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BASIC RESEARCH:

Comparison of Surface Microhardness of Portland Cement Associated with Niobium Oxide and Zirconium Nanoparticles with the Mineral Aggregate Trioxide

Comparación de la microdureza superficial del cemento portland asociado a óxido de niobio y nanopartículas de zirconio con el agregado mineral trióxido

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ABSTRACT: To determine the surface microhardness of white portland cement associated with niobium nanoparticles, white portland cement associated with zirconium nanoparticles, and mineral trioxide aggregate. The present study is an experimental *in-vitro* study. The sample consisted of 03 study groups. These were divided into 09 subgroups of 04 hours, 14 days and 28 days. The instrument used to record the surface mechanical microhardness was the Vickers microdurometer. The Shapiro-Wilk statistical analysis was then performed to identify the normality of the data. The Anova test was applied to compare between the three groups and then the Tukey test for multiple comparisons with a 95% confidence level. White Portland cement associated with zirconium nanoparticles had the highest hardness value ($p<0.05$), followed by white Portland cement associated with niobium nanoparticles and aggregate control cement of mineral trioxide. The lowest value of surface microhardness was obtained by the addition of mineral trioxide ($p<0.05$). Surface microhardness values were significantly higher at 28 days than at 04 hours for all groups evaluated. White Portland cement with/without nanoparticulate additives generated higher surface microhardness than the control group added mineral trioxide in the evaluation periods.

KEYWORDS: Surface microhardness; Compressive strength; MTA; Calcium silicate; Nanoparticles; Portland cement.

RESUMEN: Determinar la microdureza superficial de cemento portland blanco asociado a nanopartículas de niobio, cemento portland blanco asociado a nanopartículas de circonio y agregado de trióxido mineral. El presente estudio es un estudio experimental *in vitro*. La muestra estuvo compuesta por 03 grupos de estudio. Estos se dividieron en 09 subgrupos de 04 horas, 14 días y 28 días. El instrumento utilizado para registrar la microdureza mecánica de la superficie fue el microdurómetro Vickers. Luego se realizó el análisis estadístico Shapiro-Wilk para identificar la normalidad de los datos. Se aplicó la prueba de Anova para comparar entre los tres grupos y luego la prueba de Tukey para comparaciones múltiples con un nivel de confianza del 95%. El cemento Portland blanco asociado a nanopartículas de circonio tuvo el mayor valor de dureza ($p<0.05$), seguido del cemento Portland blanco asociado a nanopartículas de niobio y el cemento de control de agregados de trióxido mineral. El valor más bajo de microdureza superficial se obtuvo mediante la adición de trióxido mineral ($p<0,05$). Los valores de microdureza superficial fueron significativamente mayores a los 28 días que a las 04 horas para todos los grupos evaluados. El cemento Portland blanco con/sin aditivos nanoparticulados generó mayor microdureza superficial que el grupo control al que se le añadió trióxido mineral en los periodos de evaluación.

PALABRAS CLAVE: Microdureza superficial; Resistencia a la compresión; MTA; Silicato de calcio; Nanopartículas; Cemento portland.

INTRODUCTION

Calcium silicate (CSC)-based cements are widely used in different restorative and restorative clinical applications (1, 2). Included in this group are the aggregate of mineral trioxide (MTA) and Portland cement (CP) composed mainly of di-calcium and tri-calcium silicate, being hydraulic cements, which when mixed with distilled water produce mainly calcium hydroxide ($Ca(OH)_2$) and hydrated calcium silicate (CaOSiO2H2O); as a result, a colloidal gel of hydrated calcium silicate is formed that eventually solidifies into a Hard structure (3).

There is a great similarity in the physical-mechanical, chemical and biological properties between CP and MTA cement, which has been one of the most widely used in the clinical field of endodontic surgery, repair of furca perforations, apicoectomies and direct pulp capping (3, 4). However, PC has limitations, such as not exhibiting the radiopacity required to differentiate it from the surrounding anatomical structures and low physical-mechanical values (5).

MTA also has clinical limitations at the mechanical level, such as its difficult handling and long setting time, so that the surface mechanical microhardness values take a few days to reach the maximum level. A primary mechanical microhardness of 20N was found after mixing, increasing to 48N in periods of 28 days, due to the factor of its continuous setting of calcium silicate cements resulting in greater dimensional stability over time (5, 6).

In relation to the physical properties of MTA, it is known that it has low surface mechanical microhardness (6). This parameter is very important in some clinical conditions, such as the use of biomaterials in the repair of furcal restorations or apexogenesis of teeth with immature apices (7).

In general, many of these disadvantages have been associated with the presence of bismuth oxide (Bi2O3) (5, 6) so new particles are being tested in order to improve their properties. The type and size of the particle influence the properties of the cement. In commercial formulations, such as Biodentine and MTA Plus, which have particles smaller than 1 µm, a shorter setting time and better mechanical properties were found than with MTA (6, 7).

Some of the efforts to improve the properties of CSCs include the incorporation of some nanoparticles such as silver, zinc, niobium oxide (NiO2) and zirconium oxide (ZrO2) that have attracted attention in the field of dental materials (5, 7-9).

However, there is much controversy in the literature about the effect of heating on the physicochemical properties of hydraulic sealants, and the data are based solely on their chemical compositions and temperature ranges (8). Furthermore, there is no information on the relationship between thermomechanical compaction and hydraulic sealants in terms of the adaptation of the marginal space between dentinal walls and filling materials (8).

Niobium oxide (NiO2) is a particle widely used in biomedicine and dentistry for its integration into bone tissue (9, 11). It has been added in materials such as dental acrylic resin for its low toxicity, chemical stability, physical properties, and antibacterial activity (12-15). Adding 10% and 30% NiO2 nanoparticles to CP generated greater compressive strength, surface microhardness, and radiopacity; (16, 17) without affecting the pH, setting time and tooth coloration of calcium silicate cements (16).

On the other hand, zirconium oxide (ZrO2) was initially introduced as a biomaterial in orthopedic surgery due to its hardness, high density, and good wear resistance (12, 16). It is a bioactive, biocompatible, radiopacifier, alkaline pH material, releases calcium ions, exhibits antimicrobial activity, prevents pigmentation and reduces interference with physicochemical properties (17). It is used at the micro and nanoparticulate level in various studies at a concentration of 30%, which brings improvements in its mechanical properties, radiopacity and bioactive potential (10, 18).

The available studies by Tanomaru and Duarte on the use of Portland cement as a potential matrix in the area of biomaterials due to its chemical composition of dicalcium silicate, (12, 16) but on the incorporation of nanoparticulate agents of niobium oxide (NiO2) and zirconium oxide (ZrO2) are scarce in relation to their concentrations and particle size and their relationship with different properties; it is necessary to seek the optimization of these cements.

Therefore, the present study aims to evaluate the surface microhardness of white portland cement (CPB) associated with niobium oxide (NiO2) nanoparticles, white portland cement (CPB) associated with zirconium oxide (ZrO2) nanoparticles compared to MTA at 04 hours, 14 days and 28 days.

MATERIAL AND METHODS

The present *in vitro* study was reviewed and approved by the Institutional Ethics Committee of the Scientific University of the South with the approval code POS-53-2022-00164. The study was conducted using the experimental materials described in Table 1.

Table 1. Materials and study groups.

Group	Materials / Proportions	Factory
White Portland Cement. (CPB)	CP + 330 µL distilled water.	Huascarán, Compañía Minera de Agregados Calcáreos S.A., Lima, Peru.
White Portland Cement + Niobium Nanoparticles. (CPB + NiO2)	CPB 80% + NPs-NiO2 30% + 330 µL distilled water.	Huascarán, Compañía Minera de Agregados Calcáreos S.A.C, Lima, Peru (CPB) + Sigma Aldrich Ltd., St. Louis, Missouri, USA (NPs-NiO2).
White Portland Cement + Zirconium Nanoparticles. (CP + ZrO2)	CPB 80% + NPs-ZrO2 30% + 330 µL distilled water.	Huascarán, Compañía Minera de Agregados Calcáreos S.A.C Lima, Peru (CPB) + Sigma Aldrich Ltd., St. Louis, Missouri, USA (NPs-ZrO2).
MTA	MTA + 330 µL distilled water.	Angelus, Indústria de Produtos Odontológicos S.A., Londrina, Paraná, Brazil.

CPB: White Portland Cement; NPs: Nanoparticles; MTA: Mineral Trioxide Aggregate®.

SPECIMEN PREPARATION

The following experimental compounds were obtained: Portland cement by the company Huascarán, Comacsa-Peru. The nanoparticles of niobium oxide and zirconium oxide were acquired by Sigma Aldrich-USA. Cement control Mineral trioxide added by means of Angelus, Londrina-Brazil. Subsequently, the experimental compounds were prepared by weighing the compounds by means of a precision analytical balance and the association was carried out by means of the vortex-type agitator equipment (14, 17) in the High Technology Laboratory Certificate S.A.C. laboratories.

The experimental calcium silicate compounds were prepared and elaborated under the manufacturer's standards and authors' references (18, 21). The experimental groups were prepared in cylindrical polyethylene matrices (20) to obtain uniform specimens of 04 mm and 06 mm. The experimental samples were then removed from the matrices for verification of their dimensions by means of a digital kingfoot and surface polishing by the fine-grained polishing system, to remove the removal of splinters and roughness (21).

STORAGE OF EXPERIMENTAL SPECIMENS

Experimental calcium silicate specimens were stored in an incubator (Medical-Expo-USA) at a temperature of 28°C for homogeneous setting 16, 19, 20 in the evaluation periods of 04 hours, 14 days and 28 days.

SURFACE MECHANICAL MICROHARDNESS

The evaluation of the endodontic cylindrical blocks was carried out in three time periods of 4 hours, 14 days and 28 days, due to the continuous setting of the experimental cements reporting a greater stability over time 5, 16 of the 14 and 28 days. Therefore, the samples were placed individually by each group in HMV-2000 microdurometer with Vickers pyramidal diamond penetrator (Shimadzu Corporation, Japan), belonging to the High Technology S.A.C. mechanical testing laboratory.

Previously measured the areas and dimensions of the cylindrical blocks, they were placed in a vertical position, for the application of compressive forces by indentations, (17, 20) with a weight of 10 grams at a speed of 1 mm applied for 05 seconds.

Three readings were taken for each sample, recording the arithmetic mean as the accepted value of the surface microhardness averaged by the Vickers microdurometer program to determine the variation between the different groups in their respective evaluation times. The data were recorded specifying the study group, number of specimens and evaluation time, all this information in the data collection sheet and later in the Microsoft program Excel.

STATISTICAL ANALYSIS

The statistical analysis consisted of a univariate analysis obtaining means and standard deviation of the study groups. The Shapiro-Wilk test was then applied to identify the normality of the data. For the comparison of the surface microhardness of the three groups, the ANOVA statistical test was used, then for the multiple comparisons Tukey was applied with a confidence level of 95% by means of the IBM SPSS 21.0 program.

RESULTS

Table 2 presents the mean and standard deviation of the surface microhardness values of calcium silicate cements associated with NiO2 and ZrO2 nanoparticles. CPB + ZrO2 had the highest hardness value ($p<0.05$) followed by CPB + NiO2 and MTA control cement. The lowest value of surface microhardness ($p<0.05$) was observed by the MTA in the evaluation periods. Surface microhardness values were significantly higher at 28 days than at 04 hours for all groups evaluated.

Table 2. Comparison of surface microhardness values of calcium silicate cements.

Agent	Time	Microhardness
CPB	04 hours	22,881 ± 3.14(a)
	14 days	37,427 ± 3.14 (bc)
	28 days	42,338 ± 1.69 (cd)
CPB + ZrO2	04 hours	38,621 ± 1.83 (bc)
	14 days	56,127 ± 1.83 (f)
	28 days	64,038 ± 2.73 (g)
CPB + NiO2	04 hours	34,481 ± 2.42(b)
	14 days	45,527 ± 2.42 (de)
	28 days	49,849 ± 1.42 (e)
MTA	04 hours	35.82 ± 2.54 (b)
	14 days	49.83 ± 2.54 (e)
	28 days	55.83 ± 2.71 (f)

Mean ± standard deviation of the surface microhardness of calcium silicate cements.
*Different letters indicate significant differences between the groups evaluated ($p<0.05$).
Tukey's test ($p<0.05$): Statistically significant difference.

DISCUSSION

The effectiveness of calcium silicate cements such as MTA will depend on the ability to withstand high mechanical displacement loads, occlusal forces, and the placement of restorative materials on the material (1, 2). The objective of this study was to evaluate the surface microhardness of CPB, CPB associated with NiO2 nanoparticles, CPB associated with ZrO2 nanoparticles and MTA control cement.

All modified experimental silicate cements had higher compressive strength values compared to MTA. In the present study, experimental nanoparticulate cements were used. The CPB showed higher hardness values compared to the MTA at 04 hours and 28 days. Tanomaru *et al*. demonstrated the fundamental influence of particle size between 0-20 µm on material activity and strength development in early stages of setting (3). They highlight the preponderant role played by particles smaller than 20 µm in contributing to compressive strength, while particles between 30 and 45 µm generate longer setting times due to incomplete hydration of the particles (4). Bosso-Martelet al reported that particles larger than 40 µm affect water absorption and can affect the mechanical properties of surface microhardness and compressive strength (5).

The use of ZrO2 additive has wide applications in pharmaceuticals, pigments, cosmetics, and biomedical field for integration into bone tissue. ZrO2 is a metallic, semiconductor and extremely white oxide that is widely used for its mechanical performance, dielectric behavior, thermal properties, among others. They are considered non-toxic particles. Its structural properties allow it to be easily modified and gives it high stability in adverse conditions of temperature, humidity and pH (5, 20, 30).

The incorporation of ZrO2 into materials such as glass ionomer and acrylic resin has resulted in improved physical compressive strength properties. The ZrO2 nanoparticles associated with the experimental CPB cements generated the highest values of surface microhardness at 24 hours, 14 days and 28 days compared to all groups evaluated, this probably due to their particle size and presence of 30% ZrO2. These results were similar in mechanical properties to those of Bosso et al. who used 30% ZrO2 in combination with CPB and by Tanomaru *et al*. who incorporated 30% microparticulate ZrO2 into CPB, improving its compressive strength and solubility properties compared to MTA (3). Vazquez and Bosso *et al*. added 20% ZrO2 finding reduction in cement setting time, increased compressive strength, and flexural strength of the material (4,5). On the other hand, the addition of ZrO2 at 20% and 30% increased compressive strength properties, accelerated the hydration process, reduced setting time, presented good cytotoxicity response, and increased antimicrobial effectiveness against resistant root canal bacteria (9, 10, 20).

Regarding CPB+NiO2 30%, higher hardness values were obtained than MTA and CPB, but lower than CPB + ZrO2, at 14 days and 28 days. NiO2 is a radiopacifying agent used in 8% and 30% biomedical materials respectively. Providing alkaline pH, high compressive strength, antimicrobial activity, and biocompatibility (11, 25). Bosso *et al*. They found that the association of CPB with 30% NiO2 improved the mechanical properties of compressive strength, optical color properties, and radiopacity of the material compared to MTA.(12, 21) However, according to Tanomaru *et al*., the association of 30% NiO2 plus 10% OCA to CPB presented greater resistance than MTA, due to the presence of calcium oxide in its composition, this addition improved the hydration of the MTA Repair HP by accelerating its setting time (13).

In relation to CPB associated with nanoparticulate additives, it presented high values of compressive strength compared to MTA, probably due to the

particle size of CPB-based cements. According to Bosso Martelo *et al.*, the use of the combination of different metal additives at the microparticulate and nanoparticulate level can increase or decrease the physical, mechanical and biological properties dependent on concentration (5).

Therefore, there is little scientific evidence in the literature on the mechanical property of Vickers surface microhardness, for example the report by Dawood *et al.*, reports that adding 3% calcium phosphate additives to Portland cement and MTA can improve their results (31). On the other hand, Tanomaru *et al*. found that adding 10% OCa to the 10% study groups to Zr02+CPB and +NiO2+CPB can improve surface microhardness (32).

It is recommended that more studies be needed on the importance of white Peruvian portland cement for dental use, due to its great physical, mechanical and antimicrobial potential associated with new nanoparticulate enhancing agents according to the base study by Quea *et al*. (33) Villavicencio *et al*. (34). In addition, it would be important to initiate a line of research to be able to carry out by means of X-ray diffraction analysis for the characterization of comparative chemical compounds present in experimental calcium silicate cements.

The proposed calcium silicate (CP)-based materials with potential additives of strength and hardness may be a viable option to commercial cements such as MTA due to the improvements in their mechanical and physical properties and even thinking in the future about new combinations that can facilitate handling and setting time as well as decrease their solubility. It is necessary to study other physical, chemical and biological properties, as well as other accelerator compounds to improve the clinical performance of these materials.

CONCLUSION

CPB with/without nanoparticulate additives generated higher surface microhardness than the MTA control group in the evaluation periods. In addition, the CPB + ZrO2 experimental group presented the highest values of surface microhardness in the evaluation periods.

CONFLICT OF INTEREST

The authors declare that there is no conflict of interest.

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AUTHOR CONTRIBUTION STATEMENT

Conceptualización y diseño: A.E.P.S. and C.R.G.R.
Revisión de literatura: A.E.P.S. and C.R.G.R.
Metodología y validación: A.E.P.S. and C.R.G.R.
Análisis formal: A.E.P.S. and C.R.G.R.
Investigación y recopilación de datos: A.E.P.S. and C.R.G.R.
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