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Nanoparticle Modification of Acrylic-Based Soft Denture Liner: Effects of Samarium Oxide, Boron Nitride and Titanium Dioxide on Surface, Chemical and Adhesive Properties

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Title page

Original Article

*** Title**

Nanoparticle modification of acrylic-based soft denture liner: effects of samarium oxide, boron nitride and titanium dioxide on surface, chemical and adhesive properties

*** Running title**

Nanoparticle-Modified Soft Denture Liners

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Nanoparticle Modification of Acrylic-Based Soft Denture Liner: Effects of Samarium Oxide, Boron Nitride and Titanium Dioxide on Surface, Chemical and Adhesive Properties

Abstract

Background: This in vitro study evaluated the effects of incorporating samarium oxide (Sm_2O_3), boron nitride (BN), and titanium dioxide (TiO_2) nanoparticles into an acrylic-based soft denture liner on residual monomer release, surface roughness, contact angle, and peel bond strength.

Methods: Ten groups were prepared: a control and groups containing each nanoparticle at 1, 2, and 5 wt%. Surface roughness (Ra) and contact angle were measured by confocal profilometry and optical tensiometry (n=10), and residual monomer release was determined by HPLC at 12, 24, and 72 hours (n=10). Peel bond strength was assessed by 180° peel testing according to ASTM D903-98 (n=8). Data were analyzed by one-way ANOVA with Tukey HSD test, and by two-way ANOVA for monomer release ($P<.05$).

Results: All nanoparticle groups released significantly less residual monomer than the control at all time points ($P<.05$). Surface roughness decreased significantly only in the Sm_2O_3-containing S1 and S2 groups. Contact angle was significantly higher than that of the control in most TiO_2 and Sm_2O_3 groups. All groups exceeded the clinically acceptable peel bond strength threshold; the highest value (S5, 1.461 ± 0.598 N/mm) did not differ significantly from that of the control.

Conclusion: The effects of nanoparticle incorporation depended on particle type and concentration. Sm_2O_3 showed a favorable overall profile, reducing residual monomer release and, at low and medium concentrations, surface roughness, while maintaining bond strength above the clinical threshold, and is a promising additive candidate for acrylic-based soft denture liners. Further research is needed.

Key words: Soft denture liner; Nanoparticle; Samarium oxide; Titanium dioxide; Boron nitride; Residual monomer

INTRODUCTION

Soft denture liners are widely used to improve the functional performance of removable dentures and to enhance patient comfort. Owing to their resilient structure, they absorb the forces generated during mastication and provide a balanced distribution of stress between the denture base and the supporting tissues.[1,2] They offer important clinical advantages, particularly in patients with severely resorbed alveolar ridges, thin or sensitive mucosal tissues, and pronounced anatomical undercuts.[3] However, the long-term clinical success of these materials is influenced by several factors, including residual monomer release, deterioration of surface properties, detachment from the denture base, microbial colonization, water sorption, and loss of mechanical durability.[1,4] Currently available soft denture liners are classified into two main groups: acrylic-based and silicone-based materials. Although silicone-based materials can maintain their elastic properties for a long time, inadequate bonding to the denture base may lead to clinical problems such as detachment and microbial colonization. Acrylic-based materials, in contrast, are distinguished by stronger bonding, low cost, and clinical availability. However, their clinical service life is limited by plasticizer loss, residual monomer release, progressive hardening, increased surface roughness, and the accompanying microbial retention.[5]

The use of nanoparticles in dental materials has increased in recent years.[6] The incorporation of nanoparticles into PMMA-based materials, resin cements, and composite resins has been reported to improve mechanical, surface, and antimicrobial properties.[7] However, data on the use of nanoparticles in soft denture liners are limited. In particular, to our knowledge, the effects of Sm_2O_3 incorporation on the properties of acrylic-based soft denture liners have not yet been investigated.

Sm_2O_3 is a rare earth metal oxide with high chemical stability that has been investigated for biomedical applications. Its incorporation into polymer matrices has been reported to influence surface and mechanical properties.[8] These characteristics make Sm_2O_3 a nanoparticle worth investigating for the modification of acrylic-based soft denture liners. Hexagonal boron nitride (h-BN), also known as “white graphene,” is a material with high thermal stability and low friction. Its incorporation into polymer matrices in nanoparticle form has been reported to improve surface and tribological properties.[9] TiO_2 is one of the most extensively investigated nanoparticles in dental materials because of its biocompatibility, antimicrobial properties, and effects on mechanical properties.[10-12]

The aim of this in vitro study was to evaluate the effects of incorporating Sm_2O_3, BN, and TiO_2 nanoparticles at different concentrations into an acrylic-based soft denture liner on residual monomer release, surface roughness, contact angle, and peel bond strength to acrylic denture base resin.

MATERIALS AND METHODS

Study design and experimental groups

In this in vitro study, a total of 10 groups, including a control group, were prepared by incorporating Sm_2O_3, TiO_2, and BN nanoparticles at 1, 2, and 5 wt% into an autopolymerizing acrylic-based soft denture liner (GC Tissue Conditioner; GC Corp., Tokyo, Japan) (Fig. 1). The sample size was calculated using G*Power software (α=.05, f=0.5). For residual monomer analysis, surface roughness, and contact angle measurements, 10 disc-shaped specimens were prepared per group (n=10/group); for the peel bond strength test, 8 rectangular specimens were prepared per group (n=8/group). In all experimental groups, the nanoparticles were added to the powder phase of the soft liner.

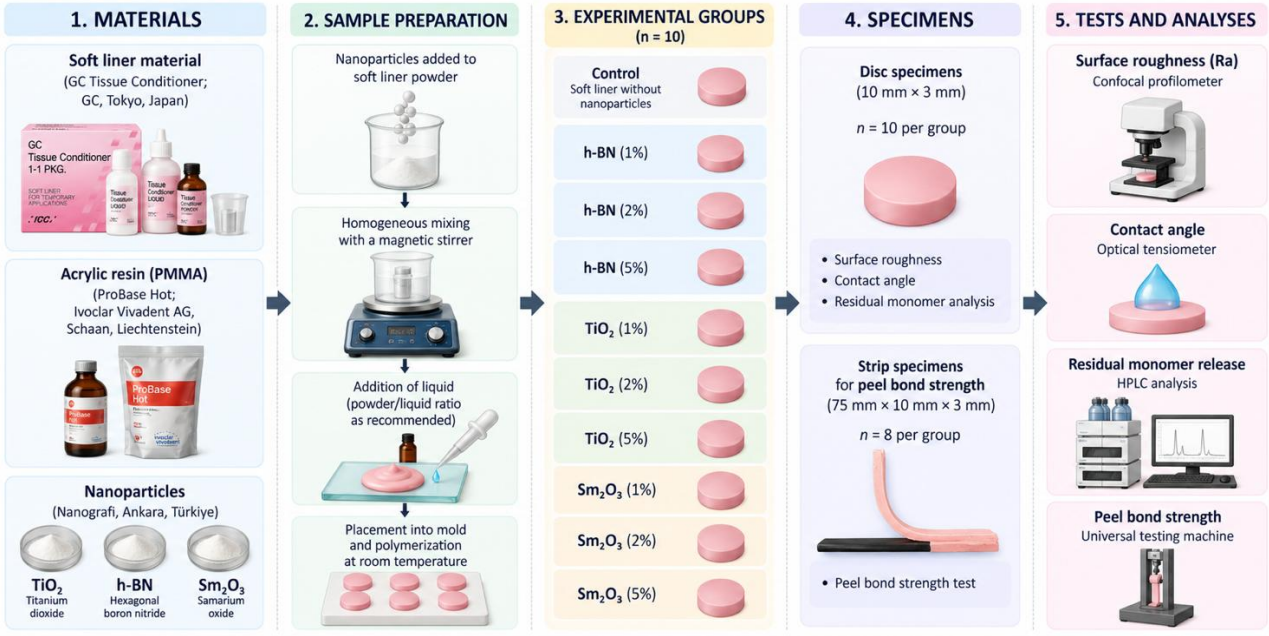


Fig. 1. Schematic overview of the study design. Sm_2O_3, TiO_2, or hexagonal BN nanoparticles (1, 2, and 5 wt%) were added to the powder of the soft denture liner, which was then mixed with the liquid and polymerized at room temperature to form 10 groups, including a control group. Disc specimens (n=10/group) were used for surface roughness, contact angle, and residual monomer analyses, and strip specimens (n=8/group) were used for the peel bond strength test. BN, boron nitride; T, TiO_2; S, Sm_2O_3; 1, 2, and 5 indicate nanoparticle concentrations of 1, 2, and 5 wt%.

Disc specimens

A silicone mold was used to prepare disc-shaped specimens 10 mm in diameter and 3 mm in thickness. The discs were used for residual monomer analysis, surface roughness, and contact angle measurements. In the control group, the powder and liquid were mixed at the manufacturer's recommended ratio and polymerized under a glass plate at room temperature for 5 minutes. In the experimental groups, Sm_2O_3 (99.99% purity, 325 mesh), TiO_2 (99.5% purity, 200 nm), and hexagonal BN (99.7% purity, 790 nm) nanoparticles (Nanografi, Ankara, Türkiye) were weighed using an analytical balance with 0.0001 g precision (XB 120A; Precisa Gravimetrics AG, Switzerland) and added to the soft liner powder. Each mixture was blended with a magnetic stirrer until homogeneous dispersion was achieved; the liquid was then added at the recommended ratio, and the mixture was placed in the mold and polymerized under a glass plate pressure at room temperature for 5 minutes.

Residual monomer analysis

After polymerization, the disc specimens were weighed with 0.0001 g precision and placed in separate amber glass vials. Twenty milliliters of extraction solution (80% methanol/20% distilled water) was added to each vial, and the specimens were incubated at 37°C in the dark. At 12, 24, and 72 hours, 1 mL of sample was withdrawn from each vial and replaced with an equal volume of fresh solution. The collected samples were stored at 4°C until analysis. Methyl methacrylate (MMA) concentration was determined using an HPLC system (LC-20AT; Shimadzu, Kyoto, Japan) equipped with a C18 column (Nacalai, 38019-81). The analytical parameters were as follows: mobile phase, 60% methanol/40% distilled water; flow rate, 0.20 mL/min; UV detection, 205 nm; column temperature, 25°C; and injection volume, 10 µL. A calibration curve was constructed using serial dilutions (0.005–1 mg/mL) prepared from a certified MMA reference standard (1000 µg/mL; Supelco, USA) (R^2=0.9959). MMA release per unit mass was calculated using the following formula:

MMA (mg/g) = Concentration (mg/mL) × Solution volume (mL) / Specimen weight (g)

Surface roughness measurement

Surface roughness was measured using a three-dimensional optical profilometer (Smartproof 5; Carl Zeiss, Oberkochen, Germany). Three randomized measurements were taken from the central region of each specimen by scanning a 500 µm × 500 µm area in fast mode at 10× magnification (Epiplan-

Apochromat 10×/0.7 DIC). The images were processed with the integrated software (ConfoMap ST 7.4.8076; Carl Zeiss), and mean surface roughness (Ra, μm) was calculated according to ISO 21920.

Contact angle measurement

Contact angle measurements were performed by the sessile drop method using an optical tensiometer (Attension Theta Lite; Biolin Scientific, Stockholm, Sweden). A 5 μL droplet of distilled water was placed on each specimen surface with a micropipette; the droplet image was recorded with a camera, and the contact angle (°) was calculated.

Rectangular specimens

For the peel bond strength test, acrylic denture base specimens measuring 75 × 10 × 3 mm were prepared with reference to ASTM D903-98(2025).[13] Heat-polymerized PMMA (ProBase Hot; Ivoclar Vivadent AG, Schaan, Liechtenstein) was prepared and polymerized according to the manufacturer's instructions. The specimen surfaces were standardized under water cooling using 200-, 400-, and 600-grit silicon carbide papers in sequence.

In the test setup, the area to which the soft liner would be bonded was standardized as 25 × 10 mm, and a silicone mold matching these dimensions was prepared. The acrylic surface outside the bonding area was covered with insulating tape. The mold cavity was filled with the soft liner material of the respective group and polymerized under a glass plate at room temperature. Before testing, the specimens were stored in distilled water at 37°C for 24 hours.

Peel bond strength test

The bond strength between the soft liner and the acrylic denture base was evaluated using a 180° peel test in accordance with ASTM D903-98. The 25-mm free end of the acrylic base that was not bonded and the 25-mm free end of the soft liner were fixed in the grips of a universal testing machine (LRX; Lloyd Instruments, AMETEK Inc., USA). The soft liner was peeled from the acrylic base surface at a 180° angle at a crosshead speed of 10 mm/min. Peel bond strength (N/mm) was calculated using the following formula:

$$PBS = F / W \times ([1 + \lambda] / 2 + 1)$$

where F is the maximum force (N), W is the specimen width (mm), and λ is the elongation ratio.

Statistical analysis

All statistical analyses were performed using IBM SPSS Statistics (v31.0; IBM Corp., Armonk, NY, USA). Data are reported as mean ± standard deviation (SD). Normality of data distribution was assessed using the Shapiro-Wilk test. Group comparisons were performed using one-way ANOVA followed by Tukey HSD post hoc multiple comparisons. Residual monomer release data were analyzed using two-way ANOVA to evaluate the main effects of formulation and time and their interaction. A two-tailed P value of less than .05 was considered statistically significant.

RESULTS

Residual monomer release

Residual monomer values were significantly lower than those of the control group at all three time points in all nanoparticle-containing groups ($P<.001$; Table 1, Fig. 2a). The highest release was observed in the control group at all time points, and the values increased steadily over time (12 h: 0.263 ± 0.012; 24 h: 0.333 ± 0.015; 72 h: 0.472 ± 0.022 mg/g). The lowest values varied according to time point and were observed in the BN1 group at 12 hours, the S1 group at 24 hours, and the T5 group at 72 hours. Among the Sm_2O_3 groups, S2 showed low and similar values at all three time points (0.052, 0.045, and 0.076 mg/g).

Surface roughness

The samarium oxide-containing S1 and S2 groups showed significantly lower Ra values than the control group and the groups containing BN and TiO_2 ($P<.05$; Table 2, Fig. 2b). The lowest Ra value was obtained in the S2 group (0.092 ± 0.018 μm), and the highest in the BN5 group (0.666 ± 0.086 μm). No statistically significant difference was found between the BN- and TiO_2-containing groups and the control group ($P>.05$).

Contact angle

The highest mean contact angles were found in the T2 (92.919 ± 5.160°) and T5 (90.724 ± 4.693°) groups, followed by the S1 (87.264 ± 6.650°) and S2 (84.572 ± 7.056°) groups. The control group had one of the lowest values (74.656 ± 5.703°). One-way ANOVA revealed a statistically significant difference among the groups ($P<.001$; Table 2). The Tukey HSD test showed that the T2 and T5 groups had significantly higher contact angles than the control, BN2, BN5, and T1 groups ($P<.05$).

Peel bond strength

The S5 group showed the highest mean bond strength (1.461 ± 0.598 N/mm) but did not differ significantly from the control group (P>.05). The lowest value was obtained in the T1 group (0.674 ± 0.314 N/mm). The BN5 group showed significantly higher bond strength than the T1, T5, and S2 groups, and the S5 group showed significantly higher bond strength than the T1, T5, S1, and S2 groups (P<.05; Table 2, Fig. 2c).

DISCUSSION

In this study, the incorporation of Sm_2O_3, TiO_2, and BN nanoparticles into an acrylic-based soft denture liner produced different effects on the evaluated parameters depending on nanoparticle type and concentration. The findings indicate that Sm_2O_3 had favorable effects on residual monomer release and surface roughness without compromising bond strength. TiO_2 and BN nanoparticles also significantly reduced residual monomer release compared with the control group but had no significant effect on surface roughness.

Residual monomer release is a critical parameter for the biocompatibility of acrylic-based materials, as it is related to both the physical and biological properties of the material. In addition to oral mucosal irritation and cytotoxic effects, residual monomer from incomplete polymerization has been associated with mechanical failure through its plasticizing effect.[14,15] In the present study, all nanoparticle-containing groups showed significantly lower residual monomer release than the control group at all three time points. This reduction may be attributed to the distribution of the nanoparticles within the polymer matrix and their restriction of free monomer diffusion.[16] The Sm_2O_3-containing groups showed significantly lower monomer release profiles than the control group (P<.05). It has been reported that Sm_2O_3 nanoparticles can exert their effects within an acrylic matrix through physical mechanisms without forming chemical bonds.[8] A similar mechanism may have contributed to the low monomer release observed in the present study. However, because this mechanism could not be evaluated directly, the finding needs to be confirmed by analyses capable of demonstrating chemical bonding at the molecular level, such as FTIR.

Rough surfaces have been reported to facilitate biofilm accumulation and *Candida* colonization.[17,18] In the present study, surface roughness was significantly reduced in the low- and medium-

concentration Sm_2O_3 groups (S1 and S2) compared with the control group. This reduction may be related to the chemical inertness of Sm_2O_3 nanoparticles, which may limit side reactions and thereby preserve matrix integrity.[12] The absence of a significant difference at the high concentration (S5) may be due to particle agglomeration; however, this assumption was not verified by SEM. The lack of a significant effect of TiO_2 on surface roughness differs from the findings of Naji et al., who reported a significant reduction in surface roughness in heat-polymerized acrylic resin-based resilient liner.[19] This difference may stem from differences in polymer network formation kinetics between autopolymerizing and heat-polymerizing systems. The lack of a significant effect of BN on surface roughness is consistent with the findings of Shittran et al.[20]

The significant increase in contact angle in most of the TiO_2- and Sm_2O_3-containing groups compared with the control group indicates that the surfaces of these groups became more hydrophobic. Increased surface hydrophobicity reduces the capacity of water molecules and water-soluble compounds to adhere to the surface; this may prevent the accumulation of organic debris and microorganisms and thus may have a favorable clinical effect.[21] Although an increase in contact angle would be expected to negatively affect bond strength according to classical adhesion theory,[22] this relationship did not appear to hold in the S5 group. It is thought that the distinctive adsorption behavior of Sm_2O_3 and its interaction with the polymer matrix may have compensated for the adverse effects of the reduction in surface energy.[23]

Regarding peel bond strength, the fact that all groups exceeded the clinical acceptance threshold reported by Craig and Gibbons[24] indicates that the nanoparticle modifications examined did not adversely affect bonding performance from a clinical standpoint. The highest numerical bond strength was observed in the S5 group; however, this value was not statistically significantly different from that of the control group. It has been reported that the addition of Y_2O_3, a rare earth oxide, increased tensile bond strength.[25] Although the tendency toward higher bond strength observed with high-concentration Sm_2O_3 in the present study is similar to this finding, it is not possible to conclude that Sm_2O_3 increased bond strength because statistical significance was not achieved. Nevertheless, this may suggest that rare earth oxides could improve bond strength without adversely affecting surface properties. The lack of change in bond strength in the TiO_2-containing groups may be explained by the limited interaction of this nanoparticle with the polymer matrix owing to its chemical inertness and is consistent with the literature.[26,27] The BN groups did

not show a statistically significant difference compared with the control group (P>.05). This finding differs from the results of Shittran et al., who reported a significant increase in shear and tear tests.[28] This may be due to the direction of the force applied by the test method used.

This study has several limitations. The in vitro design only partially reflects the long-term effects of the dynamic oral environment (e.g., salivary flow, pH and temperature fluctuations, and masticatory forces). The interaction mechanism of Sm_2O_3 with the polymer matrix and the dispersion of the nanoparticles were not directly evaluated by analyses such as FTIR and SEM-EDX. In addition, biocompatibility was assessed indirectly based on residual monomer data, and direct cell culture-based cytotoxicity tests were not performed. The findings should be supported by in vivo studies and long-term follow-up studies before they can be translated into clinical practice.

CONCLUSION

Within the limitations of this in vitro study, Sm_2O_3 showed a favorable overall performance by reducing residual monomer release and, at low and medium concentrations, surface roughness, while maintaining bond strength above the clinical acceptance threshold. These findings contribute to the gap in the literature regarding the potential of rare earth oxides in dental polymers and suggest that Sm_2O_3 may be a promising additive for acrylic-based soft denture liners.

Table 1. Residual monomer release (mg/g) of all groups at 12, 24, and 72 h (mean ± SD)

Group	Residual monomer release (mg/g)		
	12 h	24 h	72 h
Control	0.263 ± 0.012^{a}	0.333 ± 0.015^{a}	0.472 ± 0.022^{a}
BN1	0.016 ± 0.001^{g}	0.135 ± 0.008^{bc}	0.073 ± 0.004^{de}
BN2	0.075 ± 0.004^{d}	0.121 ± 0.007^{bc}	0.101 ± 0.006^{d}
BN5	0.151 ± 0.007^{b}	0.142 ± 0.006^{b}	0.069 ± 0.003^{de}
T1	0.031 ± 0.002^{f}	0.111 ± 0.006^{bc}	0.126 ± 0.007^{c}
T2	0.084 ± 0.004^{d}	0.099 ± 0.004^{c}	0.137 ± 0.006^{c}
T5	0.113 ± 0.004^{c}	0.143 ± 0.004^{b}	0.016 ± 0.001^{f}
S1	0.149 ± 0.004^{b}	0.034 ± 0.001^{d}	0.163 ± 0.005^{b}
S2	0.052 ± 0.002^{e}	0.045 ± 0.001^{d}	0.076 ± 0.002^{de}
S5	0.107 ± 0.003^{c}	0.149 ± 0.004^{b}	0.169 ± 0.004^{b}

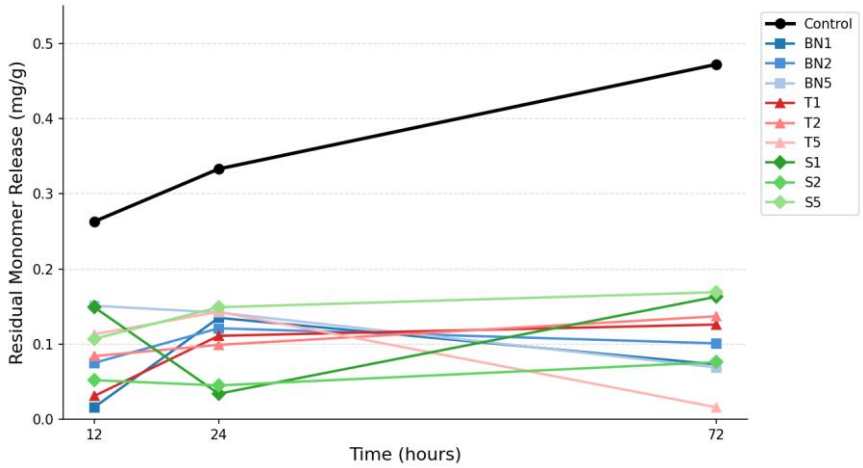
BN, boron nitride; T, TiO_2; S, Sm_2O_3; 1, 2, and 5 indicate nanoparticle concentrations of 1, 2, and 5 wt%. Within the same column, groups sharing the same superscript letter are not significantly different (Tukey HSD, $P>.05$). Two-way ANOVA: formulation F=2271.6, time F=374.5, formulation × time interaction F=329.4 (all $P<.001$).

Table 2. Surface roughness, contact angle, and peel bond strength of all groups (mean ± SD)

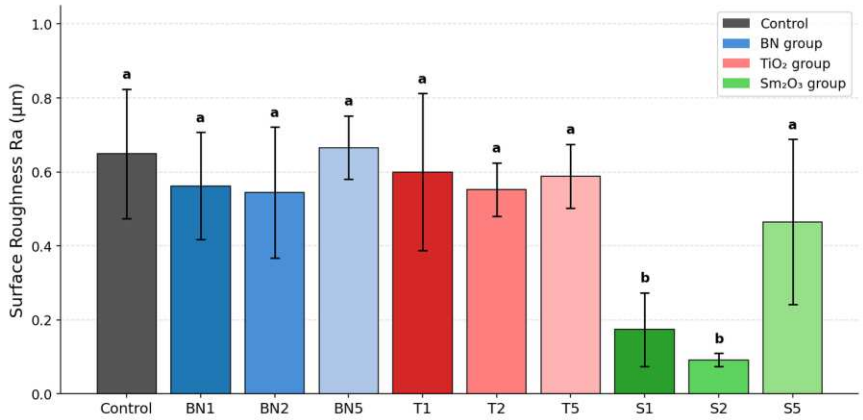
Group	Surface roughness, Ra (µm)	Contact angle (°)	Peel bond strength (N/mm)
Control	0.649 ± 0.175^{a}	74.656 ± 5.703^{d}	0.955 ± 0.242^{abc}
BN1	0.562 ± 0.145^{a}	83.133 ± 5.701^{bc}	0.907 ± 0.087^{abc}
BN2	0.544 ± 0.177^{a}	78.154 ± 5.570^{cd}	1.102 ± 0.309^{abc}
BN5	0.666 ± 0.086^{a}	74.052 ± 4.639^{d}	1.395 ± 0.464^{ab}
T1	0.600 ± 0.212^{a}	78.306 ± 5.506^{cd}	0.674 ± 0.314^{c}
T2	0.553 ± 0.072^{a}	92.919 ± 5.160^{a}	1.004 ± 0.252^{abc}
T5	0.589 ± 0.086^{a}	90.724 ± 4.693^{ab}	0.782 ± 0.329^{c}
S1	0.174 ± 0.100^{b}	87.264 ± 6.650^{ab}	0.880 ± 0.315^{bc}
S2	0.092 ± 0.018^{b}	84.572 ± 7.056^{bc}	0.788 ± 0.302^{c}
S5	0.465 ± 0.223^{a}	83.587 ± 5.282^{bc}	1.461 ± 0.598^{a}

BN, boron nitride; T, TiO_2; S, Sm_2O_3; 1, 2, and 5 indicate nanoparticle concentrations of 1, 2, and 5 wt%. Within the same column, groups sharing the same superscript letter are not significantly different (Tukey HSD, $P>.05$). One-way ANOVA: surface roughness F=18.647, contact angle F=13.033, peel bond strength F=4.484 (all $P<.001$).

(a)



(b)



(c)

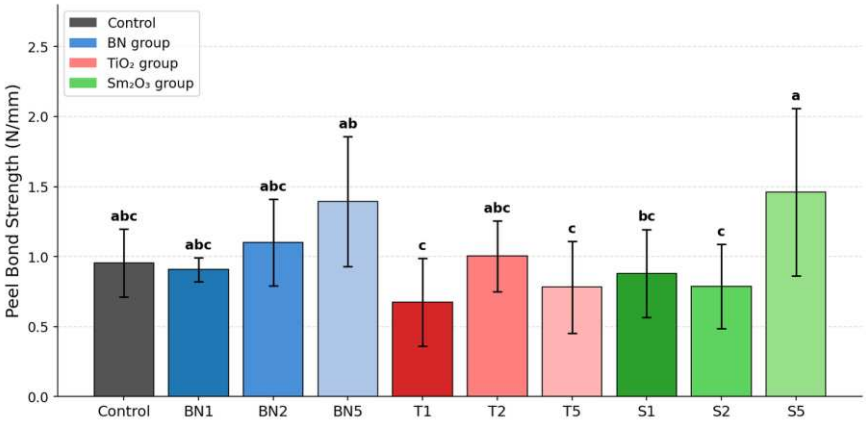


Fig. 2. (a) Residual monomer release over time for all groups (mean values at 12, 24, and 72 hours). All nanoparticle groups showed significantly lower release than the control group at all time points (P<.001). (b) Surface roughness (Ra, μm) of all groups (mean ± SD). (c) Peel bond strength (N/mm) of all groups (mean ± SD). In (b) and (c), different letters indicate statistically significant differences (Tukey HSD, P<.05).

DECLARATIONS

- **Ethics approval and consent to participate**

Not applicable.

- **Consent for publication**

Not applicable.

- **Availability of data and materials**

The datasets generated and analyzed during the current study are available from the corresponding author upon reasonable request.

- **Competing interests**

The authors declare there are no conflicts of interest relating to this study.

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- **Authors' contributions**

Berfu Erhan Güreser: Conceptualization, Methodology, Investigation, Formal analysis, Visualization, Writing – Original Draft. Cem Şahin: Conceptualization, Methodology, Supervision, Writing – Review & Editing.

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