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Research Article

Mechanical, Chemical, and Morphological Characterization of 1% Nanofiber-Reinforced Self-Cure Acrylic for Denture Applications

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ABSTRACT

Introduction: Self-curing resin acrylic (SC-AR) is widely used for biomedical appliances, particularly prostheses, due to its rapid manipulation and suitability for single-visit fabrication. However, its relatively low mechanical strength may increase fracture susceptibility. This study evaluated the effect of incorporating 1 wt% polyacrylonitrile polymethyl methacrylate electrospinning nanofibers (e-nanoPAN-PMMA) on the physicochemical, mechanical, and morphological characteristics of SC-AR.
Materials and Methods: SC-AR specimens were divided into conventional SC-AR (SC-ARc) and nano-reinforced SC-AR (SC-ARnano) groups. Chemical characteristics were evaluated using X-ray diffraction (XRD) and Fourier-transform infrared spectroscopy (FTIR). Flexural strength (FS) and flexural modulus (FM) were assessed using a three-point bending test, while fracture-surface morphology was examined using scanning electron microscopy (SEM). Data were analyzed using an independent t-test ($P \leq 0.05$).
Results and Discussion: SC-ARnano exhibited significantly higher FS and FM than SC-ARc ($P \leq 0.05$). XRD patterns showed characteristic polymeric resin features in both groups, with no additional crystalline peaks in SC-ARnano. FTIR revealed a nitrile absorption band in SC-ARnano. SEM demonstrated nanofibers embedded within the resin matrix, with no detectable surface porosity.
Conclusion: Incorporation of 1 wt% e-nanoPAN-PMMA significantly improved the mechanical properties of SC-AR while maintaining its physicochemical characteristics. SC-ARnano demonstrates potential as a reinforced acrylic resin for biomedical applications.

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INTRODUCTION

Resin is a versatile material widely used in the fabrication of biomedical appliances due to its excellent biocompatibility, chemical resistance, and ease of manipulation. Examples include orthopedic implants, maxillofacial implants, tissue-engineering scaffolds, and medical devices. In dental applications, resins are commonly used to create crowns, bridges, dentures, and orthodontic devices, providing patients with durable and aesthetic solutions. The ability to customize resins through various polymer formulations makes them suitable for precise, intricate biomedical appliances, thereby enhancing comfort and functionality. Furthermore, modern advancements have led to the development of medical-grade resins that can be easily fabricated using various techniques, such as three-dimensional (3D) printing. These innovations have improved the efficiency, cost-effectiveness, and visual appeal of prosthetic devices, greatly enhancing the quality of life for individuals who need biomedical prosthetic appliances.[1-2]

Among the various polymer resins, Self-cure resin acrylic (SC-AR) is preferred for day visits due to its ease of handling. However, SC-AR has lower mechanical strength than the commonly used heat-cured type for denture bases, making it more prone to recurrent fractures. Additionally, it has greater porosity, which can affect its durability and biocompatibility. Therefore, SC-AR has frequently been used only for corrective or immediate procedures. Unless the mechanical properties of SC-AR are modified, its advantages and applications should be improved.[3-4]

These limitations can be overcome by reinforcing the material, for example, by adding fibers to improve its mechanical performance. Fibers such as glass, natural fibers, polyethylene, polypropylene, polyester, nylon, and PAN are used to reinforce acrylic resin.[5-8] Polyacrylonitrile (PAN) is widely used as a precursor to build high-performance fibers or membranes because it has excellent resistance to heat, chemicals, and lysis.[9] PAN fibers offer a unique combination of high tensile strength, thermal stability, and chemical resistance. When used in cement, PAN fibers offer several advantages, such as superior crack control, corrosion resistance, better dispersion, and increased durability and toughness.[10.] Polymethyl methacrylate (PMMA), which has good transparency, is lightweight and durable and has also been used in many industrial and biomedical fields. In the meantime, electrospinning is a method for producing polymer fibers with nanometer-scale diameters through an electrostatically driven jet of polymer solution. The electrospinning technique is preferred because it is relatively economical and adaptable to various types of materials. Electrospinning has gained popularity for various biomedical and dental applications due to its ability to create nanofibers with superior mechanical properties.[11-12]

A previous study on heat-curing acrylic resin reported that Polyacrylonitrile polymethyl methacrylate electrospinning nanofibers (e-nanoPAN-PMMA) fibers at 0.1 w% and 0.5 w% did not effectively improve the mechanical strength.[13] Another study reported that 1% glass fibers could improve the mechanical strength of a resin composite.[14] Based on these findings, this study modified SC-AR with 1 w% e-nanoPAN-PMMA to evaluate the minimum concentration required to improve mechanical performance without compromising the properties of conventional self-curing acrylic resin. Therefore, this study aims to characterize the mechanical, chemical, and morphological properties of 1% e-nanoPAN-PMMA nanofiber-reinforced self-cure acrylic resin (SC-ARnano) for denture

applications, compared with conventional self-cure acrylic resin (SC-ARc).

MATERIALS AND METHODS

This study is a true experimental laboratory study with a posttest-only control group design. Samples were divided into two groups, with and without 1% Fiber e-nanoPAN-PMMA. Each group included 5 samples, each measuring 2×2×25 mm. The first group was SC-ARc, with a powder (P): liquid (L) ratio of 0.3 g:0.15 mL, and the second group was SC-AR powder with 1w% e-nanoPAN-PMMA and 0.15 mL of liquid.

Fiber e-nanoPAN-PMMA was prepared as follows. A total of 1.4 g of PAN (Haihang Industry Co., China) was dissolved in 10 mL of dimethylformamide (DMF, Merck, Germany), and the solutions were mixed and stirred magnetically for about 3 h to obtain a homogeneous solution. In addition, 1.2 g of PAN and 1.2 g of PMMA particles (Haihang Industry Co., China) were each dissolved in 10 mL of DMF. All solutions were then electrospun under the same conditions using standard electrospinning equipment. The solution was fed to the tip of a 5 mL needle (0.4 mm diameter) using a syringe pump at a flow rate of 0.4 mL/h. As the electric field increased, the nanofibers were spun and collected on a grounded metal sheet measuring 10 cm by 15 cm. The applied voltage was 16 kV, and the distance between the needle tip and the metal sheet was 17 cm. All non-woven fabrics were dried in a vacuum oven at 60 °C for 48 h. The weighed quantity of the non-woven nanofiber sheet was torn into small pieces before being mixed with resin powder. The sample mixture was then combined with resin liquid, manually manipulated until it reached the dough phase, placed into a mold, and allowed to harden completely for 10-15 min. Sample surfaces were polished until mirror-polished.

Acrylic resin characterization included surface morphology and physical, mechanical, and chemical properties.[15-17] The surface morphological characteristics of self-cure acrylic resin samples were comprehensively evaluated using a Scanning Electron Microscope (SEM/Hitachi; SU3500) at 20kV to assess the influence of e-nanoPAN-PMAA incorporation on surface quality and porosity. Bulk samples were sputtered with gold before analyzing the surface morphology of the resin on the top of mirror-polished samples. After three bending point tests, the samples' fractured surfaces were examined again using SEM.

The 3-point bending test was set up and calibrated using the Universal Testing Machine (UTM; Tensilon RTF 1310-10KN) according to the manufacturer's instructions. Samples were mounted on the test plate, and a maximum load was applied at a crosshead speed of 5 mm/min until the samples broke. Numerical data and graphs were collected. After the test data were obtained, they were calculated. In this study, FM data were obtained by calculating using the formula: Flexural Modulus = $mL^3/4bh^3$ while FS was obtained by calculating using the formula: Flexural Strength = $3FL/2bh^2$ where F is the maximum load applied (N), L is the length (mm), b is the width of the test sample (mm), h is the thickness of the test sample (mm), and m is the slope of the linear part of the load-deflection curve.

Functional groups and chemical interactions in the samples were identified using FTIR-ATR (Thermo FTIR iS5, S/N: ASB1706545). The crystal structure was characterized by powder XRD (D8 Advance ECO, Bruker) using Cu Kα X-rays at a wavelength of 1.54060 Å, 40 kV, and 25 mA. The data were processed using SPSS. Normality was tested using the One-Sample Shapiro-Wilk Test. The data

were analyzed using a t-test at a significance level of P≤0.05.

RESULTS AND DISCUSSIONS

The SEM analysis revealed distinct differences between the SC-ARc and the SC-ARnano variant, as shown in Figure 1. The SC-ARc exhibited significant surface porosity, with numerous voids and irregular cavities distributed across the specimen surface, as shown in Figure 1A and C. In contrast, the SC-ARnano displayed a remarkably smoother, more uniform surface morphology, with no visible porosity, as shown in Figure 1B and D. This surface characteristic may be attributed to the interaction of e-nanoPAN-PMMA with acrylic powder during the polymerization process.

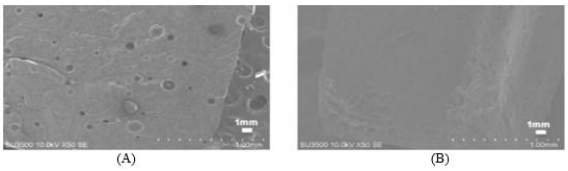

Figure 1. Surface characteristics of (A) SC-ARc. (B) SC-ARnano (C).

The surface porosity in SC-ARnano samples shown in SEM images in Figure 1B shows significant differences. This condition represents a critical improvement for SC-AC applications, whereas SC-AC (Figure 1A) is known to have higher porosity due to the chemical polymerization process and residual monomer content. Porosity in dental prostheses is widely recognized as a detrimental characteristic that significantly compromises the clinical performance and longevity of dental restorations. The presence of pores creates stress concentration points that act as crack initiation sites, leading to premature material failure under clinical loading conditions. The improved surface quality observed in our study is particularly significant for biomedical applications, where surface smoothness directly correlates with reduced bacterial adhesion, improved aesthetics, and enhanced patient comfort. Practically, SC-AC has more porosity than conventional heat-cure resin acrylic; therefore, these surface improvements are clinically relevant. These findings could broaden the clinical implications of SC-ARnano as a biomedical appliance. Previous results showed that 0.1-0.5% of e-nanoPAN-PMMA in heat-cure acrylic resin showed no differences in surface morphology and mechanical properties.[13-14]

The mechanical performance of both samples was quantitatively assessed using a 3-point bending test, measuring both FS and FM in Figure 2. This Figure demonstrates that increasing the nanofiber content from 0 wt% to 1 wt% improves both the FS and FM of the material, suggesting that the addition of e-nanoPAN-PMMA enhances the material's resistance to bending and stiffness. In this study, 1 wt% of e-nanoPAN-PMMA was added as the minimum concentration required for mechanical and physical alteration. As required, the ISO 20795-1:2013 International Standard specifies that the minimum FS, defined as the maximum stress a denture base polymer can withstand before fracture, is 65 MPa. The FM, defined as the stiffness of a material in resisting bending before deformation, is 2.4 GPa.[18] The substantial improvements in both FS and FM observed in SC-ARnano samples can be attributed to several synergistic mechanisms.[19] The primary reinforcement mechanism involves stress transfer from the polymer matrix to the high-strength nanofibers through interfacial bonding. Core-shell e-nanoPAN-PMMA provides effective reinforcement for resins, as demonstrated in our study. The high surface area of nanofibers creates an extensive interfacial area that facilitates efficient load distribution throughout the resin matrix structure.[20-21] The mechanism behind porosity reduction can be attributed to the nanofibrous network acting as a

three-dimensional reinforcing scaffold that prevents void coalescence during polymerization. Related to these findings, as shown in Figure 1.A and 1.B, reduced surface porosity can reduce stress concentration, thereby increasing FS and FM values. Surface modification of nanofibers improves interfacial adhesion between fibers and the resin matrix, thereby enhancing matrix densification.[22]

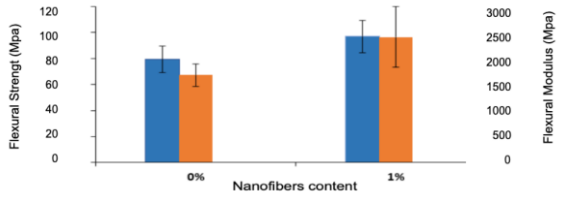

Figure 2. Flexural Strength (blue) and Flexural Modulus (orange) improvement of SC-ARc and SC-ARnano after adding 1% nanofibers.

FTIR spectroscopy was employed to confirm the successful incorporation of e-nanoPAN-PMMA and to analyze the chemical interactions within the resin system. Figure 3 displays a comparison of the FTIR spectra of SC-ARc and SC-ARnano. The FTIR analysis revealed distinct spectral differences between the two samples. Both samples show O-H/N-H functional groups as broad peaks spanning 3200-3600 cm^{-1}, with a slight shift and intensity change in SC-ARnano. This indicates possible hydrogen bonding between the nanofibers and the resin. The C-H peaks at 2850-2950 cm^{-1} were present in both samples; however, the intensity was slightly different in the SC-ARnano, related to the modification of the nanofibers' hydrocarbon environment. A noticeable change in both spectra was also observed at 1000-1300 cm-1, corresponding to C-O stretching and C-O-C vibration, due to the chemical incorporation of the resin structure and nanofibers. The most significant finding was the presence of a characteristic nitrile absorption band at 2260-2220 cm-1 in the SC-ARnano, which was absent in the SC-ARc spectrum. This nitrile peak serves as a definitive marker confirming the successful integration of e-nanoPAN-PMMA into the acrylic resin matrix, as the nitrile groups (-C≡N) are characteristic functional groups of PAN. The interfacial interactions indicated by the changes in the O–H/N–H and C–O–C bands and the successful integration of PAN have the potential to enhance stress transfer between the resin matrix and the nanofibers, thus contributing to the improvement in physical and mechanical properties, as shown in Figures 1 and 2.

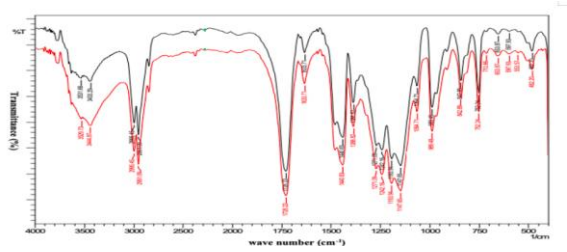

Figure 3. FTIR graphs for SC-ARnano (upper-black) and SC-ARc (lower-red) showed a similar pattern.

SC-ARc suffers from relatively low strength, which can lead to fracturing problems in prosthetic dentistry. Our results demonstrate that SC-ARnano effectively addresses these limitations, potentially extending the service life of biomedical appliances and reducing the need for frequent replacements. As shown in Figure 3, FTIR analysis provides definitive evidence of successful nanofiber incorporation without adverse chemical interactions. The modified spectra of the resin with nanofibers confirm successful incorporation and interaction of nanofibers in the resin matrix, not merely physically mixed but chemically interacting with the resin. Furthermore, the crack deflection and bridging mechanisms provided by the nanofibrous network, as shown in Figure 5, contribute to improved fracture toughness. When cracks propagate through the material, they encounter nanofibers that deflect the crack path and absorb energy through fiber pull-out and bridging mechanisms. This results in a more gradual failure mode rather than a catastrophic

brittle fracture, which is characteristic of unreinforced acrylic resins.[23-25]

Figure 3 shows the characteristic nitrile absorption band at 2260-2220 cm^{-1}, confirming that the PAN component of the nanofibers remains chemically intact within the resin structure. This is crucial for maintaining the nanofibers' reinforcing efficacy. The absence of significant peak shifts in the FTIR spectrum suggests good chemical compatibility between the nanofibers and the acrylic resin matrix. This compatibility is essential for preventing degradation reactions that could compromise the long-term stability of the resin material in the oral or body fluid environment. Understanding the controlled fabrication and properties of resin materials is required to overcome current limitations in biomedical applications. Our chemical characterization results support the feasibility of using enanoPAN-PMMA as biocompatible reinforcing agents for biomedical applications.[26]

Figure 4 presents the XRD analysis, which was conducted to investigate the crystalline structure and phase composition of both samples. The XRD results demonstrated that both samples exhibited similar amorphous patterns, indicating that the incorporation of nanofibers did not significantly alter the overall crystallographic structure of the resin matrix. The broad, diffuse peaks characteristic of amorphous polymers were observed in both samples, suggesting that the e-nanoPAN-PMMA are well distributed within the amorphous polymer network without inducing crystallization or phase separation.

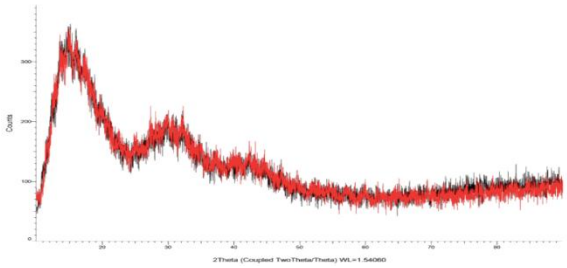

Figure 4. XRD graphs for SC-ARc (red) and SC-ARnano (black) showed a similar amorphous pattern.

Figure 4 shows that nanofiber incorporation does not disrupt the amorphous nature of the polymer matrix, which is beneficial for maintaining the optical properties and processability of the dental resin. The preservation of the amorphous structure ensures that the composite retains the aesthetic qualities required for biomedical appliances while gaining mechanical advantages. The similarity of the diffraction patterns across samples indicates excellent dispersion of e-nanoPAN-PMMA without agglomeration or phase separation. The nanofibers are most likely dispersed within the matrix, enhancing mechanical performance without creating new detectable crystalline peaks. This uniform distribution is critical for achieving consistent mechanical properties throughout the prosthetic device and avoiding weak spots that could lead to localized failures. The XRD patterns show that the polymer matrix remains predominantly amorphous after nanofiber incorporation, with no appearance of new crystalline peaks. This indicates that the nanofibers do not act as crystallization nuclei and are well dispersed within the polymer matrix without inducing phase separation or agglomeration. Maintaining an amorphous structure is crucial for preserving the optical clarity, processability, and dimensional stability of the resin. Consequently, the role of nanofibers in this system is associated with interfacial reinforcement and efficient stress transfer rather than changes in crystallinity, which is

consistent with the observed improvements in mechanical performance.[27]

SEM-based fractographic analysis was conducted on fractured specimens to examine failure mechanisms and visualize the distribution and orientation of nanofibers within the polymer matrix. Figure 5 illustrates the fracture surface morphology of both samples. The analysis provided clear evidence of nanofiber presence and distribution within the SC-ARnano. The nanofiber-reinforced sample showed distinct fibrous structures embedded in the polymer matrix, confirming the successful incorporation and dispersion of e-nanoPAN-PMMA. In contrast, the SC-ARc fracture surface displayed a typical smooth, brittle fracture pattern without fibrous reinforcement features.

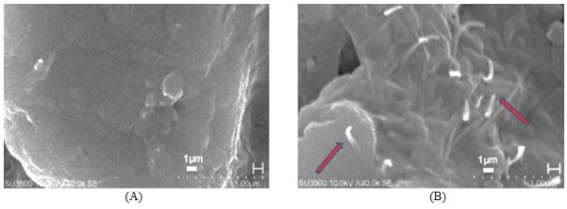

Figure 5. Fractured surface morphology of (A) SC-ARc and (B) SC-ARnano, with red arrows showing the appearance of nanofibers

The fractographic analysis demonstrates the effectiveness of nanofibers in altering the failure behavior of the resin material. The presence of visible fibers on the fracture surface of SC-ARnano, as shown in Figure 5, indicates good interfacial bonding and successful stress transfer from the matrix to the reinforcing fibers. Systematic reviews have shown that nanofibers as reinforcement significantly improve resin-based dental materials. The fiber pull-out and bridging mechanisms observed in our fracture analysis are consistent with effective reinforcement, in which fibers absorb fracture energy and prevent catastrophic crack propagation.[28-29]

A study evaluating the addition of agave sisalana and E-glass fibers to repaired acrylic denture bases found that both fiber types significantly increased impact strength compared with unreinforced acrylic resin. The authors reported that agave sisalana fiber at 3.3% produced greater impact strength than E-glass fiber, indicating that fiber reinforcement can effectively enhance acrylic resin's resistance to fracture. Similar to those findings, the current study found that incorporating 1 wt% electrospun PAN-PMMA nanofibers significantly improved the flexural strength and flexural modulus of self-cure acrylic resin. Although different fiber types and mechanical tests were used, both studies support the concept that fibers act as reinforcing agents by improving stress distribution within the resin matrix and reducing crack propagation. Unlike conventional microscale fibers such as agave sisalana and E-glass fibers, electrospun PAN-PMMA nanofibers have nanoscale diameters and a high surface-area-to-volume ratio. These characteristics likely contribute to the substantial enhancement in mechanical performance observed in the reinforced self-cure acrylic resin.[30]

Recent advances in digital dentistry and additive manufacturing have increased demand for materials suitable for the rapid fabrication of provisional and definitive prostheses. Self-cure acrylic resin offers advantages such as rapid polymerization, simple chairside manipulation, and reduced processing time. However, its relatively poor mechanical performance limits broader use in digitally assisted prosthodontics. Strengthening self-cure acrylic resin through nanofiber reinforcement may expand its potential use in customized and additively manufactured denture applications, where lightweight structures, dimensional stability, and fracture resistance are critical requirements.[31-32]

Based on the minimum value for the next improvement, this study was limited to a quantitative

1% e-nanoPAN-PMMA, consistent with the preliminary study and other studies' preferences. Future research should optimize nanofiber loading levels to maximize performance benefits while maintaining processability and cost-effectiveness for clinical applications, and should focus on long-term biocompatibility studies and clinical trials. In this study, the fabrication of SC-ARnano successfully improved the physical and mechanical properties of self-cure acrylic resin. These findings would provide new insights into the broader biomedical applications of SC-ARnano.

CONCLUSION

The flexural strength and flexural modulus of SC-ARnano increased by approximately 35% and 30%, respectively. FTIR confirmed the successful chemical integration of e-nanoPAN-PMMA within the resin matrix, while XRD showed that the amorphous structure. SEM revealed the elimination of surface porosity in SC-ARnano, reducing the risk of bacterial colonization and improving clinical suitability. These findings suggest that 1 w% e-nanoPAN-PMMA is an effective reinforcement concentration for enhancing SC-AR performance in denture applications.

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