why TiO₂'s photocatalytic properties are particularly helpful in the eradication of bacteria. In reductive reactions, these electrons join forces with an oxygen molecule to form O₂⁻, which produces hydroxyl radicals when combined with water. TiO₂'s photo-catalytic activity is highly beneficial in breaking down organic and hydrocarbon pollutants in water. Furthermore, the metal oxide is able to resist bacterial growth, limit biofilm formation, and destroy harmful substances as a result of this activity²⁴.

The highly stable particles known as titanium dioxide nanoparticles (NPs) have the ability to catalyse photosynthesis. Reactive oxygen species are produced, which leads to oxidative stress. Its ability to peroxide lipids gives it exceptional membrane fluidity and disrupts cell membranes. Additionally, it works well against strains resistant to fluconazole³².

Glass ionomer cement incorporating titanium nanoparticles is a potentially helpful restorative material to avoid cavities. The use of titanium nanoparticles into restorative glass ionomer cement significantly enhanced its microhardness,

compressive strength, flexural strength, and antibacterial activity³⁸, while maintaining the cement's adhesion to enamel and dentine.

Zirconia, because of its optical and metallic characteristics that are comparable to those of teeth, its chemical oxide has found extensive use in dentistry. ZrO₂ is a ceramic substance that has low cytotoxicity and strong antibiofilm capabilities. Zirconia-based NPs are frequently utilised as anti-microbial agents due to their high efficacy against particular bacteria, like *E. faecalis*³².

The largest value of free energy released during the creation of the oxide form from the elemental form makes yttrium oxide, a metal oxide, significant. Furthermore, NPs' antioxidant qualities guard against excessive oxidative stress-induced cell death²⁴.

This study intended to measure the release of ions from bioactive restorations in standard solutions, such as artificial saliva (positive control) and distilled water (negative control), as well as in a pH-cycling model (demineralising and remineralising solutions) to mimic oral conditions and dynamic scenarios. In addition to having a low pH, the cola beverage was chosen as a demineralising solution because of its low levels of calcium and fluoride ions. Every day, the solution was replaced to prevent fluoride from building up in the immersion solutions containing the specimens and to guarantee that the undersaturation state remained constant¹⁹.

Assessing the release of certain ions from various bioactive restorative materials that can be utilised as posterior restorations was the goal of the current investigation. Other ions were unintentionally traced during the specimens' analysis by EDX, which encouraged us to expand the scope of the ions we examined, particularly after discovering that these elements are known to have cariostatic effects in the literature. This finding altered the direction of the investigation.

The null hypothesis was rejected as the results of the current study, showed variability in ions release of tested materials in comparison to (G I) Giomer,

Some ions, like Al in (G I) Giomer, K in (G III) Ketac molar, and F, CA, T & BO in (G IV) Ketac silver, had an early burst of release before fading away; these elements vanished within seven days. An early ion burst is a common outcome that was consistent with earlier researches^{22,30,33}.

According to another study³⁹, the RMGI (Fuji II LC) group's mean values for the release of St, Bo, Na, and Al ions were substantially lower than those of the SPRG (Giomer; Beautifil II) group. Since St, Bo, Na and Al are poorly bound to glass filler, they ascribed this discovery to the S-PRG technology, which enables the development of a stable pre-reacted glass phase to prevent further release of silicon and fluoride. Water sorption happens when Giomer restorations come into touch with a moist environment, allowing S-PRG fillers to release ions like St, Bo, Na and Al.

On the other hand, a recent *in vitro* study⁴⁰ found that the ion-releasing capacity of the restorative systems examined by Giomer and GIC appears to be similar. Other ions, including Sr & Bo in (G I) Giomer, N, Y & Ba in (G II) Cention Forte, P in (G III) Ketac molar, K & Ag in (G IV) Ketac silver, and Na in (G V) Zirconomer, were released during the study. This release pattern might indicate a good likelihood of a long-lasting cariostatic effect. Another observation is that some ions, like (GV) Zirconomer, exhibit delayed release patterns that show up late on the spectrum. This delayed release of Na and F was observed after 7 days and continued for 28 days.

These findings were consistent with those of a previous study⁴¹, which found that Cention Forte does not have a burst effect but continuously releases fluoride during the test period. This could be because of a larger powder/liquid ratio and a higher proportion of alkaline glass in the final state.

According to a different study, the zirconomer's quick fluoride elution pattern can be attributed to precisely regulated glass ionomer particle micronization. Numerous studies have indicated that smaller glass particles create a greater interface because they have a higher surface area. As a result, these materials exhibit increased fluoride release due to an acceleration of acid-base reactivity. Although the amount of fluoride released may be greatly influenced by the materials' porosity, the mechanical qualities suffer as a result. According to the scientists, the enhanced mechanical qualities of this material, which make it appropriate for posterior load-bearing areas, are caused by the use of nano-zirconia fillers.

The transition toughening feature of zirconia fillers allows them to prevent cracks from spreading. Zirconia in the tetragonal phase changes into a stable monoclinic phase as a result of the stress caused by crack propagation; a small increase in volume during this transition also improves compressive strength. The material's toughness and elastic modulus are increased when alumina and yttrium stabilised zirconia are combined⁴².

Each material contains a unique cocktail of bioactive ions that work together to produce an appropriate bioactive effect, and the materials appeared to have an acceptable rate of ion release. Ion release over short and long periods of time is dependent on a number of factors, including the fact that many materials exhibit a double setting reaction that appears to impair their ultimate characteristics. The basic composition of the material, the size and proportion of filler, the type of setting reaction, and the kind of bond linking the filler particles and the matrix, are some of the variables that might affect these qualities. Each material has a unique microstructure, and the examined materials have varying solubilities.

Generally speaking, all of these bioactive materials contain ions like C, O, F, Na, Al, and Si.

Additionally, each of the tested bioactive materials has unique ions with unique activities, such as Ba, N, and Y found in (G II) Cention Forte, Ag & Ti in (G IV) Ketac silver, and Zr in (G V) Zirconomer. It is important to focus on how these various ions interact and how that affects the release of the final ions.

Another significant problem is that manufacturers withhold the precise chemical composition or identification, concentrations, and particle sizes as trade secrets, making it challenging and complex to compare the materials.

However, because the oral environment is dynamic and the results must be adequately inferred, the present outcomes cannot be generalised. Further studies with large sample size and long-term ions release need to be further explored.

Clinical studies may be carried out to estimate the amount of long-lasting ions release and to assess caries recurrence around the margins of these bio active restorations.

CONCLUSIONS

The ions release from bioactive restorative materials is time and composition dependent.

With similar releases of cariostatic ions, all studied restorative materials can be regarded as promising bioactive materials employed as posterior restorations.

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