

# Innovation of Dental Interim Prosthesis—Silver Nanowires Composite Resin

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**Abstract** - The most notable concern regarding interim fixed partial dentures are their poor mechanical properties and the formation of microbial biofilms in the oral cavity. Therefore, the objective of the current study was to develop novel enhanced interim fixed partial dentures. Polymethyl methacrylate (PMMA) and silver nanowires (AgNWs) were incorporated into manufactured PMMA/AgNW composite resin by using the uniform design (UD) method. The mechanical properties of the composite resin were evaluated using the nanoindentation method and its antibacterial properties were examined using the disk diffusion method under the JIS Z 2801-2000 experimental standard. In addition, surface hardness, roughness, and color stability were investigated after restoring the composite resin in 37 °C water. Through the UD method for manufacturing PMMA composite resin, the following factors were identified as affecting surface hardness, in order of weighting: polymerization pressure > polymerization time > aspect ratio > concentrations. The optimal conditions were found to be polymerization pressure of 1.6 bar, polymerization time of 10 minutes, AgNW aspect ratio of 14, and concentration of 0.5%. Compared with pure PMMA, the PMMA composite resin was 1.7 times harder. Furthermore, according to the antibacterial experiments, the AgNW composite resin had a superior mechanical strength and inhibitory effect against *Escherichia coli* than the composite resin did. AgNWs are an attractive alternative to traditional antibiotics because of their antimicrobial activity and low bacterial resistance. They can optimize initiator systems to produce commercially useful dental and medical resins.

**Keywords** - Silver nanowires, polymethyl methacrylate, surface hardness, uniform design, antibacterial

## 1. INTRODUCTION

The dental treatments are in needs of provisional crowns or fixed partial dentures (FPDs) for an extended period, especial for interdisciplinary therapy. They could protect the prepared teeth, stabilize the teeth position in the arch, provide vertical dimension support, chewing function, maintain the basic esthetic and periodontal health [1]. A good provisional prosthesis should own good margin integrity, mechanical strength and easy making. The most commonly component used for provisional materials are methacrylate resin and dimethacrylate resin. The material

used for fabrication of provisional restoration consists of pigments, monomers, filler and an initiator. The ability of the monomer is to convert to a polymer allows the material to set into a solid that's durable enough to withstand the oral environment for the necessary interim period. Monomers associated with different provisional materials impart different characteristics such as exothermic heat of reaction, polymerization shrinkage, and strength.

The methacrylate resin, for examples, polymethyl methacrylate (PMMA), polyethyl methacrylate (PEMA) and polybutyl methacrylate (PBMA) are popular in dental clinic. They are low-molecular-weight, linear molecules that exhibit decreased strength and rigidity [2]. The advantages of methacrylate resin are easy to repair, relines and low expensive. The disadvantages of methacrylate resin are exothermic heat of reaction, polymerization shrinkage. The dimethacrylate resin, for examples, bisphenol-a glycidyl dimethacrylate (bis-GMA) and urethane dimethacrylate (UDMA) are gaining in popularity in dental clinic. These composite materials are difunctional and capable of cross-linking with another monomer chain. This crosslinkage imparts strength and toughness to the material. The advantages of dimethacrylate resins are their cartridge delivery system. This dispensary method not only is convenient but also may allow for a more accurate and consistent mix. The disadvantages are not easy to repair and more expensive.

In recent years, various nanocomposite materials have become used in daily life. Nanomaterials have unique physical and chemical properties that have different quantum size effects, determining their specific electrophysical performance [3]. In metal nanoparticles, some properties such as magnetism, optical polarizability, and chemical reactivity are enhanced [4]. Silver nanoparticles (AgNPs) exhibit optical, thermal, chemical, and physical characteristics that differ from those of Ag (bulk). Furthermore, silver nanowires (AgNWs) have all the AgNPs characteristics. Because AgNWs are chemically synthesized [5] and have high conductivity and transmissivity [6-8], they are widely used in the field of

microelectronics for the production of microsensor devices and for processes such as catalysis, surface-enhanced Raman scattering, and photonic crystals. Therefore, AgNWs are a promising alternative to indium tin oxide used in the display industry. Furthermore, metal nanowires and nanoparticles possess high surface activity [9].

The limitations of interim fixed partial dentures include weak mechanical properties and the formation of microbial biofilms in the oral cavity. Previous research studying the antibacterial properties of provisional crowns has focused on the amount of antibiotics loaded into the crown [10, 11]. However, the increasing use of antibiotic-loaded provisional crowns may result in the development of resistant bacterial strains, leading to a relatively short-lived antibiotic action. Other research that makes use of nano-sized silver in bone cement has been proposed as a better alternative [12]. Various forms of Ag- or Ag-ion-containing fillers that have antibacterial activity in vitro have been used for dental applications. They include Ag-ion-implanted SiO<sub>2</sub> [13], Ag-containing silica glass [14], and Ag-zeolite- [15], Ag-apatite- [16], and Ag-supported zirconium phosphate [17]. However, Ag-zeolite and Ag-apatite decreased mechanical properties at loadings necessary for antibacterial effects [18]. Furthermore, Ag-ion containing fillers such as Ag-supported zirconium phosphate only kill bacteria that come in contact with the surface; protein adsorption on the surface would reduce the antibacterial action. Moreover, increasing concerns exist regarding the potential side effects of directly using nanoparticles in vivo. For effective dental and medical application, a more effective method of incorporating AgNPs into acrylic resins and delivering Ag<sup>+</sup> ions is required [19]. Fan et al. showed that the strongest antibacterial effect was obtained when an Ag-ion containing polymer was used, in which the antibiotic and silver ions synergistically inhibit bacterial growth [12, 19, 20].

Polymethyl methacrylate (PMMA) has been widely used to fabricate restorations in dental treatment. However, environmental factors such as temperature [21], pressure, and water immersion may affect the degree of polymerization and influence the clinical behaviors of PMMA [9, 11, 22]. In this study, the influence of environmental factors and AgNW parameters in PMMA were analyzed according to a parametric design method [21, 22]. The uniform design (UD) method developed by Fang and Wang in 1978 obtained more results from fewer replications. This method seeks its design points to be more uniformly scattered on the research experimental design domain [22] and can increase the efficiency and reduce the waste of experiments. Although detailed studies [23] have been conducted on the antibacterial activity of Ag ions, to the best of our knowledge, no studies have examined the antimicrobial properties of AgNWs and

PMMA. Therefore, the purpose of the study analyzed the best mechanical properties by using the UD method and antibacterial abilities of new nanomaterials combined with AgNWs and PMMA.

## 2. Materials and Methods

### 2.1 Parametric design method: UD

The UD method is designed for analysis in multifactorial studies and involves a favorable UD experiment table, uniformly distributed parametric points, and easily found rules. The benefits of the UD table are as follows. Each level is only used once in the UD table, each row and column has only one experimental point, and the parameters do not repeat any combination of two columns.

The UD table has a UD symbol  $U_n(m^k)$  or  $U_n^*(m^k)$ , where  $U$  is the UD symbol,  $n$  is experiment times,  $m$  is experiment factor level,  $k$  is the factor number, and  $*$  is the mean better uniformity [24].

This study used the ( $6^4$ ) table for the experiment, as shown in Table 1.

### 2.2 Composite resins process

The UD method was used to manufacture PMMA/AgNW composite resins. After the designed process, cold mounting, grinding, and polishing was performed on the composite resins. Subsequently, the mechanical and antibacterial properties of the composite resins were analyzed. The polyol method was used to produce AgNWs (Figure 1) and AgNPs (Table 2) [7, 8]. Different AgNW concentrations are indicated by different colors. The PMMA/AgNW composite resin (Tempron, GC Corporation, Tokyo, Japan) was fabricated according to the manufacturer's instructions. The hardness and modulus of the pure PMMA were  $192 \pm 2.4$  MPa and  $4.45 \pm 0.06$  GPa, respectively. A parametric study was designed to include pressure, time, AgNW aspect ratio, and concentration. The mechanical and antibacterial properties of the PMMA/AgNW composite resin were then analyzed. All the experiments were performed at  $24 \pm 1$  °C and 62%  $\pm$  2% relative humidity.

### 2.3 Measurement of mechanical and optical properties

#### 2.3.1 Surface roughness test with $\alpha$ -step

The surface roughness of the PMMA/AgNW composite resin was measured with an  $\alpha$ -step (Mitutoyo SurfTest SJ-400) surface profile measuring system [25] by using a probe contact and shift position. The probe was used to measure the roughness of two sides of the sample's surface (up and down) and to convert the output sample surface information into an electrical signal.

#### 2.3.2 Nanoindentation hardness test

This study used the MTS Nano-Indenter XP, a nanoindentation tester made by MTS Systems

Corporation[26]. A Berkovich tip with an angle of  $142.4^\circ$  was used for indentation tests. The micro Vickers is widely used to test hardness. The Nano-Indenter XP works on a similar principle, but on a much smaller scale. This tester can measure elastic modulus, hardness, and adhesion. The instrument automatically calculates hardness (H), defined as applied normal force divided by the projected area.

$$H = \frac{P_{\max}}{A}$$

where  $P_{\max}$  is the maximum loading force and  $A$  is the projected area of contact on the original surface at a given point in the experiment. The elastic modulus (E) is calculated using the following formula.

$$E = \frac{2}{\sqrt{\pi}} S \sqrt{A}$$

where  $S$  is the slope of the unloading curve and  $A$  is the projected area of contact on the original surface at a given point in the experiment.

#### 2.4 Antibacterial testing

Enhancing antibacterial properties was one of the purposes of the AgNW reinforcement. Antibacterial analysis is crucial for all prosthodontic treatments. In the antibacterial experiment, colony-forming units were measured according to AgNW solutions was analyzed using quantitative disk diffusion. The inhibition zone diffuses outward around the sample disk and is defined as follows:

Inhibition zone = inhibition diffusion diameter (D) – sample disk diameter (d)

Using this equation, we could easily quantify the antibacterial properties and identify the antibacterial sensitivity of sample. APEX-nanosilver (AgNPs, Apex Nanotek Corporation, Taipei, Taiwan) was used for a control.

#### 2.5 Statistics

The UD method tends to find poorer variance properties than does standard response surface designs or variance optimal designs near the boundary of the experimental region, but it tends to find superior variance and bias properties in the interior of the experimental region [27]. Therefore, direct observation or regression analysis [28] appeared to be the most suitable methods for determining the optimal orientation in the study.

### 3. Results

#### 3.1 Porosity coverage

The  $U_6^*(6^4)$  UD table was chosen for the experiment. After the designed process, the composite resins were analyzed using OM and image analysis software [Figure 2(a)]. Samples 1 and 4 had a relatively higher porosity

coverage rate than did the other samples. The porosity coverage of Sample 1 was more than 50%. This high porosity coverage rate (i.e., looser structure) resulted in poor mechanical properties of the composite resin and easy bacterial accumulation.

#### 3.2 Roughness

The composite resins were soaked in water after cold mounting, grinding, and polishing. They were then taken out at different times to measure roughness. Figure 2(b) shows the comparison of six samples. Sample 1 was synthesized with 0.3% AgNWs at 0 bar gauge pressure, and while synthesizing Sample 4, the pressure was increased to 1.6 bar. Because of the low AgNW concentration, the porosity coverage of Sample 4 also increased. Among the samples, Sample 1 had the highest surface average roughness, followed by Sample 4, because of its higher porosity coverage rate. Sample 1 had a rough morphology, but this morphology could be improved by increasing the applied pressure. Soaking had little influence on the roughness of the samples.

#### 3.3 Surface hardness and elastic modulus measurement

The hardness and elastic modulus were analyzed using the MTS nanoindenter XP systems. After being soaked, the surface hardness and modulus of all the resins increased. Figures 3(a) and (b) present a comparison of all six samples. After soaking in water for 5 days, Sample 5 had the maximum hardness of  $332 \pm 2$  MPa, and Sample 1 had the minimum hardness of  $88 \pm 3$  MPa. Moreover, the hardness of Sample 5 increased by 88.44% after being soaked in water and was 1.7 times harder than the pure PMMA ( $192 \pm 2.4$  MPa). Soaking did not have any significant effect on the modulus. Samples 5 and 6 had higher elastic modulus values than the other samples did. Compared with the pure PMMA ( $4.45 \pm 0.06$  GPa), the modulus of Samples 5 and 6 increased 1.34 and 1.28 times, respectively.

#### 3.4 Regression analysis

After the samples were soaked, the influence of experimental factors on surface hardness was analyzed using statistical software. The main factor affecting surface hardness was determined to be pressure, followed by time, aspect ratio, and concentration. The contribution ratio of each factor is shown in Figure 3(c) and (d). The factors affecting the hardness of the PMMA/AgNW composite resin were pressure (62.9%), polymerization time (22.1%), aspect ratio (10.8%), and concentration (4.3%). Pressure was the most crucial factor determining resin hardness. The higher pressure environment completely cured the PMMA, making the PMMA/AgNW composite resins harder. The factors affecting the modulus of the PMMA/AgNW composite resin were pressure (82.01%), polymerization time (3.72%), aspect ratio (7.67%), and concentration (6.60%). In addition, the contribution of

each factor was summed. As shown in Figure 3(c) and (d), pressure was the most influential factor affecting both surface hardness and modulus because resins are the main ingredient of AgNW composite resins. Resins require high pressure and more time to polymerize. The regression results showed that mixing AgNWs into the resin improved the modulus and hardness by 7.67% and 10.8%, respectively. Moreover, the contribution of AgNWs aspect ratio has 6.60% and 4.30% on the modulus and hardness.

### 3.5 Antibacterial experiment

The disk diffusion method was used to test the antibacterial effect of liquids. *Staphylococcus aureus* (S.a) and *Escherichia coli* (E.c) were chosen as the test bacteria. The PMMA/AgNW solution that had the most desirable surface hardness and the pure AgNW solution were analyzed using the disk diffusion method. Table 2 shows the S.a and E.c inhibition zones of both, Ampicillin exhibited an inhibition zone diameter of 20 mm against S.a (Fig 4-a1) and 4 mm against E.c (Fig 4-b1). The PMMA/AgNW solution was not sensitive against S.a (Fig 4-a6), but it had an inhibition zone diameter of 5 mm against E.c (Fig 4-b6). These results indicate that AgNWs have antibacterial effects against E.c, particularly when used as an AgNW/PMMA composite resin.

## 4. Discussion

The oral cavity of healthy individuals contains hundreds of different bacterial, virus, and fungal species. Most are commensal species, but they can become pathogenic in response to changes in the environment or other triggers in the oral cavity. Many gram-negative bacteria found in the mouth, especially in subgingival plaque. The plaque accumulation and deposition rate depend on the quality of an individual's personal hygiene and the quality of prosthesis. The porosity cover rate and roughness of prosthesis affect the quality of prosthesis and plaque clustering. The dental and medical fields are in need of an alternate effective long-period interim antimicrobial PMMA/AgNW composite resin that does not promote the growth of antibiotic-resistant bacterial strains. Therefore, the concept of fusing Ag nanoparticles into dental and medical resins was proposed. Ampicillin is antibacterial ( $65.6 \pm 3.9$ ). Methanol was the preserve liquid of nanosilver, it could maintain Ag. NanoAg particle was unstable, it has high surface energy. Methanol could decrease nano silver energy. Nano Silver when expose air, it could bond with Oxygen. It was not easy to reduce. Stable and black color ( $\text{Ag}_2\text{O}$ ) PMMA ( $\text{C}_5\text{O}_2\text{H}_8$ )<sub>n</sub> share O atom with Ag, it could decrease nanosilver surface energy but not complete.

In this study, similar antibacterial results were produced using AgNWs instead of AgNPs [19, 20]. Slane et al. reported that the highest biofilm inhibition level was obtained with 0.5% AgNPs for *Staphylococcus epidermidis*

and *S. aureus* [20]. In the present study, the best combination from UD was No.5 with 0.5% AgNWs for E.c (Table 2) show the good biofilm inhibition level. E.c is gram negative rod-shaped bacteria with a thin cell wall, whereas S.a is gram positive cocci with a thick cell wall. The inhibition of gram negative bacteria was good a beginning for further study and clinical test.

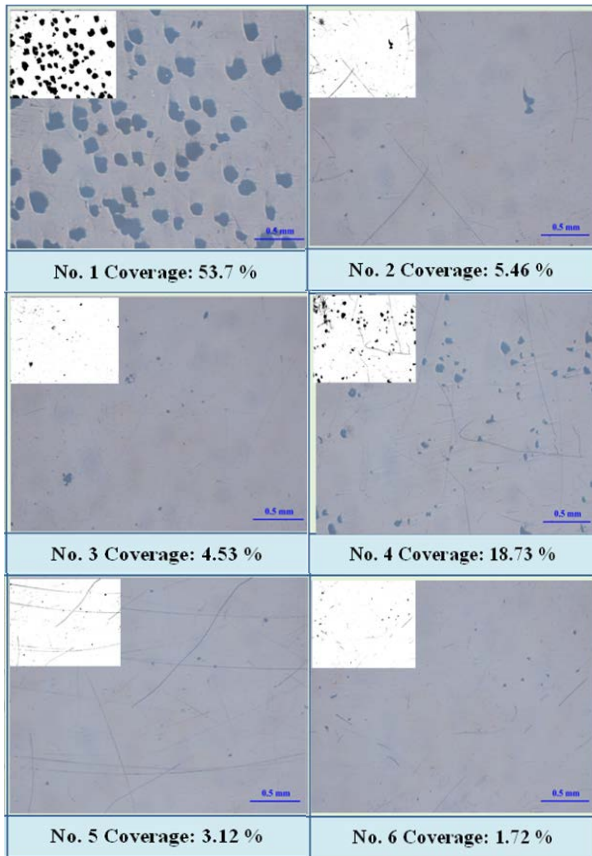
On the other hand, the literature indicates that AgNP cements significantly reduce the modulus and yield strength. Studies focusing on Ag-ions and nanoparticles have failed to improve the mechanical properties of dental resin [19, 20]. In the current research, AgNWs combined with dental PMMA resin showed obvious hardness and modulus improvement, according to the most favorable UD group. The benefit of this new dental interim prosthesis is high modulus and hardness, leading to limited wear and tear of the resin. According to UD and regression analyses, the mechanical properties (hardness, elastic modulus, etc.) of dental interim prosthesis depend on fabrication factors such as AgNW aspect ratio, AgNW concentration, time, and pressure. Sample 5 had the highest hardness and modulus. In figure 3a and 3b, the elastic module was  $6.22 \pm 0.21$  GPa and the hardness of sample 5 was  $332 \pm 2$  MPa after soaking in water for 5 days. Ishiyama et al appointed the Young's modules of PMMA decreased with increasing humidity, ranging from 3~4 GPa. The new composite resin could be achieved one and a half times than common one. In general, the Young's modules of enamel was 50~130 GPa, and dentin was 18~35 GPa. The new composite resins would be better strain performance than average ones in the same occlusal stress. Kratky et al show the nanohardness of PMMA was 260~262 MPa. The study of Chun et al appeal that the nanohardness of enamel and dentine was  $274.8 \pm 18.1$  MPa and  $65.6 \pm 3.9$  MPa.

For the successful fabrication of dental interim prosthesis resin, many factors must be considered. However, previous studies have failed to consider all these factors simultaneously. Effective experimental design methods, such as the UD and Taguchi methods [29], can help reduce manufacturing time and promptly elucidate the key factors affecting the process. However, further research with longer periods of color stability, optimal hardness values that are stable in highland, and antibacterial effects against anaerobes is warranted to support the findings of the current study.

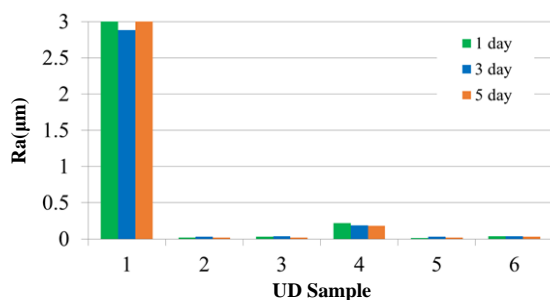
5. EXPERIMENTAL RESULTS

Table 1.  $U_6^*(6^4)$  experimental parameters

Factor Parameter Sample	Pressure (bar)	AgNWs concentration (%)	Polymerization Time (min)	AgNWs aspect ratio
1	0	0.3	30	65
2	0.4	1	60	48
3	0.8	3	20	36
4	1.2	0.1	50	25
5	1.6	0.5	10	14
6	2	2	40	1

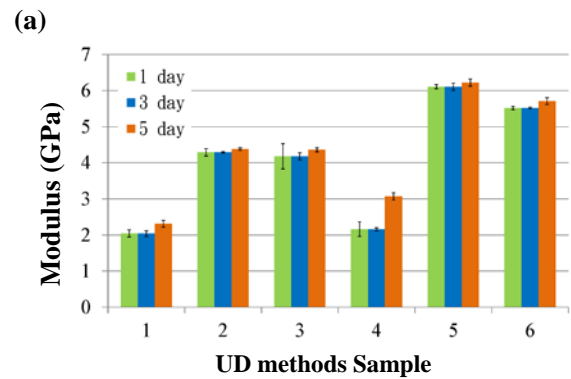


(a) Porosity coverage rate



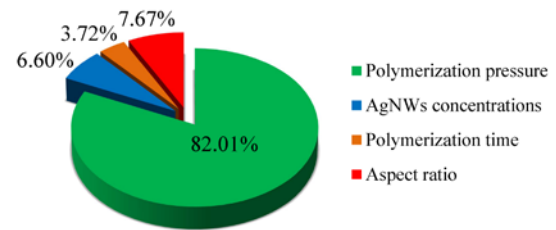
(b) surface roughness

Figure 1. (a) Samples 5 and 6 exhibited relatively low porosity coverage rates, whereas Sample 1 had the highest coverage rate. (b) The results of surface roughness were positively related to porosity coverage rate. Samples 5 and 6 had relatively low surface roughness.

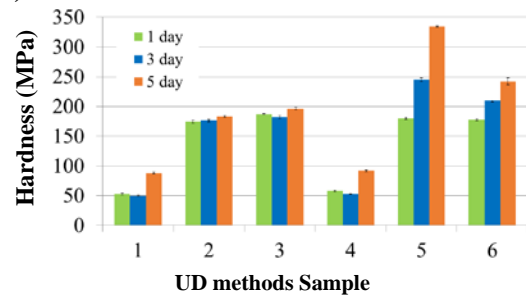


(a)

Contribution of each factor (Modulus)



(c)



(d)

Contribution of each factor (Hardness)

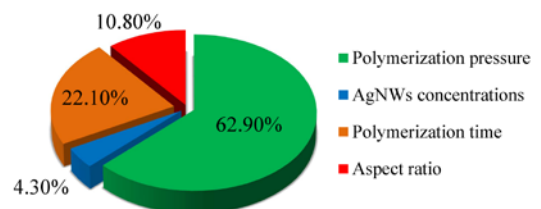


Figure 2. (a) Comparison of modulus results. Sample 5 shows the most favorable modulus. (b) Comparison of microhardness results. Sample 5 exhibits the highest value. (c)(d) Contribution of each factor influencing interim fixed

partial dentures. Polymerization pressure was the greatest contributing factor to modulus and hardness.

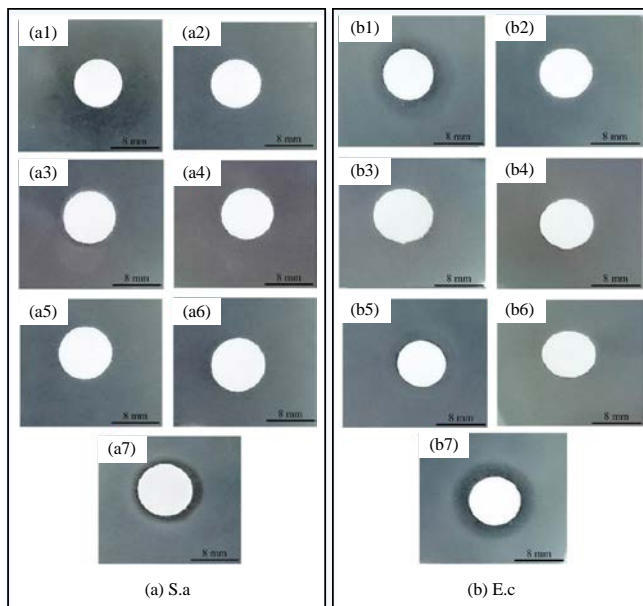


Figure 3. Antibacterial test results of (a) *Staphylococcus aureus* (S.a) and (b) *Escherichia coli* (E.c) testing. Ampicillin revealed an inhibition zone diameter of 20 mm against S.a (a1) and 4 mm against E.c (b1). The methanol (a2, b2)/AgNWs (a3, b3)/AgNPs (a4, b4)/PMMA liquids (a6, b6) were not sensitive against S.a and E.c. The AgNW/PMMA solution was not sensitive against S.a (a5) and showed an inhibition zone diameter of 5 mm against E.c (b5). The control group of APEX-nanosilver exhibited an inhibition zone diameter of approximately 2 mm against S.a (a7) and 3 mm against E.c (b7).

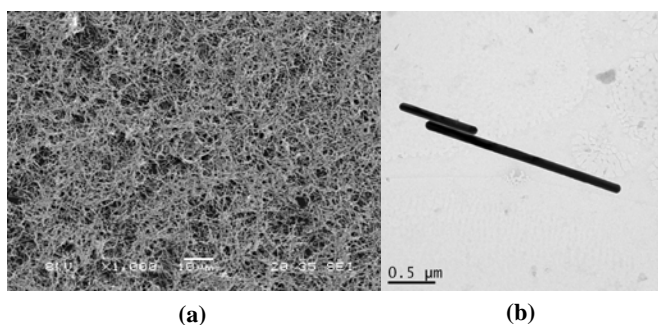


Figure 4. (a) SEM image of high-volume AgNWs at 1000× magnification and (b) TEM images of 0.7 μm in length, 1.5 μm in length, and 0.1 μm in diameter.

## 6. Conclusions

Despite the aforementioned limitations, the following conclusions can be made according to the results of this study:

1. Lower porosity, color stability, and an adequate AgNW concentration are favorable properties for a new dental interim prosthesis.

2. The proposed dental interim prosthesis exhibited desirable antibacterial and mechanical properties because of the proper mixing and processing of AgNWs and PMMA.
3. The UD method helps researchers maintain a favorable direction and minimize the production of experimental byproducts.
4. According to the UD method, the optimal conditions for producing this novel interim prosthesis are 1.6 bar pressure, 0.5% AgNW concentration, 10 minutes of polymerization time, and an Ag aspect ratio of 14.

## Declaration of interests

The authors declare that they have no conflict of interest.

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