

**EVALUATING THE HARDNESS OF PORCELAIN BY ADDING DIFFERENT
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Article Received on 30/07/2021

Article Revised on 19/08/2021

Article Accepted on 07/09/2021

ABSTRACT

Aim: To investigate the vickers hardness of FELDSPATHIC porcelain BY adding silver nanoparticles and comparing it with non reinforced porcelain. **Material and method:** porcelain fused to metal discs were made using silver nanoparticles reinforced porcelain with different concentration of silver nanoparticles and vickers hardness was evaluated using Vickers hardness tester. **Results:** Silver nanoparticle reinforced ceramics showed higher Vickers hardness values. Statistical analysis used: one-way analysis of variance (ANOVA) with Post-Hoc Tukey's test. **Conclusion:** The addition of silver nanoparticles increased Vickers hardness values as compared to the non reinforced feldspathic porcelain.

KEYWORDS: Silver nanoparticles, porcelain, hardness.**INTRODUCTION**

As of today, ceramics are considered one of the most aesthetic materials used in dentistry. The word "ceramic" is derived from the Greek word "keramos" that translates to mean, "burnt earth." It came from the ancient art of pottery. The use of ceramics in dentistry dates back to 1789 when the first porcelain tooth material was patented by a French dentist (de Chemant).^[1] The literature defines ceramics as inorganic, non-metallic material made by man by the heating of raw minerals at high temperatures.^[2] It's most prized attribute is its ability to simulate natural tooth in colour and translucency. It is also biocompatible, inert in the oral environment and able to be formed into precise shapes.

In spite of these qualities, its negative property is its brittle nature, which is responsible for unpredictable and catastrophic failure. Ceramics are thought to have low tensile strength because of the presence of flaws especially the ones located on the surface that initiate and propagate fractures.^[3] Since they act as stress concentrators when the material is loaded. Therefore; improving porcelain's hardness is the key to increase its lifetime service in the oral cavity.^[7]

Many strengthening mechanisms have been developed to deal with this problem.^[5,6] Strengthening of ceramics can be accomplished by either of the two methods: first is development of residual compressive stresses within the surface of the material and second is interruption of crack propagation through the material.

Second method of strengthening porcelain can be done either by dispersion strengthening or transformation toughening. In dispersion strengthening ceramics are reinforced with dispersed phase which interfere with crack propagation through the ceramic material.

This can be accomplished by the addition of tougher filler particles, which absorb the energy from the crack and prevent its driving force propagation. Most of the newer generation of high strength ceramics is reinforced with tougher crystalline particles, which block crack propagation, thereby increasing fracture resistance. Some of the particles used are lithium disilicate ($\text{Li}_2\text{O} \cdot 2\text{SiO}_2$), alumina (Al_2O_3), magnesia – alumina spinel ($\text{MgO} \cdot \text{Al}_2\text{O}_3$), zirconia (ZrO_2).

One of the methods to strengthen the porcelain is the addition of ductile metal particles, which has been shown

to improve toughness through crack bridging and deflection by the metal particles.^[7,10] Keeping the above in mind, this study was undertaken to evaluate the

strengthening of porcelain by addition of different quantity of silver nanoparticles.

We hypothesised that addition of these will increase hardness of porcelain.



Figure 1: Silver Nanoparticles.

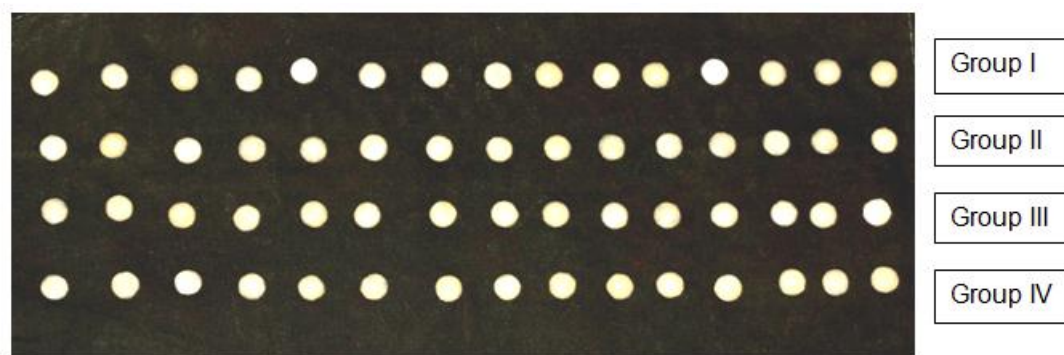


Figure 2: Ceramometal Discs.



Figure 3: Vickers Indenter.

MATERIALS AND METHODS

Method: steps followed during study.

The study was divided in the following steps:

- Making dispersion solution of different quantities of silver nanoparticles
- Fabrication of metal-ceramic discs(Fig2)
- Polishing of the ceramo-metal discs using Silicon Carbide paper.
- Inducing Vickers indents on the ceramo-metal discs using Vickershardness Tester.
- Determining the Vickers hardness of ceramic.

To obtain a uniform dispersionsolution of silver nanoparticles, silver particles (Figure 1) with an average

diameter of less than 100nm (SIGMA – ALDRICH, lot #MK BV 0729V)) were dispersed in purified water in the presence of the dispersion agent carboxymethyl cellulose (SIGMA, life science lot #BCBK4087V). The Concentration of silver in the solution was adjusted to 100 ppm, 200 ppm, 500 ppm.

To obtain metal disc a custom milled steel template with 30 standardized circular moulds(Figure5) was fabricated. Each inner mould was of the diameter $10\text{mm} \pm 1\text{mm}$ and depth $0.5\text{mm} \pm 0.1\text{mm}$. Sixty such wax patterns of standard dimensions for metal discs were fabricated and spruced. They were invested in phosphate bonded investment material. Standardized procedure was followed to invest all the wax patterns. The investment was allowed to set for one hour. After burn out in the wax burnout furnace the invested ring was placed in the induction casting machine at 1100°C . The metal used for casting was '4all' which is a nickel chromium alloy meant for metal-ceramics. The metal discs were then divested and sandblasted. Sprues were cut with the aid of diamond cutting discs and diamond wheels were used for finishing. Total sixty metal discs were fabricated. A digital calliperwas used to measure the thickness of each metal disc. Layering of the metal discs were done by using feldspathic porcelain IPS Classic; Ivoclar

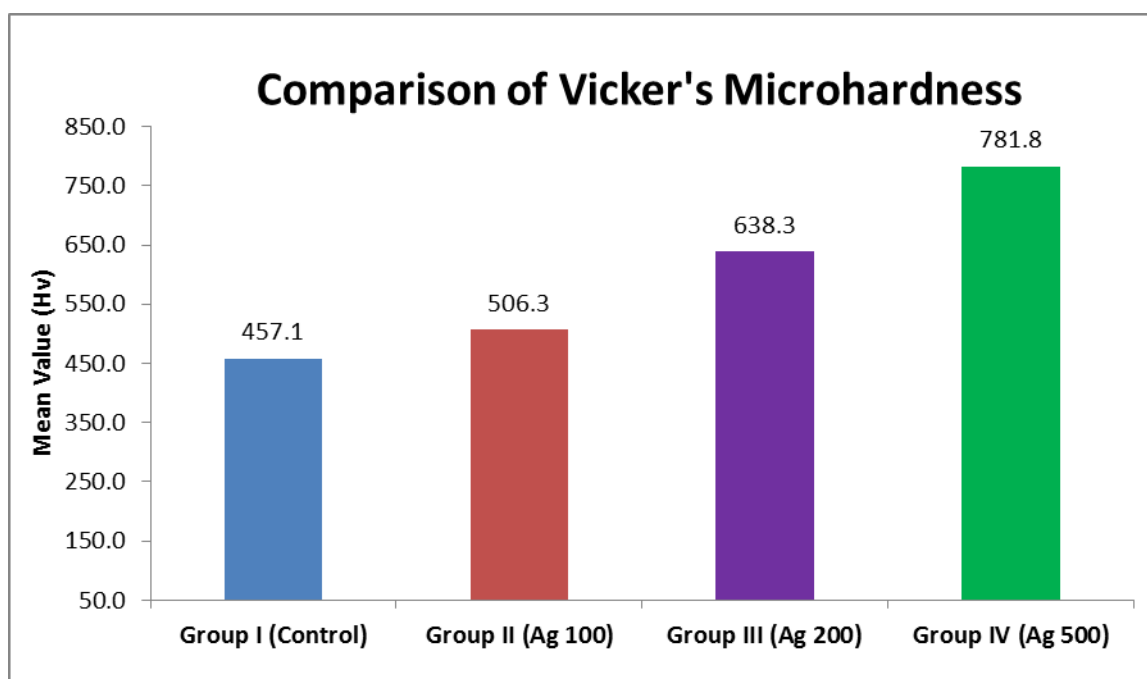
Vivadent. For group I, porcelain slurry was made using distilled water without inclusion of silver nanoparticles.

Dispersion solution of 100 ppm silver nanoparticles used for group II and 200 ppm silver nanoparticles for group III while dispersion solution 500 ppm used for group IV. Porcelain powder (2.1 g) was mixed with each silver nanoparticle-dispersed solution (0.75 g). Condensing the porcelain was done for all the discs to achieve the thickness of 1.5mm. Discs were fired according to the manufacturer's recommended firing schedule using a commercial porcelain furnace (DENTSPLY MultiMate NTpress). The firing of the specimens (drying for 5 minutes, rate of temperature increase: 50°C/minute, vacuum value: 730 mmHg, firing initiation temperature: 680°C, firing finishing temperature: 930°C, rapid cooling) was done in a porcelain furnace.

All 60 samples were first polished using SiC paper following a sequence of grit 220, 320, 400, 600, 800, 1000 and 1200 using a lapping machine (Metaserv, UK) to ensure reproducible control of the polishing procedure. All discs were then cleaned in an ultrasonic

bath for ten minutes. Discs were dried with tissue paper and Glaze liquid was applied evenly to all the discs with a sable brush and fired according to manufacturer's instructions in a commercial porcelain furnace. A Vickers hardness tester (Reichert Austria) was used to produce five Vickers indentations on each sample at widely separated locations. The load to be applied was determined using the trial and error method for each ceramic group. The load which was sufficiently high to produce indentation, but not so high that lateral chipping or crack would occur were chosen. Accordingly, a load of 100 g was applied to all specimens. The same setup of the Vickers hardness tester as mentioned above was used. For determining the micro hardness of the ceramic, the Vickers indenter was used with a load of 0.2 kg for 30 seconds.

This indent was then identified under the optical microscope at 200x magnification and the Image analysis software was used to measure the diagonals of the indent and used in the formula: $H = 1.8544 P / (2a)^2$ where P= peak load applied in grams, a= half diagonal of the indentation in microns.



Graph 1: The distribution of Micro hardness across four study groups (n=60).

A) Results For Vicker's Microhardness

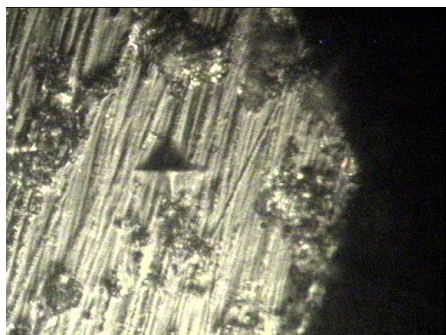
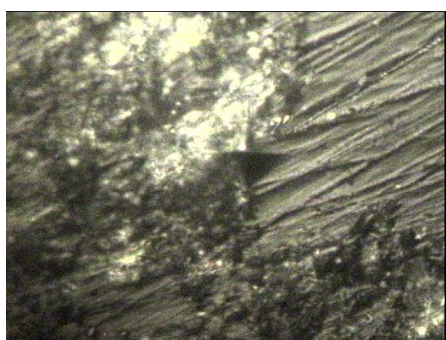
Vickers's micro hardness data obtained after testing of the specimens. The Vickers's hardness of each specimen was recorded. The mean micro hardness values (Graph 1) and the standard deviation for each group was calculated (Table 1).

The Ag 500 nm group (Group IV) had the highest micro hardness values of 781.8 Hv with a standard deviation of ± 9.9 . The second highest values were obtained for the Ag 200 nm specimens with a mean of 638.3 Hv and the standard deviation of ± 15.5 (Group III). This was

followed by the Ag 100 nm specimens with a mean of 506.3 Hv and the standard deviation of ± 4.0 (Group II). The control group without inclusion of silver Nano particles (Group I) had the least micro hardness values with mean 457.1 Hv and the standard deviation of ± 13.8 .

Table 1: The distribution of Micro hardness across four study groups (n=60).

Micro hardness (Hv)	Group I (n=15) (Control)	Group II (n=15) (Ag 100)	Group III (n=15) (Ag 200)	Group IV (n=15) (Ag 500)
Mean \pm SEM	457.1 \pm 13.8	506.3 \pm 4.0	638.3 \pm 15.5	781.8 \pm 9.9

**Figure 4(A): Group I.****Figure 4(B): Group II.****Figure 4(C): Group III.****Figure 4(D): Group IV.****Figure 5: Steel Template with wax Pattern.**

RESULTS

Vickers hardness of 457.1 Hv, 506.3 Hv, 638.3 Hv, 781.8 for group I (control), Group II (100Ag), group III (200 Ag) and group IV (500 Ag) respectively, with the P value of 0.021 between group I and II and P value of 0.001 between all other groups i.e. group I and III, group I and IV, group II and III, group II, IV and Group III and IV which are statistically significant.

DISCUSSION

Metal ceramic and veneered ceramic crowns are widely used in dentistry because of their excellent esthetics and mechanical durability. However, it is sometimes necessary to replace restorations because of fracture or chipping of the veneering porcelain. However, the metals that have been used to date are non-precious metals, which are harmful when used in the oral cavity. Furthermore, the materials contained in those metals have mainly been applied for structural applications, rather than dental applications. This study was undertaken to evaluate the strengthening of porcelain by addition of different quantity of 100 nm silver nanoparticles for testing its Vickers's Hardness and comparison of micro hardness with non-reinforced dental porcelain, to evaluate whether they could enhance the micro hardness of porcelain. The term hardness in mineralogy is the relative hardness of a substance and is based on its ability to resist scratching.^[1] In metallurgy, and in most other disciplines, the concept of hardness that is most generally accepted is the resistance to indentation.

The indentation produced on the surface of a material from an applied force of a sharp point or an abrasive particle results from the interaction of numerous properties among the properties that are related to the hardness of a material are compressive strength, proportional limit and ductility.^[1] There are several types of surface hardness tests. Most are based on the ability of the surface of a material to resist penetration by a diamond point or steel ball under a specified load. The

tests most frequently used in determining the hardness of dental materials are Barcol, Brinell, Rockwell, Shore, Vickers and Knoop. The Vickers hardness test is suitable for determining the hardness of brittle materials.^[1] In Vickers hardness test square diamond point is pressed under specified load into the polished surface of a material. HV is calculated by dividing of load by the projected area of indentation. The lengths of diagonals of the indentation are measured and averaged. Vickers hardness employ loads less than 9.8 N. The resulting indentations are small and are limited to depth of less than 19 μm .^[8] In the present study, Vickers hardness of porcelain with and without silver nanoparticles was evaluated using Vickers hardness tester (Saroj Industries, India). Specimens were made adding different quantities of silver nanoparticles of <100 nm (100ppm, 200ppm, 500 ppm) while control group did not contain particles. 15 samples were considered in each group. The results of the present study (Table 1) showed Vickers hardness of 457.1 Hv, 506.3 Hv, 638.3 Hv, 781.8 Hv for group I (control), Group II (100Ag), group III (200 Ag) and group IV (500 Ag) respectively, with the P value of 0.021 between group I and II and P value of 0.001 between all other groups i.e. group I and III, group I and IV, group II and III, group II, IV and Group III and IV which are statistically significant.

These findings are in general agreement with those of Mitsunori Uno and Masakazu Kurachi.^[10] who concluded that the addition of silver and platinum nanoparticles enhanced the mechanical properties of porcelain. Cherif A and Manal R^[9] investigated the effect of silver nanoparticles and silver hydroxyapatite nanoparticles on and fracture strength of dental ceramic.

Their observation is compatible with the results of our study. Tokushi *et al.*^[13] in (2012) evaluated the addition of nanoparticles of precious metals and found that those nanoparticles improved the mechanical properties by increasing its Young's modulus and fracture toughness of commercial porcelain.

Results proved that silver nanoparticle reinforced ceramics showed higher Vickers hardness values as compared to the non-reinforced feldspathic porcelain. The reason behind this may be that silver particles act as barriers in reinforced porcelain. These grain boundaries represent an obstacle to crack propagation. Thus cracks can only propagate through space between the grains in which the cracks detour the silver nanoparticles. As discussed by Dlouhy *et al.* "An effective crack or particle interaction may exist between the particles and the glass matrix if there is a perfect bond between both constituents". The inherent ductility of the metallic phase can be utilized to inhibit propagation of a running crack through the nanoparticles. In other words, a crack can be bridged by stretching nanoparticles. The crack bridging phenomenon occurs when the ductile metal acts to inhibit the extension of cracks, generally, the strength of composite porcelain

with particles dispersed in the glass matrix is enhanced by suppression of cracking through deflection.^[14]

According to Kon M *et al.*^[12] the strength of composite porcelains with particles dispersed in the glass matrix is enhanced by the suppression of cracking through deflection, bowing, and pinning mechanisms.^[10] Another possible mechanism is the generation of hydrostatic stress.^[19] in the glass matrix after the addition of metal nanoparticles. Hydrostatic stress is proportional to the difference between the coefficients of thermal expansion of the added metal and the porcelain matrix. Coefficients of thermal expansion of silver are $18.9 \times 10^{-6}/\text{K}$. With silver nanoparticles, greater differential thermal expansion occurs, generating compressive stress tangential to the Ag nanoparticles (if they remain in the metallic phase). This compressive stress remains in the vicinity of the Ag nanoparticles and inhibits cracks that propagate near them, since it causes cracks to deflect.^[20] This explains why the micro hardness of the reinforced ceramics (Group II, III and IV) was significantly higher than the ceramics without inclusion of silver nanoparticles (Group I).

Vickers hardness of porcelain increased as concentration of silver nanoparticles increased suggesting that it might be effective in inhibiting the mechanical failure of dental porcelains in the oral cavity. The Vickers hardness of sound enamel is 352.5 ± 13.8 .^[17] So, inclusion of 100 ppm of nanoparticles will be kinder towards opposing tooth enamel because it has Vickers hardness of 506.3 ± 4.0 closer to that of enamel.

According to our study, the recommended reinforcement would be with 100 ppm of silver nanoparticles, because this proved to have hardness closer to enamel which would be kinder to it and its non perceptible color change in porcelain. Though color change of group III specimens were not perceptible to eye, they cannot be advocated clinically because of relatively higher Vickers hardness than enamel.^[17]

Group IV samples have higher Vickers hardness but their color change after reinforcement was perceptible therefore its use is clinically not recommended.

CONCLUSION

1. All reinforced specimens showed increased Vickers hardness values.
2. Highest Vickers hardness value was seen in group IV specimens containing 500 ppm of silver Nano particles but lead to perceptible color change which is clinically not acceptable and further investigations are required for the same.
3. Specimens with inclusion of 100 ppm of silver Nanoparticles showed Vickers hardness of 506.3 which is closer to that of sound enamel.

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