Compression Strength of Metal-Ceramic Fixed Dental Prostheses Reinforced by Nanoparticle Silica from Rice Husks

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Abstract

Fracture occurred in metal-ceramic Fixed Dental Prostheses (FDPs) after being permanently cemented has been major concern in dentistry.

This study aims to evaluate the compression strength on metal-ceramic samples via the addition of nanoparticle silica (NPS) to opaque porcelain layers. Silica was synthesized via sol-gel method and sonicated for 270 minutes. The morphological and chemical characteristics of the silica were observed using SEM, TEM and XRF analysis. The NPS powder was mixed into the commercial opaque-porcelain powder with variation of 0.5; 1; 1.5; 2; 2.5; 3 wt % and as a control. Co-Cr bimetals prepared in 28 samples were used in the study divided into seven groups (n=4 each). The veneering of opaque-porcelain-NPS and commercial dentine on metals was sintered at 920-950°C. All specimens were tested for the compression strength using a universal testing machine. The XRF analysis confirmed the presence of silica, SEM and TEM showed amorphous NPS with spherical and agglomerated to form clusters.

There was a statistically significant difference of compression strength among samples, and the most significant improvements was in 0.5 wt% addition ($p<0.05$). Nanoparticle silica synthesized from rice husks via combination of sonication/sol-gel method in a small percentage would improve compression on the metal-ceramic samples.

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Introduction

Metal-ceramic Fixed Dental prostheses (FDPs) are indicated to replace edentulous in the region of anterior and posterior due to the strength of metal-coping and porcelain layers to achieve aesthetic result[1]. However, there are some reports related to the fracture, even though cementation has been applied permanently in the abutment teeth[2,3].

Opaque, dentine and enamel layers are three porcelain layers that have attached on the metal copings to form metal-ceramic FDPs. The opaque layer contains metal oxides, and it is the first layer that directly interact to the coping[4], thus; atomic migration could be occurred in the first layer, leading to an improvement in mechanical properties[5]. Thus, the opaque-porcelain layer is designed to have certain amounts of silica that would withstand forces of the metal-ceramic FDPs[6].

The silica is commonly found in rice husks and it has been reported to have amorphous structures[7]. In Indonesia, the production of the rice husks is abundant which have not been benefited optimally[8]. The utilization of nanoparticle silica (NPS) as reinforcement in dental material has been developed into resin composite material, and it has been reported to improve mechanical properties[9]. Furthermore, Sinamo and team have suggested that silica particles from rice husks are recommendable as

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reinforcer to improve flexural capacity of metal-ceramic restorations[10]. The purpose of this study is to investigate the compression strength of metal-ceramic samples reinforced by NPS within the opaque-porcelain layer. In this study, our null hypothetical statement suggests no significant differences in the compressive strength of opaque-porcelain layers with the presence of NPS.

Materials and methods

Research Methodologies

Samples and Materials

Rice husks that were used in this study were rice husks from Ciherang types. NaOH 1N, opaque-porcelain powder, dentine, and glazing were purchased from Vita VMK Master, Zahnfabrik, Germany. The bi-metals Co-Cr was produced and afforded from Bego, Germany.

Synthesis of Nanoparticle Silica (NPS) from rice husks

The procedure of NPS syntheses were carried out via a combination of sol-gel and sonication methods. As many as 200 g of rice husks were immersed in 1N HCl at 90°C for an hour, and then this sample was dried in an oven for 24 hours at 80°C. The dried husks were then immersed with NaOH 1 N for an hour at 80°C, subsequently; a cloud solution was formed, indicating the formation of sodium silicate. Afterward, HCl 1N was poured wisely to form gel, and this gelatinous-contained-solution was centrifugated to separate the liquid and gel phases at 4000 rpm for 5 minutes. After being separated, the gelatinous phase was placed into an oven for 24 hours at 80°C to form white-colour powder. This amount of powder was then placed into a sonication for 270 minutes with ultrasonic homogenizer at 40% of amplitude and 12 kHz to obtain smaller and fine powder [9,11,12].

The preparation of NPS and commercial opaque-porcelain mixture powder

The NPS powder from the previous procedure was mixed with Vita VMK Master opaque-porcelain powder. The mixture was divided into seven mixing groups, including 0.5; 1; 1.5; 2; 2.5; 3 wt% and control group (without NPS). The mixing process was carried out via grinding in a mortar until homogenous. Each mixing group consisted of four samples as followed by Federer:

$$(t-1)(r-1)>15 \quad \text{(eq.1)}$$

The preparation of metal-ceramic samples

A bi-metal Co-Cr with dimension of 25 x 3 x 0.5 mm^3 was prepared for 28 pieces. These samples were oxidized at temperature of 980°C, and then these were coated with opaque-porcelain/NPS and dentine layers that were 8 x 3 x 0.2 mm^3 and 8 x 3 x 0.9 mm^3 respectively in manual technique. Then, the veneered-oxidized metals were condensed and sintered at 920-950°C in a furnace to form metal-ceramic samples.

Characterization

Characterization of NPS from rice husks

The morphological and chemical characteristics of NPS were confirmed via Scanning Electron Microscope (SEM), Transmission Electron Microscope (TEM), and X-Ray Fluorescence (XRF) analysis.

Compression strength measurement

The metal-ceramic samples were placed on the surface of a metal spherical container with a diameter of 30 mm and a thickness of 10 mm. The surface of the porcelain layer was faced-up[13]. The load body with plate shape with 8 mm of length (in accordance with the length of porcelain layers) was connected to the UTM (Tensilon) was applied perpendicular to the surface of porcelain layer, with the average speed was 1 mm.min-1 until fractures occurred due to the compression forces.

Statistical Analysis

The One-Way Anova was selected to determine the differences of compression strength among the metal-ceramic samples with variations and control. The Post Hoc Multiple Comparison Games-Howell test was performed to investigate which mixture that had significant improvement of compression strengths. The software that was used in this study is SPSS program version 26.

Results

Characterization of NPS from rice husks

XRF Analysis

Based on Table 1 below, the percentage of silica (SiO_2) contents before being synthesized was 82.20%, and after being isolated, the content was 92.90%.

Substances	Rice husks (%)	NPS (%)
SiO_2	82.20	92.90
P_2O_5	1.80	-
K_2O	3.08	-
CaO	1.50	0.34
V_2O_5	0.34	-
Cr_2O_3	1.0	0.42
MnO	7.3	2.50
ZrO_2	0.71	-
BaO	2.00	1.10
Nd_2O_3	-	2.90

Table 1. Silica content in rice husks and nanoparticles silica.

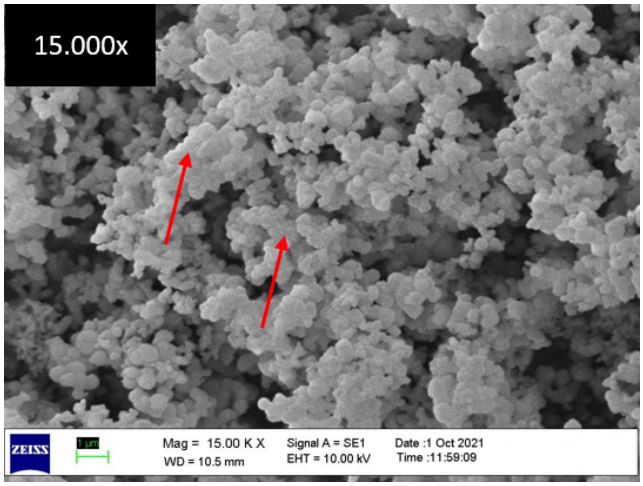

Figure 1. SEM micrographs of NPS from rice husks.

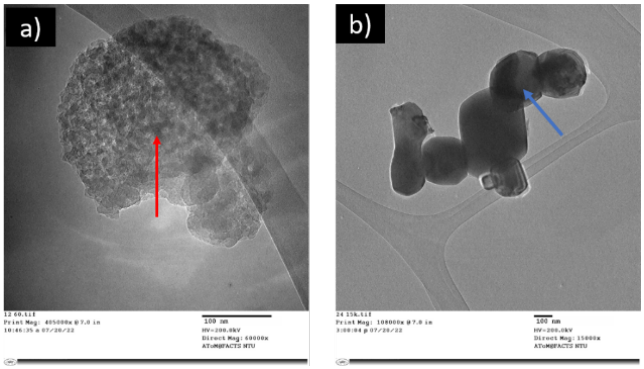

Figure 2. TEM micrographs of a) NPS from rice husks and b) opaque-porcelain powder of Vita VMK Master.

According to Fig. 1 above, at 15000x of magnification, morphological characteristics of NPS from rice husks were in amorphous phase with spherical shapes. The spheres were agglomerated to form clusters appeared to be cloudy, as indicated in red arrows.

Based on Fig. 2, the TEM micrographs showed different characteristics among both materials. The combination of sol-gel and sonication at 270 minutes yielded NPS with spherical and agglomerated characteristics (Fig. 2a), while the commercial powder had regular shapes appeared to be rectangle shapes (Fig. 2b).

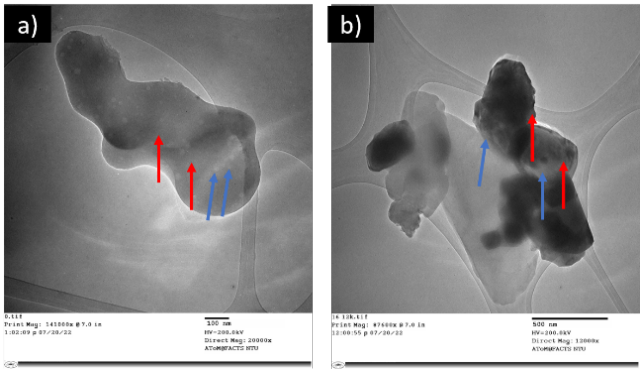

Figure 3. The TEM micrograph with the addition of 0.5% of NPS into the opaque porcelain (a), and with the addition of 1% of NPS

The TEM micrograph results showed that with the addition of 0.5 wt% of NPS into the opaque porcelain, it appeared to be more amorphous than the one with the addition of 1 wt% (Fig. 3). As can be seen in Fig. 3 red arrows represent the NSP and black arrows represent Vita VMK Master commercial opaque-porcelain powder.

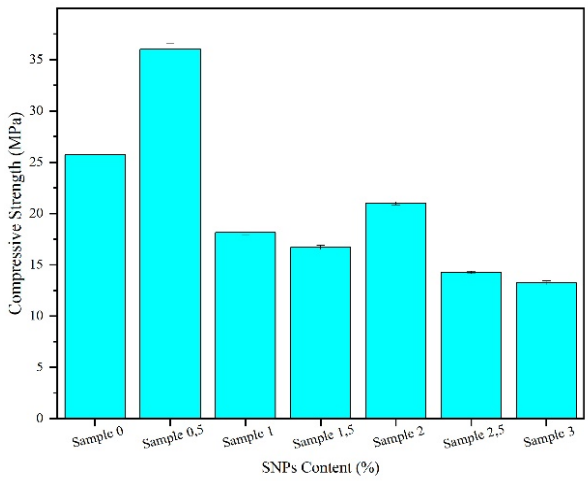

Figure 4. Average value of compression strength on metal-ceramic samples with and without the addition of NPS (n=4).

Compression results

The average strength of compression strength of samples is shown in Figure 4.

Based on Fig.4, the average compression strength of samples was in between 25 – 13 MPa. The highest compression strength was observed in sample with 0.5 wt% of NPS, accounted for (36.02±2.27 MPa), whereas the lowest strength was found in 3 wt% of NPS (13.23±0.79 MPa). Based on the One-Way Anova analysis, the average compression strength among the metal-ceramic samples had p=0.000 ($p<0.05$). Whilst the Post-Hoc Games-Howell test in Table 2 shows that 0.5 wt% sample of NPS had significant improvement of compression strength ($p<0.05$).

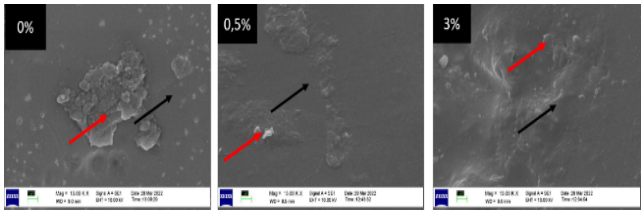

Figure 5. SEM micrographs for metal-ceramic samples after being tested for compression measurement.

Based on Fig. 5, the SEM micrographs display accumulated rough particles in samples without the addition of NPS (Fig. 5 on 0 wt%), whereas in 3 wt% of concentration, wavy and curvy textures were observed on the surface, indicated by red and black arrows. Unlike both concentrations, the 0.5 wt% concentration had flat and smooth surface, with a few clusters of rough surfaces seen in black and red arrows (Fig. 5 on 0.5 wt%).

Discussion

The null hypothesis was rejected as the results showed that the compression strengths were significantly different amongst the experimental groups ($p<0.05$). Rice husks are by-products of paddy-rice plantation with abundant amounts which potentially can be used as silica sources[14–16]. During the synthesis, the 1N HCl had the ability to dissolved impurities and organic matters within the husks, and at 90°C, the reaction could be occurred in a quick stage. The 1N NaOH had a function to react to the minerals, including silica to form sodium silicate. After being dropped with acid, such as HCl, the sodium silicate solution would form gelatinous phase in neutral condition [17,18]. This gelatinous phase could be converted into powder by drying treatment to obtain white-colour of silica powder[19]. The sonication treatment for 270 minutes aims to separate cluster of smaller particles within liquid system with simple process. The successful treatment is characterized by the mixing of liquid phase and the powder without accumulating precipitation of powder[12,20]. The 270-minutes was able to reduce chemical reagents that are high cost and harmful. The vibration from ultrasonic homogenizer could yield smaller particles of silica[12,20].

Based on the SEM and TEM micrographs in Fig. 1 and 2, amorphous silica with spherical shapes were observed, and the particles were agglomerated to form cluster of particles. These results were in line to a study conducted by Rojas et al., 2018 that stated agglomerated features silica from rice husks via pyrolysis combined with acid treatments. Morphological and agglomeration level are due to the introduction of strong acid, such as HCl in changing base conditions of sodium silicate after reaching neutral system. A higher concentration of sodium silicate solution would affect the size and morphological features of silica particles into higher dimension. Therefore, the use of HCl as strong acid would damage the crystalline structure of silica particle as well as changing the crystalline structures into amorphous structure, resulting in smaller size of particles due to the introduction of sonication treatment[11,12].

Retainers and pontic in metal-ceramic FPDs are the first layer that withstand mastication forces. Thus, as long as the mechanical characteristics of these two parts were poor, fractures could occur[21]. Compression testing on these retainers and pontic has a function to evaluate the withstand ability against the force that considerably reduce the denture size, especially in the posterior teeth region. In term of mastication forces, the posterior region frequently faces a high force, which eventually may lead to fractures[22]. The 0.5 wt% of NPS concentration showed the most significant improvement with 36.02±2.27 MPa. Based on the SEM micrograph on Fig. 5, this sample had smoother flat surface with smaller number of pores compared to those in control samples and 3 wt% of NPS samples. Despite of the composition, it is assumed that the amorphous

NPS diffused to the commercial powder, whereas the SEM micrographs showed smoother surface compared to that in 3 wt% of NPS sample with wavy and curvy surface. As the FPDs requires a high compression strength in order to prevent fracture during mastication or even more clenching and bruxism, homogenous condition between opaque and dentine layers have a great impact to mechanical properties. The addition of silica nanoparticle in a small amount on poly methyl-methacrylate acrylic (PMMA) have improved mechanical properties by Balos and team[23]. A high number of pores due to agglomeration may occur even though it has been mixed with commercial opaque-porcelain powder[24]. Thus, a sintering process would create a diffusion system between the opaque layers that have been filled with amorphous NPS to dentine, indicating optimum composition contributes importantly as it seen in 3 wt% of NPS with the lowest compression strength.

Partial fractures are considered as light fractures as this occurs cohesively. Subsequently, the porcelain surface might have been irregular and elongated, while the in severe fractures, separation between porcelain layers and metal-coping adhesively occurred[25].

Based on the compression strength in our study, percentages that can be recommended as a reinforced material is 0.5 wt%. It is assumed, in these two concentrations, stronger interaction between the opaque-porcelain/ NPS with commercial dentine porcelain occurred based on the morphological and statistical analysis. It is also necessary to investigate another commercial product that may have different contents of silica in order to obtain optimal reinforcement. Thus, certain guidelines can be used as a reference in preparing the new-locally made of opaque-porcelain powder with ideal compression strength. With highly competitive and sustainable demands particularly in field of dentistry, the use of rice-husks can be alternative solution to obtain a better product with more affordable price.

Study limitation

Difficulties in evaluating the homogenous features of mixture of NPS powder and opaque-porcelain powder with manual method may impact the distribution of both powders. As a result, it may have affected the mechanical strengths, thus; it is necessary to investigate further about the mixture process with the mechanical properties of metal-ceramic samples.

Conclusions

The combination of sol-gel and sonication methods have successfully yielded silica in nanoparticle size. Based on morphological characteristics, the NPS were in amorphous structure, with spherical and agglomerated behaviours. By mixing the NPS powder into opaque-porcelain layer in smaller concentration have improved the compression properties of metal-ceramic samples, which accounted respectively for 36.02±2.27 MPa and mostly significant ($p<0.05$). The compression testing also demonstrated that a high concentration of NPS showed the lowest value of characteristics due to wavy and curvy surface in metal-ceramic sample, indicated by the agglomeration of amorphous NPS after being sintered. Within the limitation, this study demonstrates a significant improvement of compression strength via the introduction of silica nanoparticle in a small percentage.

Declaration of Interest

The authors report no conflict of interest.

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