RESEARCH PAPER

Comparative Evaluation of Diode Laser, Novamin, and Active Biosilicate Technology for Dentinal Tubule Occlusion: An In Vitro Scanning Electron Microscope (SEM) and Energy Dispersive X-Ray Analysis (EDX) Study

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Abstract

Background: Throughout the years, various management approaches have been explored to address dentine hypersensitivity. Recently, a tricalcium silicate-based cement has gained popularity as a “Dentine substitute” due to its mineralization properties, sealing dentinal tubules, and preserving pulp vitality. This potential can be harnessed for obliterating the tubular structure in dentine for managing dentine hypersensitivity. This comparative study investigates the efficacy of 810 nm diode laser, Novamin, and Active Biosilicate Technology on dentine tubule occlusion.

Methods: This in vitro, single-blinded research was conducted on thirty prepared dentine sections. The samples were allocated in three treatment groups: Group A - 810nm diode laser; Group B – Novamin paste; and Group C – Active Biosilicate Technology. The samples were observed for ‘Full Open’ and ‘Partial Open’ dentinal tubules using a Scanning Electron Microscope (SEM) combined with energy-dispersive X-ray (EDX) spectroscopy.

Results: Active Biosilicate Technology showed the fewest fully and partially open tubules at all magnifications. ANOVA revealed significant intergroup differences at ×5,000 ($F = 7.56$, $p = 0.011$) and ×10,000 ($F = 8.63$, $p = 0.001$). Tukey’s test confirmed greater tubule occlusion in Groups B and C than in Group A ($p < 0.05$), with no difference between B and C. EDX showed higher calcium and phosphorus deposition in Group C.

Conclusion: Active Biosilicate technology demonstrated the most effective and consistent dentine tubule occlusion, followed by Novamin and diode laser. The superior sealing ability of Active Biosilicate Technology suggests its strong potential as a desensitizing agent in clinical practice, offering a reliable option for the long-term management of dentine hypersensitivity.

Keywords: Dentin Sensitivity, Tooth Demineralization, Dentin Desensitizing Agents, Lasers, Microscopy.

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Introduction: Dentine Hypersensitivity (DH) is a widespread oral health issue characterised by sharp, transient pain arising from an exposed dentine surface induced by factors such as variation in temperature, air exposure, physical contact, osmotic shifts, or chemical agents. This condition is distinct from other dental diseases and significantly impacts the quality of life, limiting the consumption of hot, cold, sweet, or acidic foods and beverages.[1] Despite the availability of desensitizing agents, achieving long-term relief remains a clinical challenge. Many commonly used agents provide only temporary relief requiring repeated applications. This highlights the need for more effective and lasting solutions.[2]

Among the most promising long-lasting approaches for in-office treatment of managing DH is the occlusion of dentinal tubules either by melting and re-solidifying the dentine surface or remineralising or deposition of minerals on the exposed tubular dentine.[3] Two of the most efficient agents are Novamin paste, which releases calcium and phosphate ions, forming a hydroxycarbonate apatite layer[4] and a new agent popularly known as a dentine substitute, a tricalcium silicate-based cement with Active Biosilicate Technology, commercially available as Biodentine® that creates a dense, mineralized barrier over the dentine surface, effectively sealing exposed dentinal tubules and limiting fluid movement—an essential factor in managing dentine hypersensitivity[5].

While these treatments show promise, comparative studies among the three different modalities, with precise evaluation methods such as Scanning Electron

Microscopy (SEM), remain limited. This research aims to evaluate the comparative efficacy of diode laser, Novamin, and Active Biosilicate Technology in sealing dentinal tubules using SEM and Energy Dispersive X-ray analysis.

Materials and Methods: The comparative, in vitro, single blind research was carried out on 30 randomly selected extracted teeth. The teeth were randomly collected from the Outpatient section of the Department of Oral and Maxillofacial Surgery. Teeth with root caries, fractured roots, and teeth with root surface caries or external resorption were excluded. The sample size (n = 30) was selected based on sample size justification, ensuring adequate statistical power for intergroup comparison.

Teeth were rinsed with distilled water, root planed with Gracey curettes, and then stored in Hank's Balanced Salt Solution. Sections were prepared following Beaumont’s Protocol for sectioning human dentine.[6] The teeth were decoronated, and the roots were sectioned using a diamond disc bur to obtain 2-millimetre-thick dentine segments. Surfaces were finished with silicon carbide paper, etched with 17% Ethylenediaminetetraacetic acid for 40 minutes, and rinsed for 2 minutes to expose dentinal tubules.

The dentine samples, after preparation, were randomly assigned to three experimental groups:

Group A - Diode laser treatment on dentine surfaces of 810 nanometres wavelength at 1 Watt in continuous mode for 15 seconds, with a 1centimeter^2 spot size and an energy density of 15 Joules/centimeter^2.

Group B - Treatment with Novamin paste (927 parts per million fluoride), which was applied on dentine surfaces using a cotton pellet for 3 minutes.

Group C - Treatment with Active Biosilicate Technology (Biodentine®, a tricalcium cement-based material). Following the manufacturer’s guidelines, five drops of liquid were combined with the powder present in the capsule and then triturated in an amalgamator at 4000 rotations per minute for 30 seconds, resulting in a smooth, creamy paste. The prepared paste was left on the dentine surface for 60 seconds.

Samples were preserved in 2.5% glutaraldehyde prepared in 0.1 M Hanks Basic Salt solution for 24 hours, washed, and dehydrated in a graded alcohol series (25%-100%) for 10 minutes. Samples were mounted on 1 cm^2 SEM stubs, air-dried for 48 hours, and sputter-coated with 30-40 nm of gold using an ion sputtering device (JEOL, JEC-3000FC). Samples were examined under Scanning Electron Microscope (JEOL 7610F) at 40 kV. Each sample was scanned at ×2,000,000, ×5,000, and ×10,000 magnifications to classify dentinal tubules as ‘full open’ or ‘partial open’. The same dentine specimens were subsequently examined under EDX analysis. Samples will be examined at 20 kilovolts with a 5 nanometer spot size and a 300 second counting time to qualitatively detect elements including Calcium (Ca), Phosphorus(P), Potassium(K), and Oxygen(O).

All data were statistically analysed using IBM SPSS (Statistical Package for Social Sciences) software (Version 20). The results were expressed as Mean ± Standard Error of the Mean. To compare the degree of dentinal tubule occlusion among the three experimental groups, a one-way analysis of variance (ANOVA) was performed. Where significant differences were found, Tukey’s post hoc test (q value) was applied to determine pairwise comparisons between the groups. A p-value of less than 0.05 was considered to indicate statistical significance.

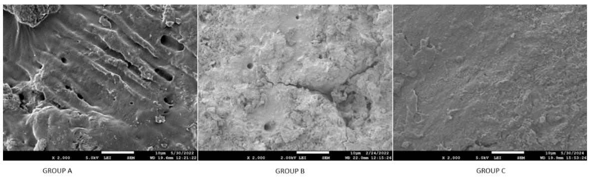

Figure 1: SEM photomicrograph of Group A (810 nm Diode laser), Group B (Novamin Paste), and Group C (Active Biosilicate technology) samples showing Full open dentinal tubules and partial open dentinal tubules at ×2,000 magnification, internal scale bar = 10 µm. SEM – Scanning electron microscope.

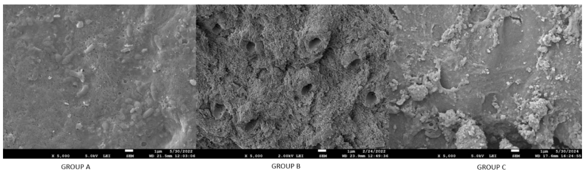

Figure 2: SEM photomicrograph of Group A (810 nm Diode laser), Group B (Novamin Paste), and Group C (Active Biosilicate technology) samples showing Full open dentinal tubules and partial open dentinal tubules, respectively, at ×5,000 magnification, internal scale bar = 1 µm. SEM – Scanning electron microscope.

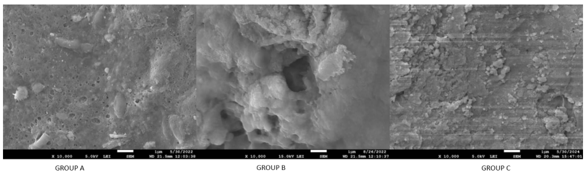

Figure 3: SEM photomicrograph of Group A (810 nm Diode laser), Group B (Novamin Paste), and Group C (Active Biosilicate technology) samples

showing Full open dentinal tubules and partial open dentinal tubules, respectively, at ×10,000 magnification internal scale bar = 1 µm. SEM – Scanning electron microscope.

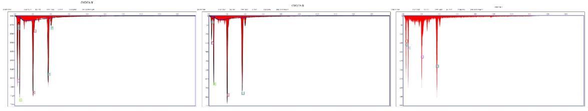

Figure 4: EDX analysis of Group A (Diode laser), Group B (Novamin), and Group C (Active Biosilicate Technology) samples, showing elemental composition on treated dentine surfaces. EDX – Energy-dispersive X-ray.

Results: SEM photomicrographs were obtained at ×2,000, ×5,000, and ×10,000 magnifications at 40 kV (Figures 5-7). The mean number of fully open dentinal tubules decreased with increasing magnification. ANOVA showed no significant difference among groups at ×2,000 (F = 5.03, P = 0.001), but significant differences were found at ×5,000 (F = 7.56, P = 0.011) and ×10,000 (F = 8.63, P = 0.001) (Table 1).

On inter-group comparison, the Tukey test showed a similar (P > 0.05) mean number of fully open dentinal tubules between the three groups at magnification ×2,000 (Table 2).

At ×5,000 and ×10,00 magnifications, Groups B and C showed significantly fewer full open tubules than Group A (p < 0.05 or p < 0.01), with no significant difference between Groups B and C (p > 0.05)

ANOVA revealed significant differences in the mean number of partially open tubules at ×2,000 (F = 10.97, p = 0.0001) and ×5,000 (F = 8.82, p = 0.0012), but not at ×10,000 (F = 2.77, p = 0.079). Mean ± SE values for all groups at each magnification were compared using ANOVA (Table 3).

Tukey's test showed significantly fewer partially open tubules in Groups B and C than Group A at ×2,000 and ×5,000 (p < 0.05 or p < 0.001), with no difference between Groups B and C (p > 0.05). At ×10,000, no significant differences were observed among the groups (p > 0.05) (Table 4).

EDX analysed elements were Carbon, Calcium, Phosphorus, Potassium, and Oxygen (Figure 9). Among the groups, Oxygen had the highest weight (%) and Phosphorus the lowest (Table 5).

Table 1: Surface Characteristics at Different SEM Magnifications

Group	**Number of Samples (n)**	**Magnification ×2000 (Mean ± SD)**	**Magnification ×5000 (Mean ± SD)**	**Magnification ×10000 (Mean ± SD)**	**p-value**
A	10	3.60 ± 0.58	7.10 ± 1.56	1.7 ± 0.6	0.01
B	10	2.20 ± 0.39	3.10 ± 0.55	0.8 ± 0.1	0.002
C	10	1.70 ± 0.30	1.40 ± 0.31	0.4 ± 0.1	0.001

Table 2: Intergroup Comparison at Different SEM Magnifications (Post-hoc Test)

Magnification	**Comparison**	**Mean Difference**	**q value**	**p-value**	**95% Confidence Interval of Difference**
×2000	Group A vs. Group B	-1.4	0.46	P > 0.05	-2.94 to 0.08
	Group A vs. Group C	-1.9	0.55	P < 0.05	-3.44 to 0.013
	Group B vs. Group C	-0.5	0.18	P > 0.05	-2.04 to 0.703
×5000	Group A vs. Group B	-1.4	0.46	P > 0.05	-2.399 to 0.201
	Group A vs. Group C	-1.9	0.55	P < 0.05	-0.999 to 1.199
	Group B vs. Group C	-0.5	0.18	P > 0.05	-1.646 to 0.154
×10000	Group A vs. Group B	-0.90	2.13	P < 0.05	-1.6454 to -0.1546
	Group A vs. Group C	-1.20	2.83	P < 0.01	-1.9454 to -0.4546

	Group B vs. Group C	-0.30	0.71	P > 0.05	-1.0454 to 0.4454

Table 3: Post-hoc Intergroup Comparison at Different SEM Magnifications

Magnification	Comparison	Mean Difference	q value	p-value	95% Confidence Interval of the Difference
×2000	Group A vs. Group B	3.7	4.11	P < 0.05	-5.9823 to -1.4177
	Group A vs. Group C	5.9	6.55	P < 0.001	-8.1823 to -3.6177
	Group B vs. Group C	2.2	2.44	P < 0.05	-4.4823 to 0.0823
×5000	Group A vs. Group B	-2.01	2.10	P < 0.05	-3.527 to -0.494
	Group A vs. Group C	-2.41	2.45	P < 0.05	-3.927 to -0.894
	Group B vs. Group C	-0.40	0.41	P > 0.05	-1.916 to 1.116
×10000	Group A vs. Group B	-0.80	-2.68	P > 0.05	-1.91 to 0.31
	Group A vs. Group C	-0.10	-3.35	P > 0.05	-2.11 to 0.11
	Group A vs. Group C	-0.10	-3.35	P > 0.05	-2.11 to 0.11

Table 4. Number of Partial-Open Dentinal Tubules of Three Groups at Different Magnifications

Group	Number of Samples (n)	Magnification ×2000 (Mean ± SD)	Magnification ×5000 (Mean ± SD)	Magnification ×10000 (Mean ± SD)
A	10	7.10 ± 1.30	3.11 ± 0.63	1.4 ± 0.45
B	10	3.40 ± 0.72	1.10 ± 0.28	0.6 ± 0.22
C	10	1.20 ± 0.47	0.70 ± 0.33	0.4 ± 0.22
p-value		**0.0001**	**0.0012**	**0.079**

Table 5. Distribution of Element Weight (%) Among Three Groups

Element	Group A (%)	Group B (%)	Group C (%)
Carbon / Potassium	**15.76**	**35.81**	**0.54**
Oxygen / Potassium	**52.17**	**43.74**	**48.83**
Phosphorus / Potassium	**12.40**	**8.10**	**13.04**
Calcium / Potassium	**19.67**	**12.36**	**37.59**

Discussion: Dentine hypersensitivity (DH) is mediated by the fluid movement within dentinal tubules, which is detected by odontoblastic processes and nerve fibres at the dentine–pulp interface. Dentine's capacity to respond to both normal and harmful stimuli is enabled by the presence of tiny tubules occupied by odontoblastic extensions, as well as the formation of the dentine-pulp interface.[7]

Therefore, any intervention aimed at reducing DH must either reduce dentine permeability by occluding tubules or modulate neural activity to diminish pulpal response. The present study compared three desensitising approaches, that is, Diode laser, Novamin, and Active

Biosilicate technology, to determine their effectiveness in promoting dentinal tubule occlusion through morphological and elemental analysis.[8]

Group A (diode laser) showed partial occlusion with surface melting and recrystallization (Figure 2). SEM images revealed irregular surface changes, with a noticeable number of fully open tubules across all magnifications. This suggests that while the laser induces thermal modifications, complete sealing is limited. The observed surface melting may be attributed to heat-induced denaturation of organic components, leading to narrowing of tubules rather than comprehensive occlusion. This may be due to heat-induced protein coagulation and tubule sealing, consistent with findings by Matys et al. (2013), who reported tubule narrowing or closure with 810 nm diode laser without structural damage.[9]

The mean number of Full open dentinal tubules at ×2,000, ×5,000, and ×10,000 magnification of the Diode laser was maximum among the groups (Table 1). These results are comparable with the research conducted by Patil CL et al. in 2020,[10] in which the diode laser group had a mean open dentine tubule area of 55.6 ± 5.34 as compared to other groups. In Group A, ×2,000 magnification revealed a large number of full open tubules, indicating limited desensitization compared to Groups B and C.

EDX analysis in this study showed a higher percentage of oxygen in diode laser-treated dentine (Table 5; Figure 4). Similar effects were observed with other lasers, where irradiation induces superficial oxidation, enhancing acid resistance and structural stability.[11] While this chemical modification may enhance durability, it does not compensate for the inadequate degree of physical occlusion, warranting further investigation into optimizing diode laser parameters to improve clinical outcomes.

Novamin demonstrated a higher degree of tubule occlusion compared to the diode laser, as shown by the reduced number of fully open tubules across all magnifications. This is consistent with the established mechanism of action of bioactive glass, which releases calcium and phosphate ions in the presence of saliva. These ions precipitate as a hydroxycarbonate apatite (HCA) layer, which integrates with the dentine surface and reduces dentine permeability.[12]

The mean number of dentinal tubules full open for Group B at ×2000, ×5,000, and ×10,000 magnifications is given in Table 1. However, the mean number of partial open dentinal tubules presented in Table 3 is significantly higher than full open dentinal tubules. These findings were comparable with the research by Kakodkar G in 2013, in which it was observed that the treatment resulted in a marked decrease in open tubules, with a greater proportion of tubules being partially or completely occluded.[13]

Novamin demonstrated greater dentinal tubule occlusion than the diode laser across all magnifications in the study. Similar findings were reported by Srivastava et al. (2024), where Novamin-treated samples showed significantly fewer fully and partially open tubules under SEM analysis, along with higher calcium and phosphate deposition as determined by EDX analysis. This supports the effectiveness of Novamin in forming a hydroxyapatite-like layer that seals tubules and reduces hypersensitivity.[14]

Group C, treated with Active Biosilicate technology exhibited the most extensive and uniform occlusion among all groups. The SEM image of Group C shows a mineral-rich layer with visible granular formations, indicating the formation of a reactionary layer due to the precipitation of calcium hydroxide (Figures 1-3). Calcium silicate particles react with phosphate ions in saliva, forming hydroxyapatite that chemically bonds to dentine. This layer mimics natural dentine, providing durable tubular occlusion.[15]

Active Biosilicate technology promotes early mineralization by enhancing the expression of Transforming Growth Factor-β1 from odontogenic cells of the pulp following application. It also functions by encouraging odontoblast activity and cellular differentiation, leading to the formation of reactionary and tertiary dentine.[7]

In a separate study involving tricalcium silicate cement, findings demonstrated that it can drive transformed murine Odontogenic Progenitor Cells to differentiate into odontoblast-like cells and initiate biomineralization.[16] This process supports dentine regeneration and effectively seals dentinal tubules through its bioactive properties.

Group C consistently demonstrated significant impermeability compared to Groups A and B at all magnifications. (Table 1 and Table 3).

Group C's superior occlusion capability is consistent with Kuru E et al. (2023), which observed tubule sealing by tag-like structures, indicating intratubular mineralization.[17]

EDX analysis of Group C revealed wt.% of Ca/K highest in Group C. Gjorgievska et al. (2011) noted that tricalcium silicate cements forms a bioactive layer, restoring enamel integrity.[18] (Table 5; Figure 1).

The EDX analysis of samples treated with Active Biosilicate Technology showed a notably low carbon content of 0.54% by weight, consistent with its predominantly inorganic composition of tricalcium silicate, calcium carbonate, and zirconium oxide.[19] The hydration reaction forms calcium silicate hydrate and calcium hydroxide, limiting carbon incorporation.[16]

This finding aligns with Camilleri and Grech et al, [16,20] who reported minimal carbon content in calcium silicate-based materials due to their inorganic nature. Proper sample preparation and high-vacuum conditions during EDX analysis may have further reduced surface hydrocarbons. These characteristics contribute to the biocompatibility of the Active Biosilicate technology and effectiveness in dentine regeneration, reducing hypersensitivity.

One limitation of this study is the lack of assessment of the depth of penetration of the agents into the dentinal tubules. Additionally, the durability or resistance of the occlusion achieved was not evaluated. Future research should include in vivo or long-term clinical trials, larger sample sizes, and methods such as confocal microscopy or micro-CT to determine penetration depth. Evaluating abrasion resistance, ageing behaviour, and durability under pH cycling will help validate the long-term performance of these desensitising agents.

Conclusions: SEM analysis confirms that Active Biosilicate Technology is effective in providing long-term relief from dentine hypersensitivity by promoting the occlusion of dentinal tubules. Future studies with larger sample sizes, standardised sample preparation, and extended observation periods are recommended to validate these findings. Additionally, clinical trials correlating SEM observations with patient-reported outcomes would help establish the long-term clinical efficacy of this technology.

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