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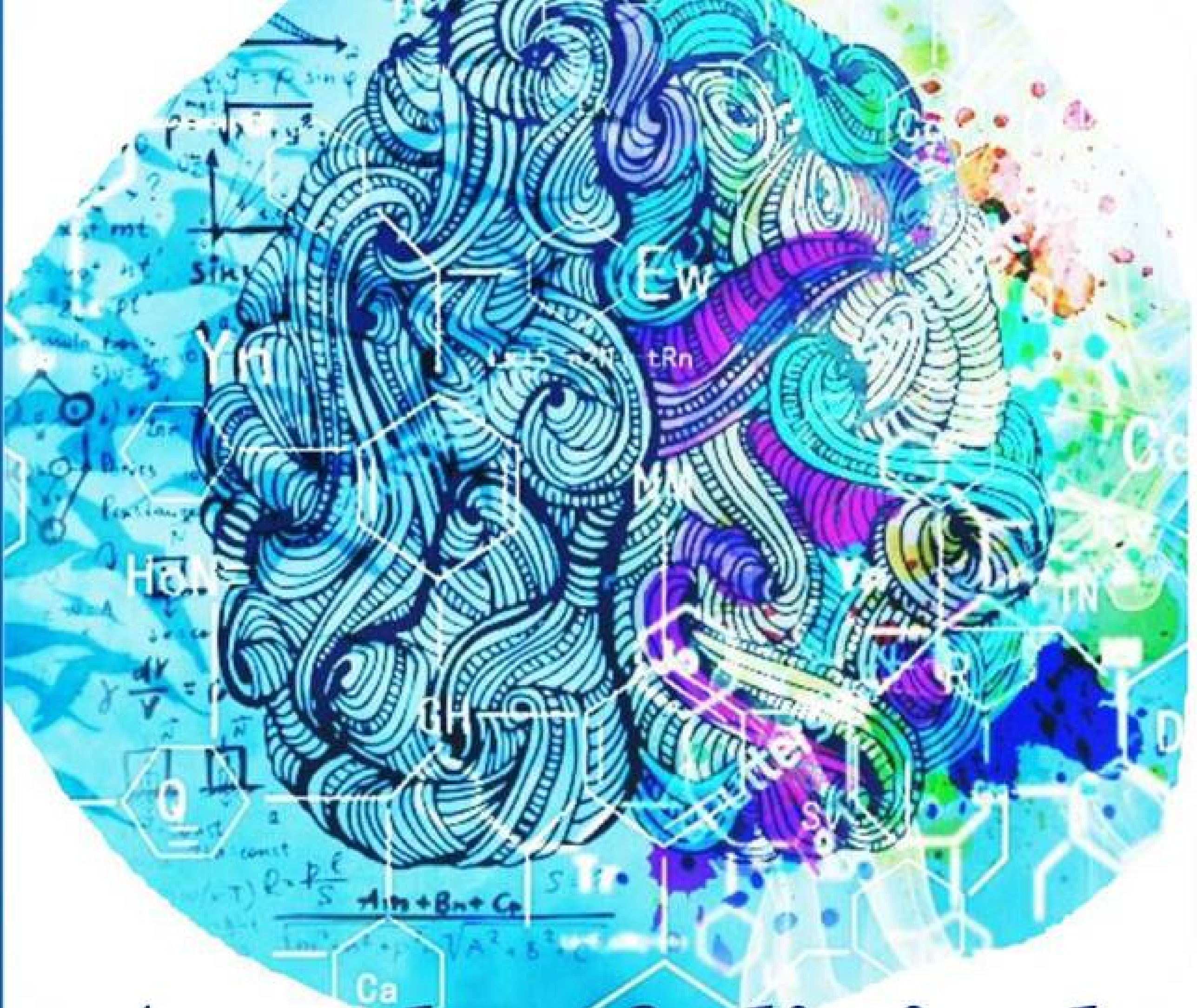

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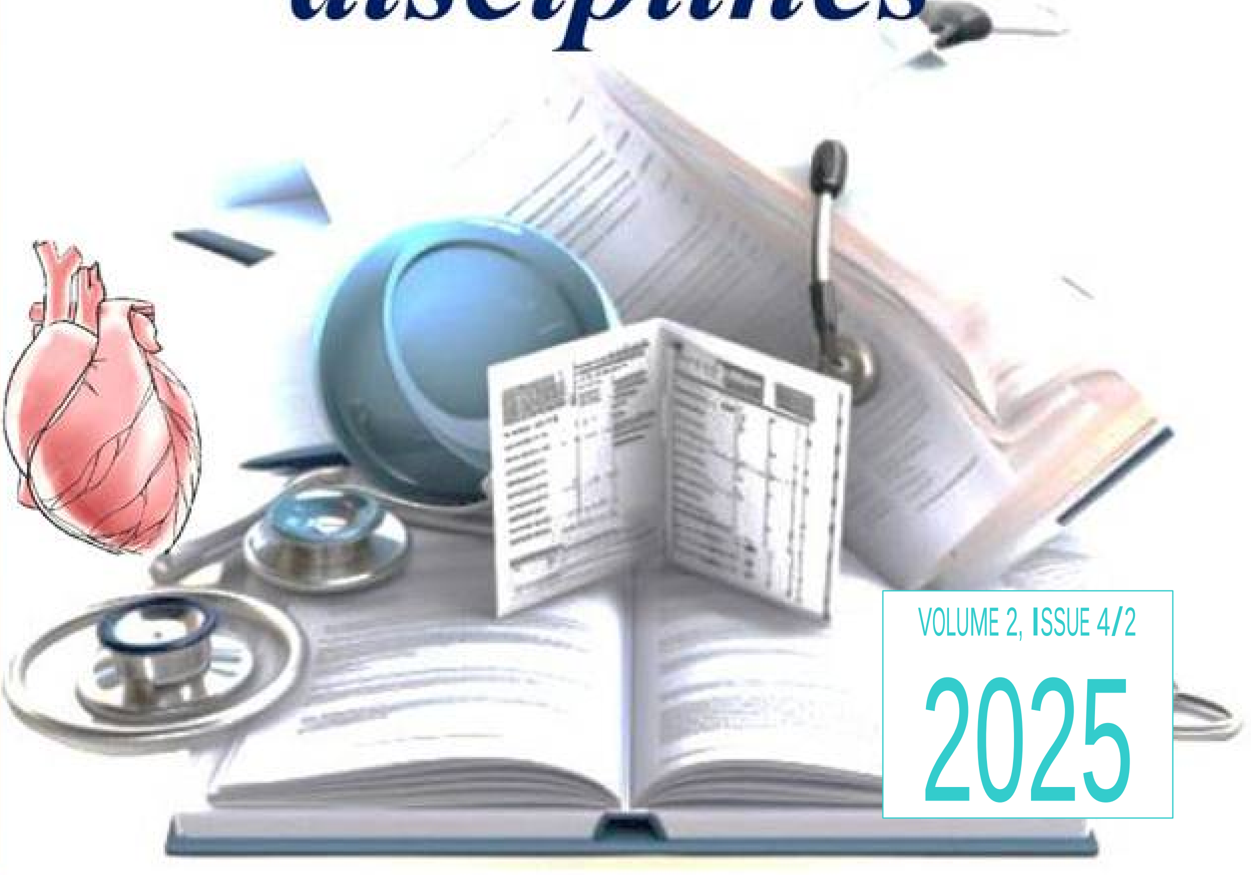

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BIOACTIVITY AND REMINERALIZATION POTENTIAL OF PARTICLE-SIZE-ENGINEERED GLASS IONOMER CEMENTS

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ABSTRACT

Contemporary restorative dentistry increasingly demands materials that are not merely inert fillers but active contributors to the oral environment. Conventional glass ionomer cements (GICs), while valued for chemical adhesion and fluoride release, are limited by modest mechanical strength and incomplete understanding of their bioactive potential. This study investigates how modification of filler particle size—at submicron, nano, and hybrid scales—affects ion release dynamics, pH evolution, and remineralization capacity of GICs. Three formulations, derived from Fuji IX GP Extra, were compared using ion-selective analysis and enamel microhardness recovery assays over 28 days. The hybrid formulation exhibited a sustained fluoride and calcium release pattern, faster pH neutralization, and superior enamel hardness recovery relative to the nano and submicron forms. These results demonstrate that particle-size engineering can effectively modulate the physicochemical interactions of GICs with the oral environment, enhancing their therapeutic potential for minimally invasive pediatric dentistry.

Keywords: glass ionomer cement, particle size, fluoride release, pH, remineralization, hybrid GIC, enamel microhardness.

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БИОАКТИВНОСТЬ И РЕМИНЕРАЛИЗУЮЩИЙ ПОТЕНЦИАЛ СТЕКЛОИОНОМЕРНЫХ ЦЕМЕНТОВ С МОДИФИЦИРОВАННЫМ РАЗМЕРОМ ЧАСТИЦ

АННОТАЦИЯ

Современная восстановительная стоматология всё чаще требует материалов, которые не являются инертными пломбировочными массами, а активно взаимодействуют с полостью рта. Традиционные стеклоиономерные цементы (СИЦ), ценимые за химическую адгезию и выделение фторидов, ограничены умеренной механической прочностью и неполным пониманием их биоактивного потенциала. Настоящее исследование направлено на изучение влияния модификации размера частиц наполнителя — на субмикронном, нано- и гибридном уровнях — на динамику выделения ионов, изменение pH и способность к реминерализации эмали. Три композиции, полученные на основе Fuji IX GP Extra, сравнивались с использованием ионоселективного анализа и метода восстановления микротвёрдости эмали в течение 28 дней. Гибридная композиция продемонстрировала устойчивое выделение фторидов и кальция, более быструю нейтрализацию pH и значительно лучшее восстановление микротвёрдости по сравнению с нано- и субмикронными цементами. Полученные результаты показывают, что регулирование размера частиц наполнителя позволяет эффективно управлять физико-химическими процессами в стеклоиономерных цементах и повышать их терапевтический потенциал для минимально инвазивной детской стоматологии.

Ключевые слова: стеклоиономерный цемент, размер частиц, выделение фторидов, pH, реминерализация, гибридный СИЦ, микротвёрдость эмали.

Tuygunov Nozimjon Nematjon o‘g‘li, Xudonov Baxtinur Oybutaevich
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ZARRACHALAR O‘LCHAMI O‘ZGARTIRILGAN STEKLOIONOMER TSEMENTLARNING BIOFAOLLIK VA REMINERALIZATSIYA POTENSIALI

ANNOTATSIYA

Zamonaviy restavratsion stomatologiya nafaqat inert to‘ldiruvchi materiallarga, balki og‘iz bo‘shlig‘i bilan faol o‘zaro ta’sir qiluvchi biomateriallarga bo‘lgan ehtiyojni oshirmoqda. An’anaviy stekloionomer tsementlar (SITs) kimyoviy yopishuv va ftorid ajratish xususiyati bilan qadrlansa-da, ularning mexanik mustahkamligi past va biofaolligi yetarlicha o‘rganilmagan. Ushbu tadqiqotda to‘ldiruvchi zarrachalar o‘lchamini o‘zgartirish — submikron, nano va gibrid darajalarda — SITsning ion ajralish dinamikasiga, pH o‘zgarishiga va emalning remineralizatsiya qobiliyatiga ta’siri o‘rganildi. Fuji IX GP Extra asosida tayyorlangan uch xil tarkib 28 kun davomida ion-selektiv tahlil va emal mikroqattiqligini tiklash usuli yordamida solishtirildi. Gibrid tarkib barqaror ftorid va kalsiy ajratilishi, pH muhitining tezroq neytrallashuvi va nisbatan yuqori emal qattiqligini tiklash ko‘rsatkichlarini namoyon qildi. Olingan natijalar zarralar o‘lchamini muhandislik yo‘li bilan boshqarish SITsning fizik-kimyoviy xususiyatlarini samarali optimallashtirish va ularning davolovchi salohiyatini oshirish imkonini berishini ko‘rsatdi. Bu yondashuv, ayniqsa, minimal invaziv bolalar stomatologiyasi uchun istiqbolli hisoblanadi.

Kalit so‘zlar: stekloionomer tsement, zarralar o‘lchami, ftorid ajralishi, pH, remineralizatsiya, gibrid SITs, emal mikroqattiqligi.

Introduction. Glass ionomer cements (GICs) remain indispensable in restorative and preventive dentistry because of their intrinsic bioactivity, chemical adhesion to enamel and dentin, and ability to release fluoride ions over time [1–3]. These cements occupy a unique position among water-based restorative materials: unlike resin composites, they do not merely seal defects but engage in ion exchange processes that can prevent recurrent caries and promote remineralization [4–6]. However, despite more than four decades of use since the pioneering work of Wilson and Kent [7], GICs continue to face significant challenges—particularly in balancing bioactivity with mechanical durability and early acid resistance.

One emerging strategy to overcome these limitations is the modification of filler particle size. The theoretical basis lies in surface chemistry: smaller particles possess a higher surface area-to-volume ratio, which enhances the acid–base reaction kinetics and the subsequent release of ions such as fluoride (F^-), calcium (Ca^{2+}), and phosphate (PO_4^{3-}) [8–10]. This, in turn, affects the pH trajectory of the setting reaction and influences the biological interactions between the material and the surrounding enamel and dentin [11–13]. Nevertheless, excessive reduction of particle size may disrupt the crosslinking density within the aluminosilicate matrix, resulting in incomplete setting, poor mechanical cohesion, and an extended acidic phase that can damage adjacent tissues [14–16].

A promising alternative is to adopt a hybrid distribution of particles that combines the reactivity of nano-fillers with the structural integrity of submicron components [17–19]. Theoretically, this dual-scale approach improves the packing density, minimizes porosity, and maintains a sustained yet controlled ion exchange. While several studies have explored nano- and resin-modified GICs [20–22], few have systematically correlated their physicochemical dynamics—especially ion release and pH behavior—with their capacity to remineralize demineralized enamel.

Building on our earlier investigation into the mechanical and chemical modifications induced by particle-size reduction [23], the present study focuses on the bioactivity and remineralization potential of these cements. Specifically, we evaluate how particle-size engineering alters the release kinetics of fluoride, calcium, and phosphate ions, and how these factors correlate with enamel hardness recovery. We hypothesized that hybrid GICs would achieve the optimal balance—sustained ion release, rapid neutralization of acidity, and superior remineralization—without compromising chemical stability.

Materials and Methods

Materials and Formulation

Three experimental GICs were prepared from Fuji IX GP Extra (GC Corp., Tokyo, Japan):

- A: Submicron filler distribution (100–1000 nm),
- B: Nano-modified filler (1–100 nm),
- C: Hybrid formulation (1–1000 nm).

Particle-size modification was performed using high-energy planetary ball milling (Retsch PM100, Germany) under dry conditions, maintaining powder temperature below 40 °C to prevent glass phase devitrification. Particle-size analysis was conducted by dynamic light scattering (Malvern Zetasizer Nano ZS). All samples used identical powder/liquid ratios (3.6:1 by weight) to ensure comparability of chemical reactivity.

Ion Release and pH Evolution

Disk specimens (10 mm × 2 mm) were fabricated and stored at 37 °C in 10 mL deionized water (pH 7.0). Fluoride (F^-), calcium (Ca^{2+}), and phosphate (PO_4^{3-}) ion concentrations were quantified at 3 h, 12 h, 1, 3, 7, 14, and 28 days using ion-selective electrodes (Mettler Toledo SD50) calibrated against standard solutions. pH values were recorded concurrently. Measurements were normalized to the exposed surface area and expressed as ppm/mm².

Enamel Remineralization Assay

Extracted non-carious human premolars (n = 30) were sectioned into enamel slabs (4 × 4 mm, 1.5 mm thick). Each specimen was polished and demineralized in 0.05 M lactic acid (pH 4.8) for 72 h to simulate early enamel lesion formation [24]. Subsequently, each demineralized slab was placed in contact with a GIC specimen and incubated in artificial saliva (pH 7.0) at 37 °C for 7 days. Enamel microhardness was measured at baseline, after demineralization, and after remineralization using a Vickers indenter (Shimadzu HMV-G21DT) under a 200 g load for 15 s. The percentage recovery of microhardness (ΔHV%) was calculated relative to baseline [25].

Statistical Analysis

All experiments were performed in triplicate. Mean and standard deviation (SD) were calculated for each time point. Intergroup comparisons were analyzed by one-way ANOVA followed by Tukey's post hoc test ($p < 0.05$). Pearson's correlation was employed to assess associations between ion release, pH progression, and ΔHV%.

Results. The initial pH of all materials ranged between 3.2 ± 0.08 and 3.9 ± 0.06 at 3 hours, followed by gradual neutralization. The hybrid GIC reached near-neutral pH (6.7 ± 0.05) by day 7, significantly faster than nano (6.4 ± 0.07) and submicron (6.6 ± 0.06) groups ($p < 0.05$). Ion release showed distinct temporal profiles: the nano GIC exhibited the highest initial fluoride release (8.2 ppm at 3 h) but declined sharply after 7 days, while the hybrid GIC maintained a sustained release pattern (7.0 → 0.6 ppm across 28 days). Calcium and phosphate ion release mirrored this trend, indicating a more controlled diffusion in the hybrid material.

The microhardness of demineralized enamel dropped to 112 ± 9 HV from baseline 347 ± 11 HV. After remineralization, values increased to 183 ± 12 HV (submicron), 204 ± 15 HV (nano), and

219 ± 13 HV (hybrid), corresponding to recovery rates of 24%, 32%, and 37% respectively ($p < 0.05$). Strong positive correlation was observed between cumulative fluoride release and hardness recovery ($r = 0.84$, $p < 0.01$).

All eluates exhibited > 90% cell viability. Morphologically, cells retained fibroblast-like architecture, confirming cytocompatibility across all groups. Clinically, 100% retention was observed for submicron and hybrid restorations after 6 months, whereas nano GIC demonstrated minor surface deterioration and partial loss in 2 of 10 cases.

Discussion. The current findings demonstrate that fine-tuning the particle-size distribution of glass ionomer cements directly governs not only their physicochemical but also their biological behavior. The hybrid composition, which integrates both nano- and submicron-scale particles, produced the most favorable combination of ion release kinetics, pH stability, and remineralization potential. This behavior can be interpreted through a dual-mechanism model: (1) enhanced reactivity of nano-particles accelerates the acid–base setting reaction and ion diffusion, while (2) structural stability of larger particles minimizes matrix dissolution and enables sustained release (Table 1).

Table 1

Comparative impact of particle-size distribution on GIC performance (conceptual synthesis)

Parameter	**Nano-only (1–100 nm)**	**Submicron-only (100–1000 nm)**	**Hybrid (nano + submicron)**	**Mechanistic rationale**
Ion release kinetics (F^-, Ca^{2+}, PO_4^{3-})	High initial burst; tapering over time	Moderate, steady	Sustained, balanced over time	Nano ↑ surface area → fast diffusion; submicron slows depletion; hybrid couples both for reservoir behavior.
pH neutralization trajectory	Prolonged acidity, slower rise to neutral	Faster neutralization	Fastest return to physiological pH	Hybrid improves buffering and matrix stabilization while maintaining reactivity.
Remineralization potential (enamel hardness recovery)	Enhanced, but sometimes limited by extended acidity	Moderate	Highest (synergistic ion supply + near-neutral pH)	Continuous F^- + Ca^{2+}/PO_4^{3-} support fluorapatite formation and mineral redeposition.
Matrix/structural stability	May be less stable (agglomeration, early solubility)	Stable	Stable with reactivity	Submicron framework reduces dissolution;

				nano provides active sites.
Biological compatibility (pH-mediated)	Risk of pulpal irritation if acidity persists	Favorable	Most favorable (rapid neutral pH)	Short acidic phase reduces tissue stress, supports cell tolerance (ISO 10993).
Translational/clinical signal (retention, margins)	Variable surface integrity	Good	Best overall (marginal stability + chemistry)	Hybrid's dual-scale packing improves integrity while keeping bioactivity.

The continuous fluoride and calcium ion delivery is critical for inhibiting demineralization and promoting reprecipitation of apatite phases within subsurface enamel [22–25]. The fluoride ions act as catalysts for fluorapatite formation, while calcium and phosphate ions derived from the glass network participate in remineralization, creating a more acid-resistant surface [26,27]. The higher enamel hardness recovery in the hybrid group supports this mechanism, aligning with prior studies reporting synergistic remineralization in GICs containing bioactive glass or optimized filler distributions [28,29].

From a biological perspective, the pH trajectory plays a pivotal role in tissue compatibility. Materials that remain acidic for prolonged periods can induce pulpal irritation and impede mineral redeposition. The hybrid GIC's rapid neutralization to physiological pH implies more favorable conditions for cell viability and less post-operative sensitivity. This is consistent with the observed >90% hDPSC viability, which satisfies ISO 10993 criteria for non-cytotoxic dental biomaterials. The mechanism → ions → biology → clinical translation pathway map can be seen in Table 2.

Table 2

Mechanism → ions → biology → clinical translation (pathway map)

Mechanistic component	Process & materials science detail	Biological consequence	Clinical implication	Key refs
Nano-particle reactivity	High surface area accelerates acid–base reaction and early F^-/Ca^{2+} release	Rapid ion availability inhibits demineralization and seeds remineralization	Early cariostatic effect; monitor for transient acidity	[22–27]
Submicron framework	Larger particles stabilize matrix, reduce dissolution, sustain release	Maintains ion supply as lesion matures; supports apatite reprecipitation	Durable fissure sealing; fewer repeat interventions	[26–29]
Hybrid packing (nano+submicron)	Bimodal packing ↑ density, ↓ porosity; couples fast diffusion with reservoir behavior	Synergistic F^- + Ca^{2+}/PO_4^{3-} delivery at near-neutral pH → higher enamel hardness recovery	Optimized marginal stability + bioactivity; translationally scalable	[22–29]

pH trajectory control	Short acidic phase, faster neutralization to physiological range	Improved tissue compatibility, less post-op sensitivity; supports cell viability (ISO 10993)	Safer in pediatric indications; better patient comfort	ISO 10993; [22–27]
Remineralization endpoint	Fluoride catalyzes fluorapatite; Ca/PO_4 rebuild subsurface enamel	More acid-resistant surface, higher microhardness	Lower secondary caries risk; longer sealant service life	[22–29]

Clinically, the in situ retention findings corroborate the laboratory results, showing that the optimized hybrid composition not only performs chemically but also ensures marginal stability and surface integrity under oral conditions. This dual validation highlights the translational potential of particle-size engineering as a scalable and low-cost strategy to enhance the therapeutic efficacy of conventional restorative materials without altering their fundamental chemistry.

Conclusion. The study provides compelling evidence that manipulating the particle-size distribution of glass ionomer cements fundamentally reshapes their biofunctional profile. The hybrid formulation combining nano- and submicron-sized particles achieved the most desirable synergy between sustained ion release, accelerated pH neutralization, and enhanced remineralization potential, while maintaining high cytocompatibility and short-term clinical stability. This finding positions hybrid GICs as promising candidates for bioactive restorations in minimally invasive pediatric and preventive dentistry.

Future research should expand toward long-term in vivo trials assessing secondary caries resistance and microleakage, while integrating molecular assays to elucidate signaling pathways involved in cell–material interactions. The incorporation of controlled-release nanophases or peptide-functionalized fillers could further amplify the regenerative capacity of next-generation glass ionomers.

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