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Comparative evaluation of beautisealant, wave flowable composite, SDR flow+ and conventional flowable composite: SEM-EDS analysis

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Abstract:

Enamel demineralisation at the enamel-composite interface remains a concern with conventional restorative materials under cariogenic conditions. Therefore, it is of interest to compare the remineralisation potential of four restorative materials at the enamel-composite interface using SEM and EDS. Forty extracted premolars (n=10/group) underwent restoration and 14-day pH cycling, with calcium and phosphorus measured at baseline, day 1, day 7 and day 14. BeautiSealant showed the highest remineralisation, followed by SDR Flow+, Wave Flowable Composite and conventional flowable composite. SEM showed dense apatite deposition with BeautiSealant, uniform mineralisation with SDR Flow+, partial deposition with Wave Flowable Composite and erosion in the control. Thus, data shows the use of bioactive ion-releasing restoratives for enamel remineralisation and advance evidence for their clinical application under cariogenic conditions.

Keywords: Remineralisation, BeautiSealant, S-PRG fillers, SDR Flow+, pH cycling, scanning electron microscope (SEM), energy-dispersive x-ray spectroscopy (EDS), bioactive composite, enamel-composite interface, calcium phosphate

Background:

Dental caries remains one of the most prevalent chronic diseases globally and is particularly burdensome in developing nations such as India, where pit and fissure surfaces of posterior teeth are most commonly affected [1]. The caries process is governed by the dynamic balance between demineralisation and remineralisation at the tooth-biofilm interface and when these balances shifts toward net mineral loss, subsurface enamel lesions either progress toward cavitation or are reversed by adequate ion availability [2]. Conventional composite resins while providing excellent aesthetics and mechanical performance are biologically inert and do not promote remineralisation of adjacent enamel. Marginal caries at the tooth-restoration junction remains the primary mode of restoration failure, particularly in cariogenic oral environments [3]. The development of bioactive, ion-releasing composites capable of delivering calcium, phosphate and fluoride ions to the surrounding enamel represents a significant clinical advancement. Sustained ion release and recharge capability are particularly desirable for long-term protection of the enamel-composite interface [4]. Giomer technology, exemplified by BeautiSealant (Shofu), utilises Surface Pre-Reacted Glass (S-PRG) fillers embedded within a resin matrix. S-PRG fillers release multiple bioactive ions including fluoride, strontium, aluminium, silicate, sodium and borate through an acid-base reaction mechanism. This multi-ion release confers remineralising, acid-buffering and antibacterial properties while combining the aesthetics and mechanical benefits of composite resins with therapeutic bioactivity [5]. SDR Flow+ (Dentsply Sirona) is a bulk-fill flowable composite featuring stress-decreasing resin technology and Wave Flowable Composite (SDI) incorporates fluoride-releasing components. Comparative evaluation of these systems under controlled pH cycling conditions using SEM and EDS analysis is warranted to establish evidence-based material selection criteria in minimally invasive

restorative dentistry [6]. PH cycling models simulate the alternating acid and neutral conditions of the oral environment and are widely accepted for *in vitro* evaluation of remineralisation potential [7]. EDS provides quantitative elemental analysis of calcium and phosphorus, enabling precise measurement of mineral changes at the enamel interface, while SEM allows morphological assessment of surface and crystal changes [8]. Therefore, it is of interest to report these four materials under standardised conditions and provide quantitative, time-point-specific evidence of their remineralising capability.

Materials and Methods:

Study design, ethical approval and specimens:

This *in vitro* experimental study was conducted at the Department of Conservative Dentistry and Endodontics, Santosh Dental College and Hospital, Ghaziabad, following Institutional Ethics Committee approval. Forty freshly extracted non-carious, non-hypoplastic human premolars were selected. Roots were sectioned at the cemento-enamel junction and crowns were embedded in self-cure acrylic resin leaving the occlusal surface exposed. All surfaces except the occlusal window were coated with acid-resistant nail varnish. Specimens were cleaned with pumice slurry, stored in 0.1% thymol solution and transferred to distilled water prior to use.

Artificial demineralisation:

Artificial enamel lesions were created by immersing specimens in a demineralising solution (2.2 mM CaCl_2 , 2.2 mM NaH_2PO_4 , 0.05 M acetic acid, pH 4.5, adjusted with NaOH) at 37°C for 96 hours, simulating early subsurface caries.

Group allocation and material application:

Specimens were randomly allocated to four groups (n = 10 each): Group I - Conventional flowable composite (control, Prime);

Group II - BeautiSealant (Shofu); Group III - Wave Flowable Composite (SDI); Group IV - SDR Flow+ Bulk Fill (Dentsply Sirona). Occlusal enamel surfaces were etched with 37% phosphoric acid gel for 20 seconds rinsed for 20 seconds and gently air-dried. Respective bonding agents and materials were applied per manufacturer instructions and light-cured at approximately 1200 mW/cm² for 20–40 seconds under standardised conditions.

PH cycling regimen:

All specimens underwent a 14-day pH cycling regimen at 37°C: 3 hours daily in demineralising solution (pH 4.5: 2.2 mM CaCl₂, 2.2 mM NaH₂PO₄, 0.05 M acetic acid) followed by 21 hours in remineralising solution (pH 7.0: 1.5 mM CaCl₂, 0.9 mM NaH₂PO₄, 0.15 M KCl). Fresh solutions were prepared daily.

SEM and EDS analysis:

Following pH cycling, specimens were sectioned buccolingually, dehydrated through graded ethanol, gold sputter-coated and examined by SEM at ×500, ×1000 and ×1500 magnification for surface morphology, crystal deposition and interfacial changes. EDS was performed at baseline, day 1, day 7 and day 14 to record weight percentages of calcium (Ca) and phosphorus (P) and calculate Ca/P ratios at the enamel-composite interface using point analysis, line scanning and elemental mapping.

Statistical analysis:

Data were analysed using SPSS version 26.0. Descriptive statistics were expressed as mean ± SD. Intergroup comparisons at each time point and intragroup comparisons across time points were conducted using one-way ANOVA with Tukey post-hoc testing or Kruskal–Wallis test as appropriate. A p-value < 0.05 was considered statistically significant.

Results:

Baseline Ca and P values were homogeneous across all groups, confirming equal mineral status prior to pH cycling: Ca (Group I: 36.42±1.21%; Group II: 36.38±1.18%; Group III: 36.45±1.25%; Group IV: 36.41±1.19%) and P (Group I: 17.82±0.62%; Group II: 17.79±0.58%; Group III: 17.85±0.60%; Group IV: 17.81±0.57%). Ca/P ratios were stable within physiologic range across groups

(approximately 2.04–2.05). Baseline SEM confirmed smooth, intact enamel surfaces in all groups (**Table 1**). Following 1 day of pH cycling, Group I showed an early calcium decrease to 35.12±1.30%, while bioactive groups maintained or increased mineral levels (Group II: 36.95±1.15%; Group III: 36.28±1.22%; Group IV: 36.54±1.18%). Phosphorus paralleled calcium trends (Group I: 17.05±0.66%; Group II: 17.98±0.54%; Group IV: 17.74±0.56%; Group III: 17.62±0.59%). Ca/P ratios remained similar across all groups at day 1 (~2.06), indicating that absolute mineral content rather than ratio is the more sensitive early indicator. By day 7, divergence was substantial. Group I continued to lose calcium (34.26±1.34%) and phosphorus (16.48±0.70%), while all bioactive groups showed net mineral gain. BeautiSealant achieved the highest calcium (38.72±1.10%) and phosphorus (18.84±0.52%). SDR Flow+ recorded calcium 37.88±1.12% and phosphorus 18.39±0.53%; Wave Flowable showed calcium 37.46±1.15% and phosphorus 18.21±0.55%. The Ca/P ratio in Group I raised to 2.08±0.07, indicating disproportionate phosphorus loss, while bioactive groups maintained stable ratios (~2.06). At day 14, the remineralisation hierarchy was clearly established (**Table 2**). Group I declined to calcium 33.58±1.42% and phosphorus 15.94±0.74%, with an abnormally elevated Ca/P ratio of 2.11±0.08. BeautiSealant achieved calcium 40.86±1.08% and phosphorus 19.92±0.48% with a stable Ca/P of 2.05±0.04. SDR Flow+ reached calcium 39.68±1.10% and phosphorus 19.28±0.50%; Wave Flowable showed calcium 39.24±1.14% and phosphorus 19.06±0.52%. SEM evaluation confirmed the EDS quantitative hierarchy. Group I showed progressive surface deterioration with visible porosities, erosion of enamel rod architecture and absence of organised crystal formation by day 14. BeautiSealant (Group II) exhibited a dense continuous mineral layer with well-formed apatite crystals throughout the cycling period. SDR Flow+ (Group IV) showed uniform mineral coverage with compact crystal clusters. Wave Flowable Composite (Group III) demonstrated partial mineral deposition with irregular crystal morphology. These SEM features are summarised in **Table 3**. Integrating quantitative EDS findings and SEM observations, the overall remineralisation efficacy ranked as: BeautiSealant (Rank 1) > SDR Flow+ (Rank 2) > Wave Flowable Composite (Rank 3) > Conventional composite (Rank 4).

Table 1: Baseline calcium and phosphorus content (%) by group

Group	Calcium % (Mean±SD)	Phosphorus % (Mean±SD)	Ca/P Ratio (Mean±SD)
I (Control)	36.42±1.21	17.82±0.62	2.04±0.05
II (BeautiSealant)	36.38±1.18	17.79±0.58	2.05±0.04
III (Wave Flowable)	36.45±1.25	17.85±0.60	2.04±0.06
IV (SDR Flow+)	36.41±1.19	17.81±0.57	2.04±0.05

All groups homogeneous at baseline (p > 0.05, one-way ANOVA)

Table 2: Calcium and phosphorus content (%) after 14 days of pH cycling

Group	Ca % (Day 14)	P % (Day 14)	Ca/P Ratio	Net Ca Change (%)	Net P Change (%)
I (Control)	33.58±1.42	15.94±0.74	2.11±0.08	-2.84	-1.88
II (BeautiSealant)	40.86±1.08	19.92±0.48	2.05±0.04	+4.48	+2.13
III (Wave Flowable)	39.24±1.14	19.06±0.52	2.06±0.05	+2.79	+1.21
IV (SDR Flow+)	39.68±1.10	19.28±0.50	2.06±0.04	+3.27	+1.47

Ca/P ratios in Groups II–IV reflect physiologic apatite stoichiometry (1.67–2.0); elevated Ca/P in Group I indicates disproportionate phosphorus loss

Table 3: SEM crystal morphology and surface features by group

Group	SEM Crystal Pattern	Interfacial Features
I (Control)	Disrupted enamel rods	Erosion, porosities, no organised crystals
II (BeautiSealant)	Well-formed apatite crystals	Dense, continuous mineral layer
III (Wave Flowable)	Irregular crystal deposits	Partial mineral deposition
IV (SDR Flow+)	Compact crystal clusters	Uniform mineral coverage

SEM assessed at ×500, ×1000 and ×1500 magnification

Discussion:

The principal finding of this study was that BeautiSealant showed superior enamel remineralisation potential compared to all tested materials across the 14-day pH cycling period, as evidenced by the highest net calcium gain (+4.48%), net phosphorus gain (+2.13%), stable physiologic Ca/P ratios and SEM evidence of dense apatite crystal formation. This aligns with the multi-ion release mechanism of S-PRG fillers, which release fluoride, strontium, aluminium, silicate, sodium and borate ions in a sustained and pH-responsive manner [9]. Bioactive composites capable of releasing and recharging calcium and phosphate ions have consistently showed superior remineralisation outcomes compared to conventional inert resins in both *in vitro* and laboratory models. The superior performance of BeautiSealant over SDR Flow+ and Wave Flowable Composite reflects the distinct ion-delivery mechanisms of these materials. SDR Flow+ achieved the second-highest remineralisation rank, consistent with its fluoride-releasing formulation providing early mineral protection [10]. Wave Flowable Composite showed intermediate mineral recovery. The conventional composite showed progressive mineral loss at all time points (net Ca −2.84%; net P −1.88%), confirming that inert materials without bioactive fillers cannot counteract acid-driven demineralisation and act solely as passive barriers. These findings are consistent with prior work demonstrating that calcium-phosphate releasing restorative materials significantly reduce lesion depth compared to conventional materials [11]. The pH-responsive nature of ion release from bioactive restorative materials is a clinically relevant property. Acidic environments generated during demineralisation episodes trigger enhanced calcium and phosphate release from bioactive fillers, providing a self-reinforcing protective mechanism precisely when the tooth is most at risk. This dynamic ion release, confirmed through EDS measurements showing greater mineral deposition in bioactive groups at each cyclic time point, supports the concept that these materials function as active remineralising depots rather than passive barriers [12]. Antibacterial bioactive composites combining NACP with antimicrobial monomers have further extended this principle by simultaneously reducing cariogenic biofilm activity and providing sustained ion release, demonstrating that multifunctional bioactive composites offer a dual-action strategy against secondary caries [13]. The Ca/P ratio findings provide important mechanistic insight. The physiologically stable Ca/P ratios maintained in bioactive groups (2.05–2.06) throughout 14 days of pH cycling indicate coordinated, stoichiometrically balanced deposition of calcium and phosphate consistent with hydroxyapatite formation [14]. In contrast, the progressively elevated Ca/P ratio in Group I (2.11 by day 14) reflects disproportionate phosphorus loss and stoichiometric imbalance characteristic of an active

demineralisation-dominant process. Studies on CPP-ACP combined with other ion-releasing systems have similarly showed that true biomimetic remineralisation requires concurrent and proportional delivery of both calcium and phosphate ions [15]. The adhesive interface can act as a barrier limiting ion diffusion from underlying bioactive materials and optimisation of adhesive system compatibility with ion-releasing composites remains an important consideration in clinical application. SEM findings corroborated the quantitative EDS data. The well-formed apatite crystals and dense mineral layer observed with BeautiSealant, compact crystal clusters with SDR Flow+ and partial deposition with Wave Flowable are consistent with the remineralisation hierarchy established by EDS. Prior studies employing calcium phosphate nanoparticle formulations have confirmed superior dentinal tubule occlusion and surface integrity compared to conventional fluoride-only products, further supporting the superiority of multi-ion bioactive systems [16]. The progressive erosion and disrupted enamel rod architecture observed in the control group aligns with established observations from pH cycling studies in the literature and validates the effectiveness of the pH cycling model used [17]. This study has several limitations. The *in vitro* design does not replicate the full complexity of the intraoral environment, including salivary pellicle, temperature variation, masticatory forces and diverse microbiome. A sample size of n=10 per group, while adequate for primary comparisons, limits subgroup analytical power. A 14-day pH cycling period, although widely adopted, may not fully capture long-term ion release and recharge behaviour. Additionally, only macroscopic mineral changes were assessed; nanoscale characterisation of crystal structure and subsurface mineral density warrants investigation in future work.

Conclusion

Bioactive restorative materials promoted greater enamel mineral recovery than conventional flowable composite under pH cycling conditions. BeautiSealant showed the highest remineralisation potential, followed by SDR Flow+, Wave Flowable Composite and conventional composite. Further long-term *in vivo* studies are needed to confirm these findings and establish their clinical relevance.

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Conflict of Interest: The authors declare no conflict of interest.

Data Availability: Data are available from the corresponding author on reasonable request.

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