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Incorporating of Nano-Calcium Silicate into Kavitan Plus and its effect on the Surface Microhardness: In-Vitro Study

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Abstract

Kavitan Plus is a conventional glass ionomer-based restorative material. This study aimed to evaluate the effect of adding silica dioxide (Ca_2SiO_4) nanoparticles at two concentrations, 5% and 10%, to Kavitan Plus on surface hardness using a Shore D surface hardness tester. Methods: Twenty-four samples were prepared and divided into three groups (8 per group): Group 1: control group; Group 2: 5% Ca_2SiO_4 ; Group 3: 10% Ca_2SiO_4 . Surface hardness was measured, and the data were statistically analyzed using one-way analysis of variance (ANOVA) and Fisher's one-way analysis of variance (LSD)

Results:

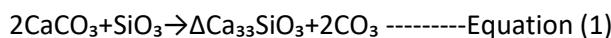
Statistically significant differences were observed between all groups ($F=94.523$, $p<0.001$). This indicates that the addition of dicalcium silicate nanoparticles (C_2S NPs) at different concentrations significantly affected the studied microhardness property of conventional glass ionomer cement (GIC) cements using Shore D. G1 and G2 recorded similar average values (45.42 and 45.23, respectively), while the 10% C_2S nanoparticle group (G3) showed significantly lower average values. This suggests that the addition of 10% nanoparticles reduced the property value, reducing mechanical integrity and homogeneity at higher addition ratios [1,2,3,14].

Conclusion:

The addition of calcium silicate nanoparticles significantly enhanced the surface hardness, depending on the concentration, indicating potential benefits for improving restoration durability and periodontal health.

Keywords: Kavitan Plus, calcium silicate nanoparticles, microhardness, Shore D, periodontal health.

around the The concept of this study is novel and recently explored. It involves adding silica nanoparticles, which we prepared, to a conventional cement ionomer class [19,20]. We used the cement, a commercially available conventional biocomposite, Cavitan Plus (Markova 238, 506 01 Užiny, Czech Republic). We prepared the nanomaterial at the University of Kufa, College of Science and Engineering, Department of Materials (Nano). The material is composed of calcium disilicate (Ca_2SiO_4) nanoparticles using a high-temperature physical process from calcium carbonate (CaCO_3) and silicon dioxide (SiO_2), following a solid-state reaction according to the following equation:



The following method and equipment were used: a mortar and pestle or ball mill, a high-temperature furnace ($\geq 1200^\circ\text{C}$), alumina or ceramic crucibles, a high-energy ball mill, and grease to prevent particles from rising to the surface. These materials must remain at the bottom of the grinding cylinder until completely ground by the balls to ensure nanoparticle formation. The final result is nanoparticles with a size of (28.43622) nm, as shown in Table 1.

Dental restorative materials must withstand mechanical stress, chemical reactions, and bacterial activity. Glass ionomer cements (GICs) are widely used in restorative dentistry due to their effective properties, such as chemical adhesion to tooth structure, biocompatibility, fluoride stability, and corrosion resistance [1, 7]. These properties make them particularly useful in Class V

restorations, where non-carious cervical lesions and root caries are common. Cavitan Plus is a type of glass ionomer-based restorative material that offers several advantages, such as chemical bonding and fluoride release, but has limited mechanical strength. Reinforcement with nanoparticles, particularly bioactive nano-calcium silicate, may improve hardness and bioactivity. Calcium silicate can release Ca^{2+} and Si^{4+} ions, which promote mineral deposition and durability [2, 6]. A previous study demonstrated improved bioactivity and surface properties of modified Cavitan formulations. The study investigates the effect of 5% and 10% nano- Ca_2SiO_4 , as well as the micro-hardness of Cavitan Plus, and their potential clinical effects on periodontal health [1,6,7].

Materials and Methods

Twenty-four samples of Kavitan Plus were divided into three groups, each containing eight samples: the control group (without additives), the second group (5% dicalcium silicate nanoparticles), and the third group (10% dicalcium silicate nanoparticles). The powders were mixed homogeneously and placed in molds (10 mm in diameter and 2.5 mm in height), then allowed to dry. After curing, all samples were stored in deionized water at 37°C for 24 hours. A Shore D microhardness test was performed using a standardized hardness scale. Statistical analysis was performed using one-way analysis of variance (ANOVA) and Fisher's LSD test, with a significance value of $p < 0.05$ [4,5,14].



Fig.1 Three Groups of Samples

The nanoparticles were mixed with the glass ionomer using a cement spatula on a glass plate to ensure even distribution of the nanoparticles. The samples were covered on both sides with nylon strips and compressed to obtain a smooth, ridge-free, and homogeneous surface in all directions.

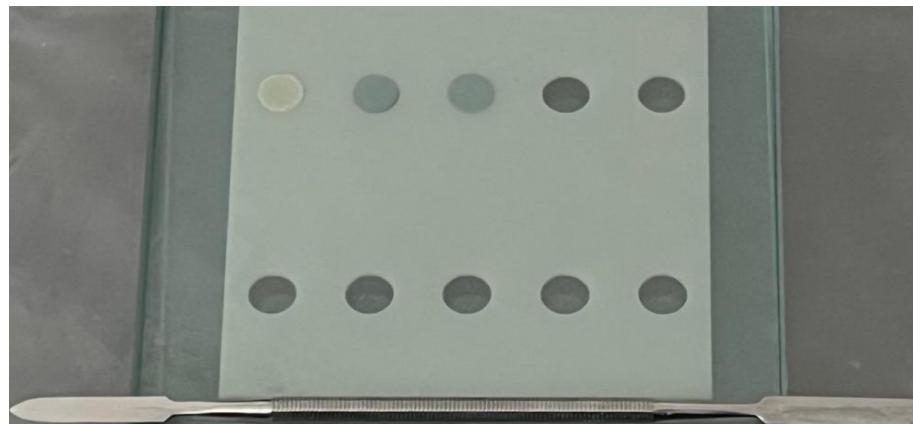


Fig. 2 The sample making mold is 2.5 x 10 mm in size and the mold is covered with a nylon cover from the bottom and top with a spagola cement tool.



Fig. 3 Shows final form of samples

Characterization of Dicalcium silicate:

A powder diffraction system (AL-2700B X-ray diffractometer - Manufacturer: Danang Oolong Radiation Instrument Group Co., Ltd., Shanghai, China) was used to generate the X-ray diffraction pattern, with parameters 2θ (20-80°), a minimum 2θ step size of 0.001, and a wavelength (λ) of 1.54614°. The production of the prepared NCSP was confirmed using XRD. Philips

pw3040, Netherlands. The samples were also examined by FTIR, EDS, and SEM. A small amount of the GIC and Ca_2SiO_4 , were taken, and the chemical composition of the composite was confirmed by EDS, the cross-linking was confirmed by FTIR, and the nanoparticle sizes were obtained by SEM. It was observed that the nano-preparation process was successful through the above tests.

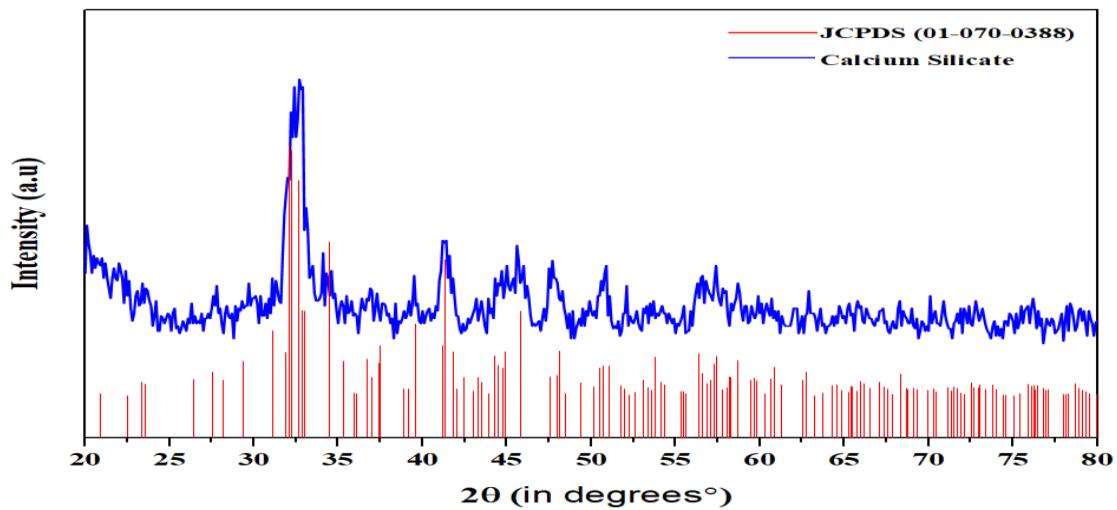


Fig.4 Shows XRD



Fig. 5 Shows device XRD

Table. 1: The preparation of dicalcium silicate nanoparticles is shown with the final particle size after preparation.

PEAK: 13-pts/Parabolic Filter, Threshold=0.3, Cutoff=1.5%, BG=3/1.0, Peak-Top=Summit									
2-Theta	d	BG	Height	I%	Area	I%	FWHM	D nm	ave(D nm)
22.065	4.0251	26	33	30.3	23	3.3	0.279	29.0103	28.43622
23.478	3.786	18	28	25.7	28	4	0.238	34.0924	
27.761	3.2109	14	27	24.8	29	4.1	0.19	43.0698	
30.037	2.9726	17	23	21.1	40	5.7	0.567	14.5066	
32.716	2.735	31	109	100	705	100	0.768	10.7805	

36.877	2.4354	17	26	23.9	21	3	0.198	42.2939	
39.526	2.2781	14	31	28.4	41	5.8	0.205	41.178	
41.382	2.1801	14	45	41.3	206	29.2	0.565	15.0301	
45.018	2.0121	23	35	32.1	99	14	0.701	12.2673	
45.987	1.9719	13	35	32.1	143	20.3	0.553	15.6057	
47.696	1.9052	12	37	33.9	162	23	0.551	15.7638	
50.87	1.7935	16	35	32.1	50	7.1	0.224	39.2721	
56.595	1.6249	19	31	28.4	59	8.4	0.418	21.5849	
73.209	1.2918	12	18	16.5	12	1.7	0.17	58.2123	
76.195	1.2484	12	22	20.2	35	5	0.298	33.8754	

Microhardness Measurement

The surface hardness of the specimens was calculated using a digital Shore tester Figure 6. The specimen surfaces were loaded with a weight of 5000 g for 10

seconds. Three scratches were found on the surface of each specimen, evenly distributed in a circle, and the average of the three readings was taken, at least 0.5 mm apart. The radial length of the scratches was measured using a calibrated integrating microscope.

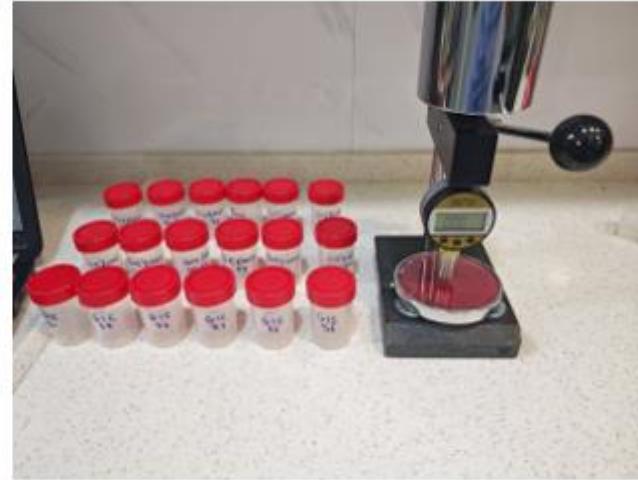


Fig. 6 shows the use of a Sure D device to measure microhardness.

Results

The mean "Shore D" hardness values were as follows: Group 1 (10%) = 39.2203, Group 2 (5%) = 45.2333, and Group 3 (control) = 45.4236. One-way ANOVA revealed significant differences among groups ($F=94.523$, $p=0.000$). Post hoc Fisher's LSD was confirmed all

Table. 2; Descriptive Statistics for Microhardness Shore D and percentages (5%, 10%) of silica nanoparticles added

to (GICc) using one-way analysis of variance

Descriptives			
Sh.D			
Groups	N	Mean	Std. Deviation
1.00	8	45.4236	.84799
2.00	8	45.2333	.92423
3.00	8	39.2203	1.25965
Total	24	43.2924	3.10154

Table. 3 Using one-way ANOVA for the percentages (5%, 10%) of Ca_2SiO_4 added to GICc

ANOVA		
Sh.D		
Between Groups	F	Sig.
	94.523	.000

Table:4 Post Hoc Pairwise Comparisons (Fisher's LSD) for Microhardness Shore D for the percentages (5%, 10%) of Ca_2SiO_4 added to GICc.

Multiple Comparisons		
Dependent Variable:		
LSD		
(I)	Groups	Sig.
1.00	2.00	.715
	3.00	.000
2.00	1.00	.715
	3.00	.000
3.00	1.00	.000
	2.00	.000

- The mean difference is significant at the 0.05 level.

The LSD table shows the two-tailed significance (p) for each pair of groups.

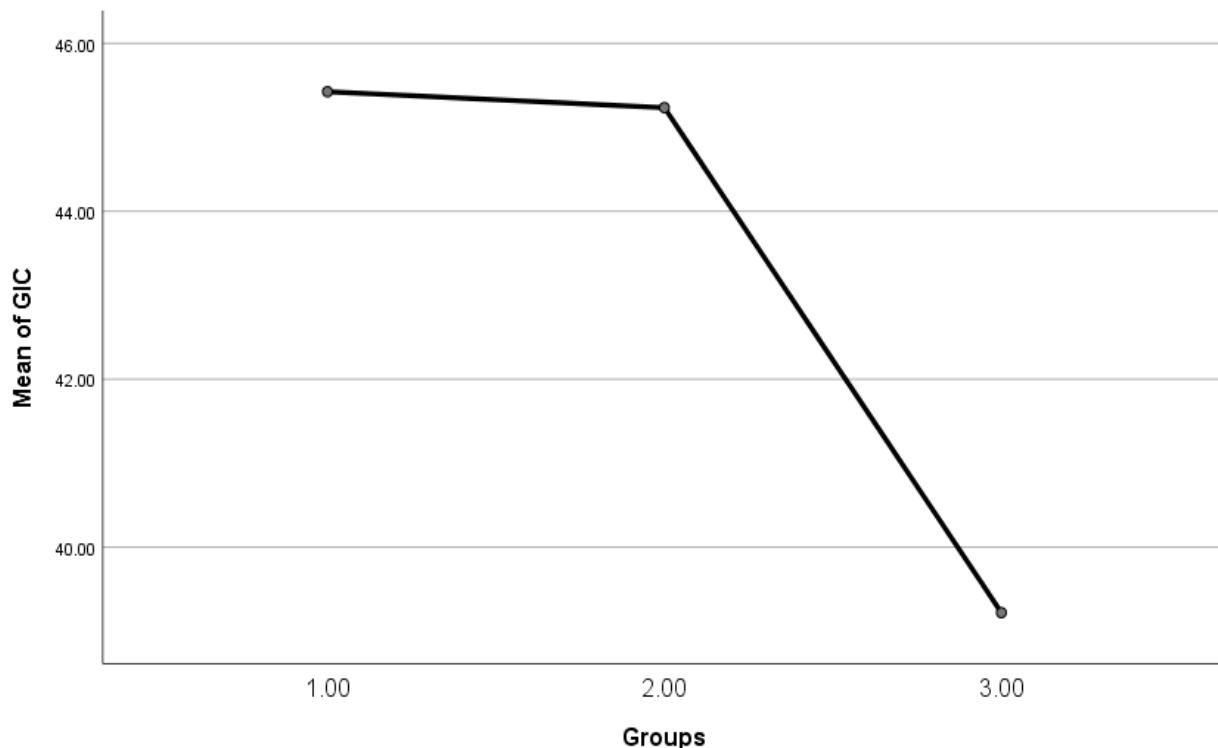


Fig.7 A chart showing the relationship between the GICc & Ca_2SiO_4 microhardness test.

Discussion:

However, the clinical use of conventional GICs is often limited due to their relatively poor mechanical properties, including low fracture toughness, wear resistance, and microhardness. This can lead to premature restoration failure, particularly in areas subject to high stresses or in patients at high caries risk. The observed improvement in their microhardness is attributed to several factors. First, the nanoparticles have a high surface area-to-volume ratio, allowing for more efficient and homogeneous distribution within the GIC matrix. This can result in a denser composite structure, acting as an effective filler that reinforces the polyacrylate matrix. Second, nC_2S molecules may participate in the acid-base reaction of GICs. The silicate ions of nC_2S are likely to react with polyacrylic acid, forming additional silicate-based polysalts that contribute to a stronger interconnected network [2,7]. The superior performance of the 10% nC_2S group compared to the 5% group suggests a dose-dependent strengthening effect. Higher filler loading generally results in a greater decrease in the volume fraction of the polymer matrix, which is typically the weakest phase in the composite. However, it is important to consider the existence of an optimal threshold for filler addition.

Exceeding this threshold can lead to particle agglomeration, which acts as stress concentration points and can compromise mechanical properties [8,9,15].

The results of analysis of variance (ANOVA) and LSD test for GIC modified with calcium silicate nanoparticles indicate that a one-way ANOVA test revealed a statistically significant difference between the three groups ($F = 94.523$, $p = 0.000$), indicating that the addition of calcium silicate nanoparticles (C_2S NPs) at different concentrations significantly affected the studied microhardness property of conventional glass ionomer cement (GIC) with the Shore D device.

Descriptive statistics also indicated that the control group (G1) and the group modified with 5% C_2S nanoparticles (G2) recorded similar values (45.42 and 45.23, respectively), while the 10% C_2S nanoparticle group (G3) showed significantly lower mean values (39.22). This suggests that the addition of 10% nanoparticles reduced the property value, possibly due to interference with the GIC matrix structure or particle agglomeration, which reduced mechanical integrity and homogeneity.

The LSD test also confirmed no significant difference between the control group and the 5% C_2S NP groups (p

= 0.715), while a very significant difference was found between these two groups and the 10% C₂S NP group (p = 0.000). Therefore, the optimal nanoparticle addition level appears to be 5%, with lower additions being better, as this preserves the original GIC properties without compromising performance.

The significant increase in microhardness is of clinical importance, particularly for its proposed application in Class V restorations for patients with periodontal disease [1,12]. The harder surface directly translates into better resistance to brushing and occlusal stresses, potentially extending the life of the restoration. This, combined with the expected bioactivity of silica dioxide nanoparticles, makes modified GIC a promising material for the management of root surface lesions in high-risk populations. The incorporation of nano-Ca₂SiO₄ significantly improved the surface hardness of Kavitan Plus. This improvement is attributed to the nanoparticles' ability to fill microcavities and ionic interactions within the matrix, enhancing structural density and stability. Calcium and silicon ions were also released, promoting secondary mineralization. Improving hardness increased abrasion and dissolution resistance, which is vital in oral conditions. Clinically, a harder surface may reduce bacterial adhesion and plaque retention, ultimately impacting indirect benefits for periodontal tissue. These findings are consistent with recent studies on bioactive fillers in restorative materials. However, further *in vivo* studies are needed to confirm these effects and evaluate long-term biocompatibility [17,18].

Conclusion:

Adding even small amounts of calcium dioxide nanoparticles (5%) can improve or maintain the desired properties of glass ionomer cements, while large and excessive additions of nanoparticles (10%) negatively impact their stability and mechanical properties [1,6,11].

Limitations of the Study:

One limitation of this study is that it is an *in vitro* study. The oral environment, with its dynamic conditions of pH cycling, temperature changes, and humidity, can differently affect the long-term performance of the material. Future studies should focus on evaluating the bioactivity (e.g., apatite formation capacity) and adhesion strength of this modified cement to dentin, especially root dentin.

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