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ORIGINAL RESEARCH

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Physical and mechanical characterizations of experimental pit and fissure sealants based on bioactive glasses

Aftab Ahmed Khan[1,*], Abdulaziz Abdullah Al-Khureif[1], Manal Al-Mutairi[2], Ibrahim Al-Majed[2], Sanam Aftab[3]

[1]Dental Health Department, College of Applied Medical Sciences, King Saud University, 11451 Riyadh, Saudi Arabia
[2]Pediatric Dentistry and Orthodontics Department, College of Dentistry, King Saud University, 11451 Riyadh, Saudi Arabia
[3]Community Health Sciences Department, College of Applied Medical Sciences, King Saud University, 11451 Riyadh, Saudi Arabia

***Correspondence**
aftkhan@ksu.edu.sa
(Aftab Ahmed Khan)

Abstract

Fissure sealants commonly exhibit weak mechanical and physical properties, potentially compromising their effectiveness in preventing dental caries. Therefore, this laboratory study aimed to synthesize and characterize experimental pit and fissure sealants in comparison with a commercially available sealant. Three different formulations of experimental pit and fissure sealing materials were synthesized using a blend of bisphenol A-glycidyl methacrylate (Bis-GMA) and triethylene glycol dimethacrylate (TEGDMA) monomers. The resin composition encompassed 70 wt.%, with 30 wt.% fillers comprising 10 wt.% silica in each composition, while 20 wt.% bioactive glasses. Specifically, group G1 employed Biomin F powder, group G2 utilized Biomin C powder, and group G3 incorporated S53P4 powder. The control group (GC) was a commercially available pit and fissure sealant (Seal-Rite). Ten disk-shaped specimens from each study group were fabricated (n = 10/group). The surface roughness, water contact angle, nanohardness (nH), elastic modulus (EM), water solubility and sorption were statistically evaluated using a one-way analysis of variance ($p < 0.05$). The surface roughness of the G1 & G2 groups and the water contact angle of G1, G2 & G3 groups were significantly lower compared to the control group ($p < 0.05$). However, nH, EM, water solubility, and sorption were notably higher in the control group compared to the experimental groups, except G1, which exhibited no significant difference from the control group ($p > 0.05$). The inclusion of micron-sized Biomin F powder in the experimental Bis-GMA/TEGDMA resin formulation demonstrated advantageous effects in reducing surface roughness and forming a lower contact angle without compromising the mechanical attributes.

Keywords

Bioactive glass; Caries; Pit and fissure sealant; Physical properties; Mechanical properties

1. Introduction

The prevalence of occlusal fissure dental caries can be attributed to the intricate morphological features that impede the effective removal of biofilm through routine tooth brushing [1]. Remarkably, it has been observed that roughly 90% of instances of dental carious lesions and cavities manifest within the fissures and pits located on the occlusal surfaces of both primary and permanent posterior teeth [1, 2]. Recognizing the critical significance of caries prevention during the developmental stages is imperative, as the consequences of caries can encompass speech impairments, aesthetic concerns, psychological ramifications, the development of irregular tongue habits, and deficiencies in masticatory function [3].

Indeed, there is a range of efficacious preventive measures, such as brushing, flossing, and fluoride treatment employed for the prevention. However, pit and fissure sealing, a strategy aimed at obstructing the advancement of cariogenic bacteria within the fissures of posterior teeth, is considered a cost-effective and painless procedure. Sealing occlusal surfaces yields the benefit of reduced caries incidence when compared to non-sealed teeth, while also presenting a cost-effective alternative to restorative procedures [4, 5]. Within the realm of sealants, two prominent variants are glass ionomer cement (GIC, based on acid-base reaction) and Resin-Based Sealants (RBSs, based on light or self-cure polymerization mechanism), both widely utilized in dental practice [6].

Amongst RBSs, filled and unfilled sealants are available based on the presence or absence of filler particles [7]. While RBSs have demonstrated satisfactory retention rates, the gradual loss of RBSs can be attributed to occlusal wear and shearing forces. The ability of the RBS to withstand these chewing

forces is contingent upon its physico-mechanical, chemical and biological characteristics [8]. Incorporating fillers at nanometer or micron scales has been explored to enhance the mechanical properties of RBS, as documented in the existing literature [9].

Bisphenol A-glycidyl methacrylate (Bis-GMA) enjoys extensive utilization in the fabrication of dental restoration materials due to their superior mechanical properties and notable advantages over dimethacrylate-based analogues including reduced shrinkage, adequate mechanical strength and robust adhesion [10]. However, its high viscosity presents a significant obstacle when attempting to integrate a substantial amount of filler material into the resin matrix [11]. Considering this, triethylene glycol dimethacrylate (TEGDMA) is used in the formulation to reduce the viscosity and improve the handling properties but the increasing content of TEGDMA induces polymerization shrinkage and the resultant shrinkage stress. Water solubility and sorption also increase and may affect the clinical life of restoration [12].

It is imperative to make alterations to both the organic and inorganic matrices to fine-tune the physical and mechanical characteristics of RBS. micron-sized particles, owing to their expansive surface area, significantly improve the interaction between the matrix and filler. The filler particles such as Biomin C, Biomin F and S53P4 are considered biocompatible with the ability to release calcium, phosphorus and fluoride ions. The prevention of a biofilm on restoration is essential otherwise biofilm accumulates bacterial colonization that eventually leads to demineralization of teeth, dental caries and other periodontal problems [13].

Evaluating physical and mechanical properties such as surface roughness, contact angle measurement, water solubility and sorption, nanohardness and elastic modulus safeguards dental materials to survive oral stresses, promoting durability and longevity of restorations. Therefore, the objective of this research was to formulate viable RBSs with a simplified production process for their application as a dental sealant to conduct an *in vitro* performance evaluation of these experimental RBSs and contrast them with a commercially available dental sealant (Seal-Rite). It was postulated that the formation of a new resin system would be compatible with the bioactive fillers for the improved physical and mechanical properties of RBS.

2. Materials and methods

The raw materials purchased from Sigma-Aldrich (Taufkirchen, Germany) and used for the synthesis of experimental formulations were triethylene glycol dimethacrylate (TEGDMA; >95%), 2-dimethylamino ethyl methacrylate (DMAEMA; 98%), camphorquinone (CQ; 97%), bisphenol A-glycidyl methacrylate (Bis-GMA; 98%), tetraethyl orthosilicate (>98%), silica fillers (0.5 μm in size). The glass fillers, *i.e.*, Biomin F, Biomin C and S53P4, were purchased from Cera Dynamic (Kent, England). The filler size was in the range of 5–25 μm. While the composition of commercially available pit and fissure sealant, *i.e.*, Seal-Rite could not be precisely determined due to the proprietary nature of the manufacturer's formulation. According to available information, Seal-Rite was composed of uncured methacrylate resins (66 wt.%), amorphous silica (5 wt.%) and sodium fluoride (2 wt.%).

2.1 Synthesis of polymer matrix

A blend of Bis-GMA and TEGDMA, as previously reported [10], was synthesized using a 2:3 weight ratio, respectively. The blend was mechanically mixed with an asymmetric double centrifuge (SpeedMixer™; DAC 150 SP Hauschild & Co. KG, Hamm, Germany) at 3000 rpm for 2 min. The blend was stored in dark containers at 8 °C to avoid premature polymerization.

2.2 Silanization of silica and bioactive glass fillers

The functionalization of silica fillers and bioactive glasses, *i.e.*, Biomin F, Biomin C and S53P4 was performed in a solvent mixture consisting of distilled water (40 mL), ethanol (250 mL), and ammonium hydroxide (25 mL) as previously reported [14]. The solution mixture was cooled, and then 45 mL of tetraethyl orthosilicate (>98% purity) was added drop wise over 4–6 minutes. Additional tetraethyl orthosilicate (TEOS) in ethanol was introduced, and the reaction continued for 8 hours. 3-methacryloxypropyltrimethoxysilane (MPS) coupling agent (10% vol) was stirred overnight. The resulting silica was separated by centrifugation, washed with ethanol, and vacuum-dried for 24 hours.

2.3 Preparation of experimental fissure sealant

For the preparation of the experimental formulation, 30 wt.% fillers were incorporated in the 69 wt.% experimental resin composition. The 0.2 wt.% of CQ and 0.8 wt.% of DMAEMA were mixed with a blend of Bis-GMA/TEGDMA. In Group 1 (G1), 20 wt.% of Biomin F with 10 wt.% of silica fillers were added and initially mixed manually with the blend of Bis-GMA/TEGDMA, followed by mechanical mixing with an asymmetric double centrifuge (SpeedMixer™; DAC 150 SP, Hauschild & Co. KG, Hamm, Germany) at 3000 rpm, three times with a two-minute gap in between. Similarly, Group 2 (G2) and Group 3 (G3) had 20 wt.% Biomin C and 20 wt.% S53P4, respectively in place of Biomin F in the composition. The composite mixture was again stored in dark containers at 8 °C for 24 h.

By using $\alpha = 0.05$ and $\beta = 80\%$, it was presumed to use 10 specimens/group. The specimens were prepared by carefully packing the experimental composite into the silicon mould by the same operator with uniform pressure to minimize variability to achieve uniformity across all specimens. The silicon mould dimensions (3 mm × 6 mm) were carefully measured using a digital calliper. The specimens of the control group (GC) were prepared with a widely used commercially available fissure sealant (Seal-Rite; Pulpdent Corp., USA). Specimens from the control and experimental groups were light cured with a device Elipar Freelight 2 (3M ESPE, Seefeld, Germany) with a 430–480 nm and light intensity of 900 mW/cm^2 for 30 s from the top and again 30 s from the bottom after removing from the mould. A total of ten specimens were prepared

from each formulation (Fig. 1). The specimens were stored in an incubator (IB-05G-60, Medline Scientific, Loughborough, UK) at 37 °C with 100% humidity for 24 h before testing.

2.4 Physical properties

A 3D optical non-contact surface profilometer (ContourGT, Bruker, Campbell, CA, USA) was employed to determine the surface roughness. The Sa (arithmetic average roughness) was determined through scanning white light interferometry, employing an objective lens with a 5× magnification. The scanning area was positioned at the central part of the specimen. With Vision64 (v 5.30) application software (Bruker, Campbell, CA, USA), the Sa value for each specimen was computed.

Water contact angle assessment was done by employing a Tensiometer (Theta Lite, Dyne Technology, Staffordshire, UK) to estimate variations in contact angle occurring due to the incorporation of different bioactive glasses along with SiO_2 filler in the experimental resin composite formulation. The contact angle of water was determined by dropping 3 μL over the specimen surface and gauging the angle formed by a water droplet on the specimen surface after a lapse of 20 s by using a camera connected to the tensiometer. The image was processed with the proprietary software.

For evaluation of Wsp and Wsol, specimens of the study groups (n = 10/group) were first desiccated using a desiccator (Vidrolabor, 76335L, Poá, SP, Brazil) having silica gel for 24 h. Next, the specimens were placed in an incubator at 37 °C for 72 h until a consistent mass was achieved. The precise electronic scale (Precisa, EP 320A; Dietikon, Switzerland) with an accuracy of 0.1 mg was first calibrated and utilized to measure the initial mass (m1). Next, the specimens underwent immersion in distilled water for 7 d, resulting in the determination of the wet mass (m2). Subsequently, they were again dehydrated in a desiccator for 24 hours to calculate the final drying mass (m3).

$$Wsp = 100 \times (m2 - m1)/m1 \quad (1)$$

$$Wsol = 100 \times (m1 - m3)/m1 \quad (2)$$

2.5 Mechanical properties

Nanohardness (nH) and elastic modulus (EM) tests were conducted using a nanomechanical tester (UMT1; Bruker, Campbell, CA, USA). The instrument was equipped with a diamond indenting nano tip having a radius of 100 nm. Before commencing the test, calibration was performed using a fused silica block to ensure accurate determination of the indenter area function and instrument compliance. The tests were carried out at room temperature with loading and unloading rates set at 2.0 mN/s and a 10 s dwell time at peak load. A maximum load of 20.0 mN was applied during the experiments. The nH and EM were computed using proprietary software by taking three measurements on each specimen for average reading (n = 10/group).

2.6 Statistical analysis

The acquired data underwent analysis utilizing Statistical Package for the Social Sciences software (SPSS ver. 28; IBM Corp., NY, USA). To compare groups with a 95% confidence level ($p < 0.05$), the one-way analysis of variance (ANOVA) followed by Bonferroni's *post hoc* tests were employed.

3. Results

Fig. 2 illustrates the mean surface roughness values (Sa, μm) for the study groups while Fig. 3 demonstrates the surface profiles of the study specimens. Notably, the GC group exhibited the highest surface roughness (3.63 ± 0.54 μm), followed by the G3 group (3.17 ± 0.18 μm) and then the G1 group (2.35 ± 0.21 μm). In contrast, the G2 group displayed the lowest surface roughness value (1.71 ± 0.03 μm). Significant differences were noted among the study groups ($p < 0.05$).

Fig. 4 depicts the average water contact angles recorded on the examined specimens. Notably, the GC group demonstrated

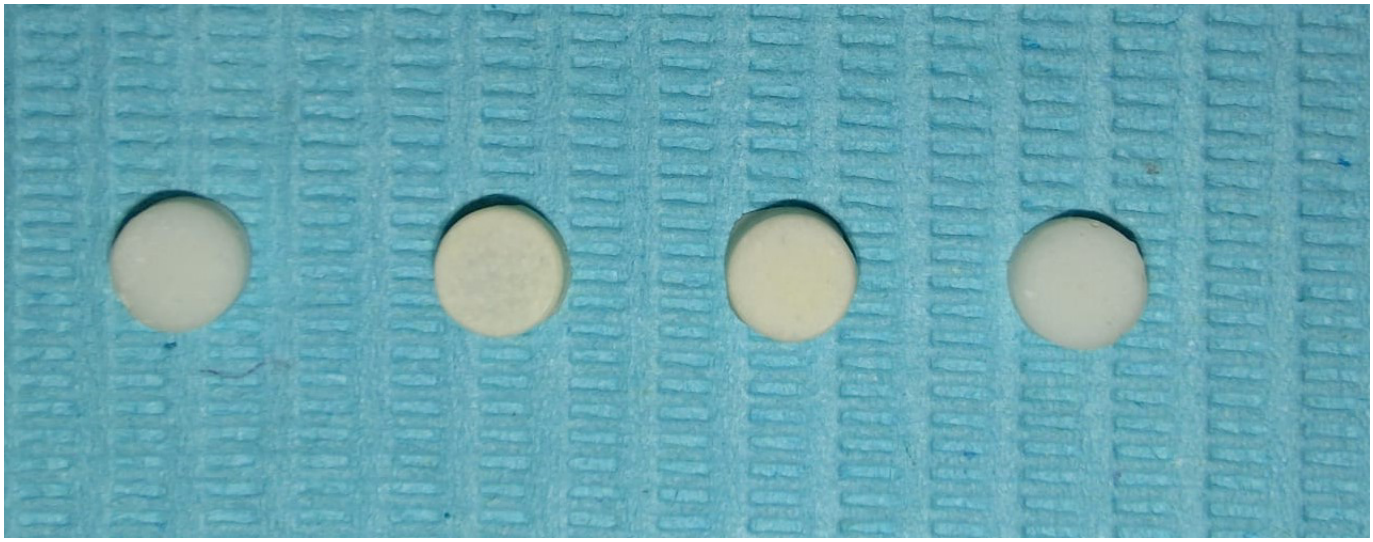

FIGURE 1. Disk-shaped specimens prepared from each formulation (from left to right: GC, G1, G2 and G3).

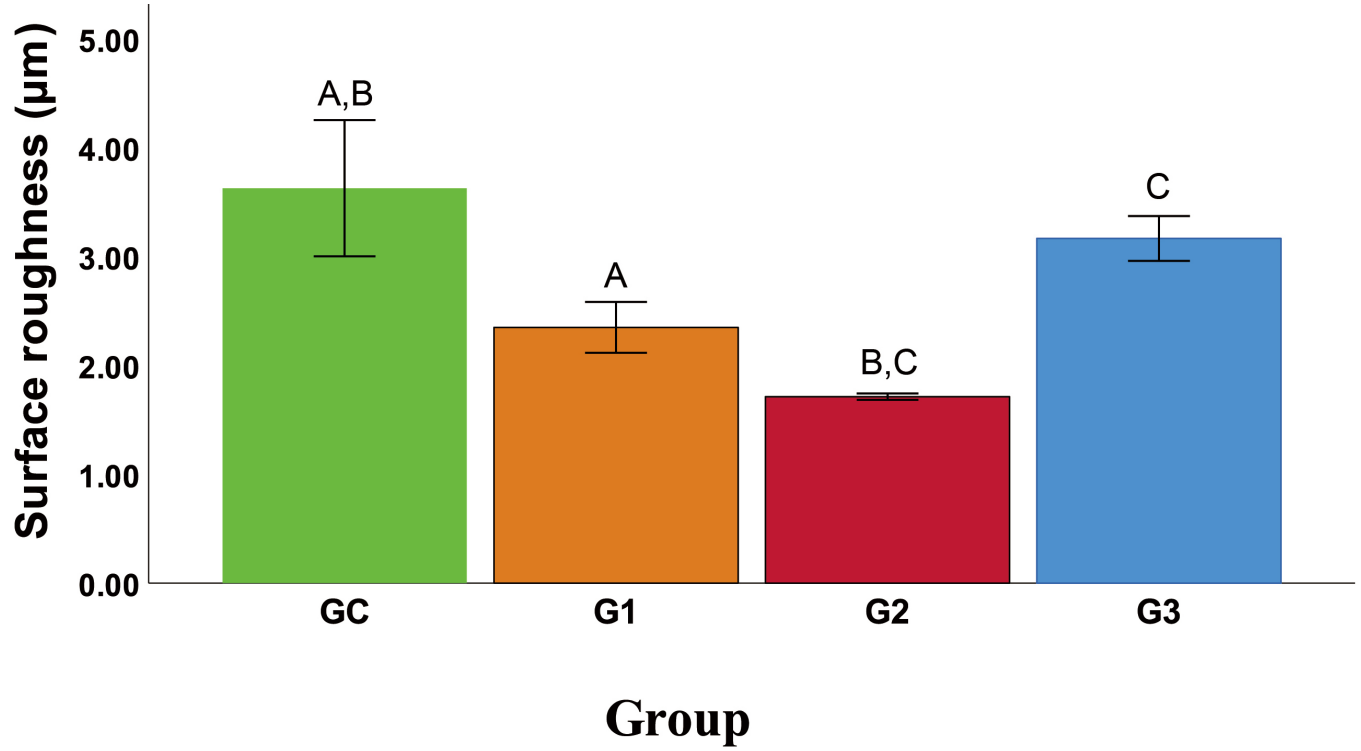

FIGURE 2. Mean surface roughness (Sa, μm) across study groups. The same uppercase alphabets depict significant differences between the groups.

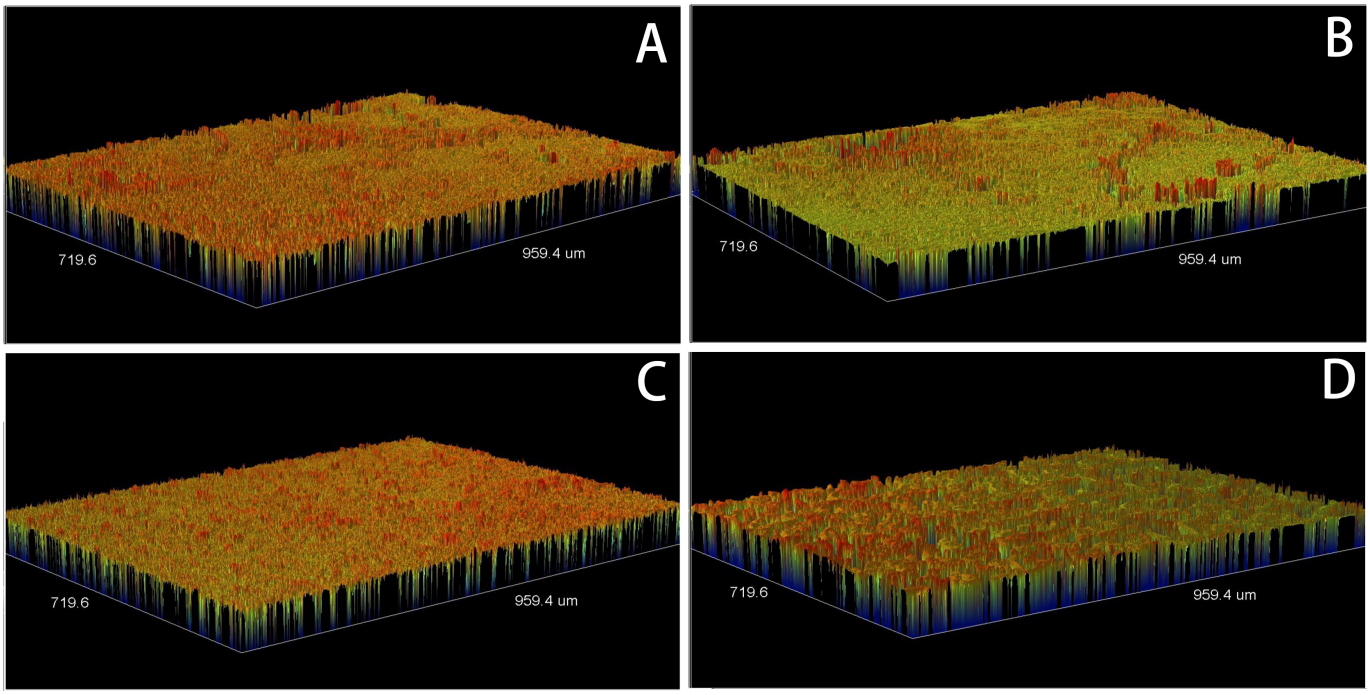

FIGURE 3. Surface profiles of the study specimens labelled A–D representing GC, G1, G2 and G3, respectively.

the greatest water contact angle (67.57 ± 1.76°), succeeded by the G1 group (57.33 ± 0.67°), and subsequently by G2 (46.67 ± 2.94°). In contrast, G3 exhibited the lowest contact angle (41.98 ± 1.15°). Statistically significant variations were noted among the study groups ($p < 0.05$).

Fig. 5 demonstrates the mean nH values of the study groups. The highest nH was observed in the GC group (0.42 ± 0.10 GPa) followed by the G1 group (0.33 ± 0.11 GPa) and then the G3 group (0.19 ± 0.07 GPa). The G2 group exhibited the lowest nH value of all (0.10 ± 0.02 GPa). Statistically significant variations were noted among the study groups ($p < 0.05$).

Fig. 6 demonstrates the mean EM values of the study groups. A similar trend as we observed in nH data was observed in EM. The highest EM was observed in the GC group (7.83 ± 2.26 GPa) followed by the G1 group (6.10 ± 2.43 GPa) and

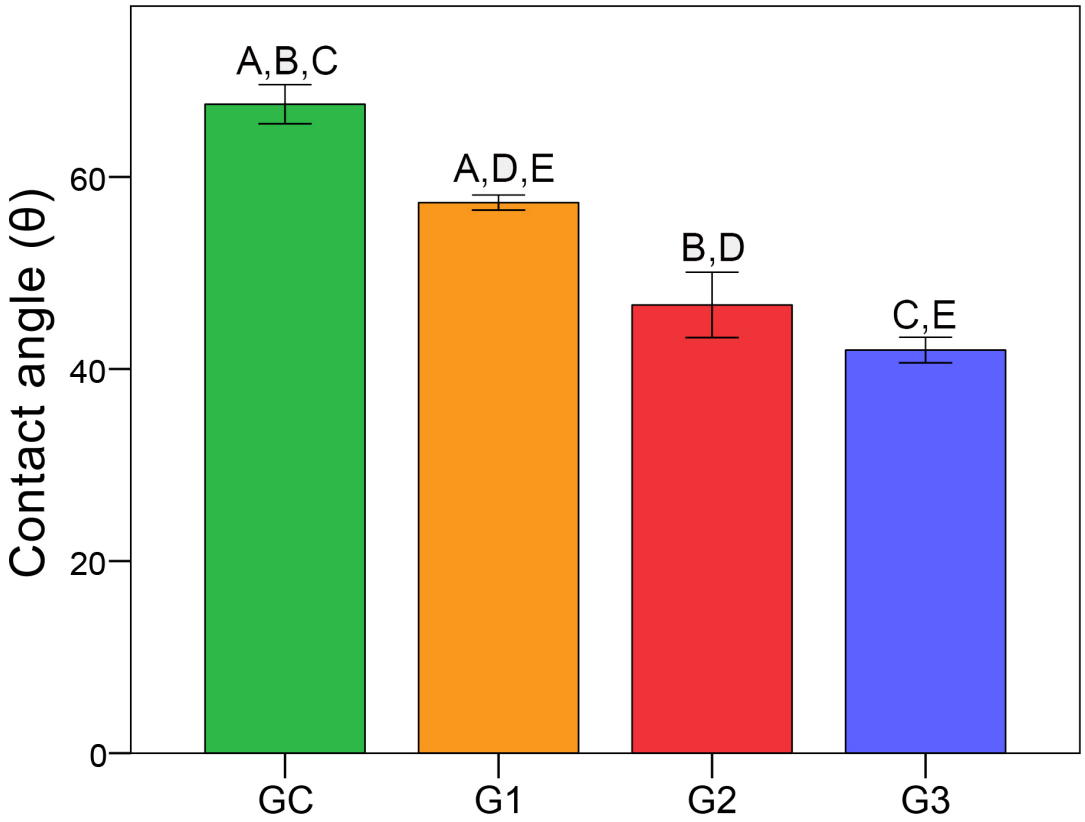

FIGURE 4. Mean water contact angle (θ, °) across study groups. The same uppercase alphabets depict significant differences between the groups.

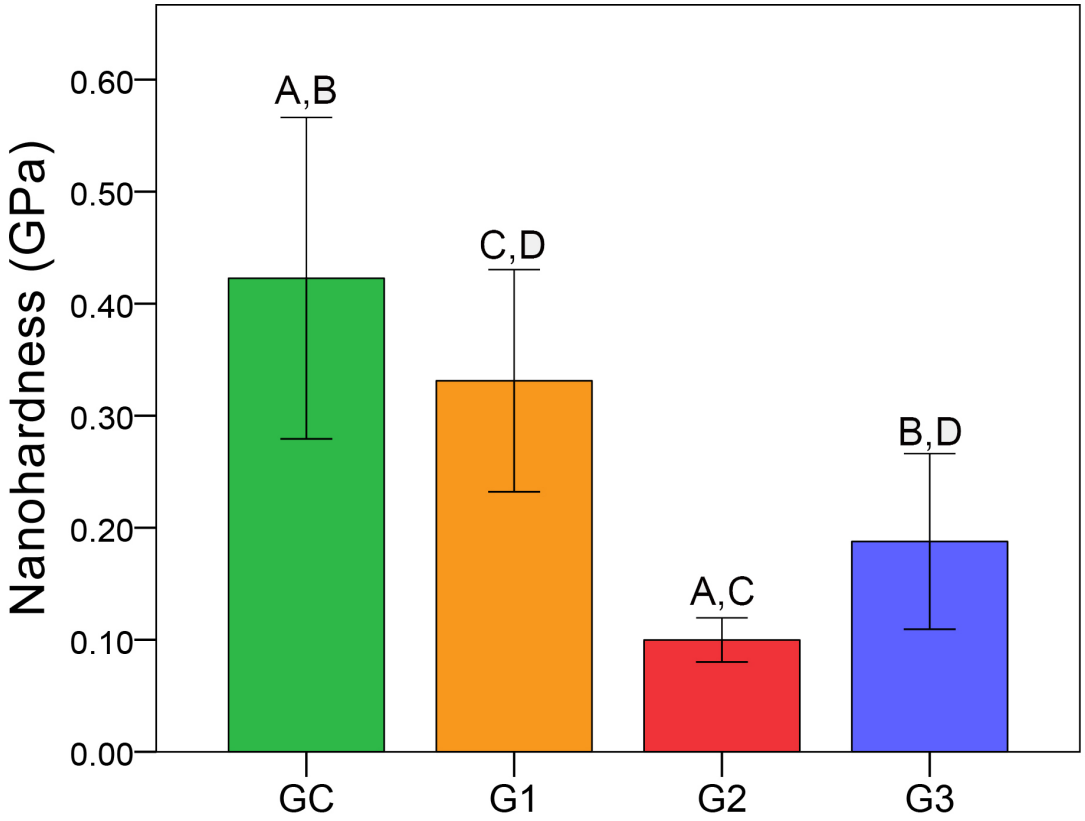

FIGURE 5. Mean nH (in GPa) across study groups. The same uppercase alphabets depict significant differences between the groups.

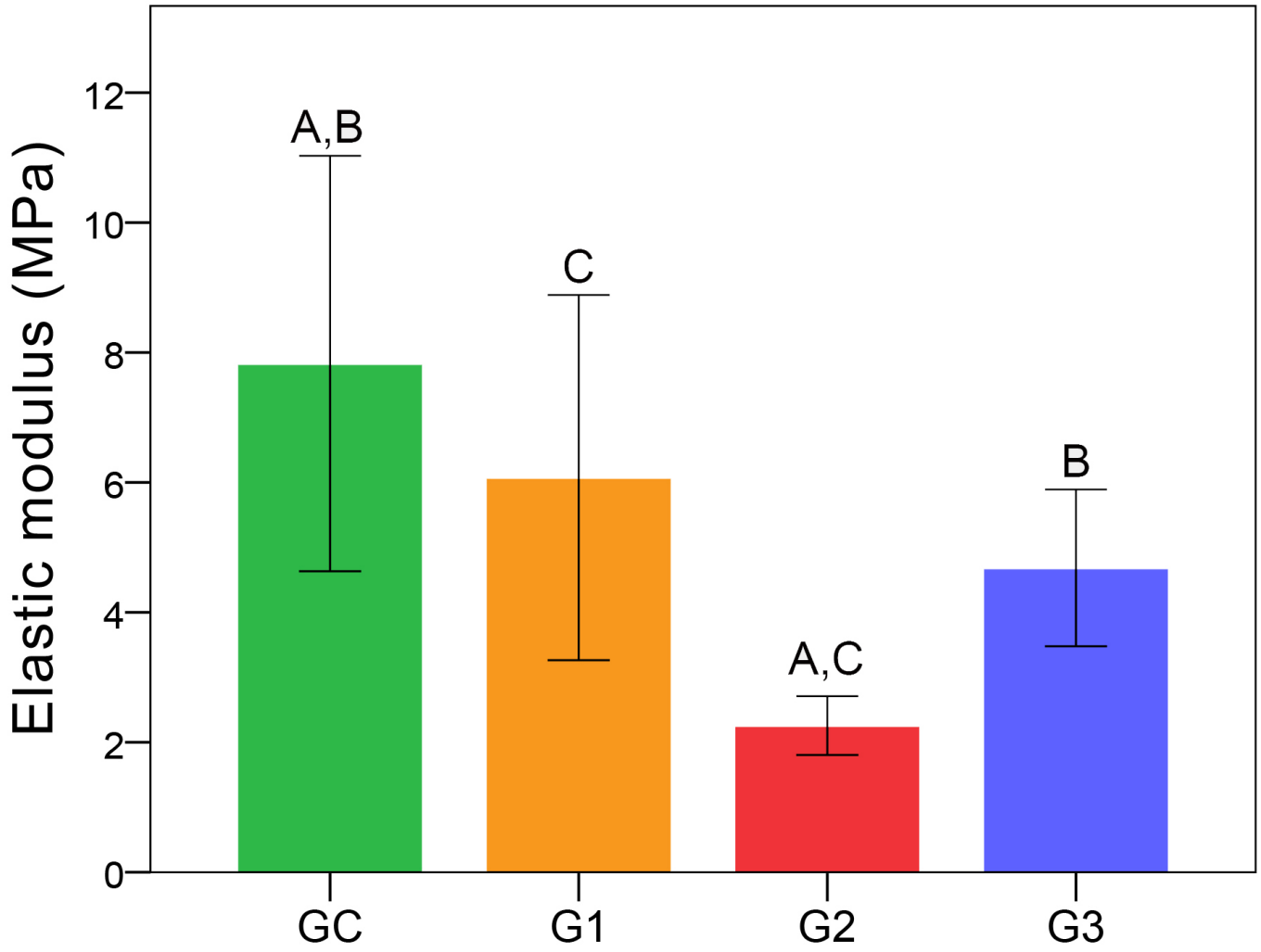

FIGURE 6. Mean EM (in GPa) across study groups. The same uppercase alphabets depict significant differences between the groups.

then the G3 group (4.68 ± 1.04 GPa). The G2 group exhibited the lowest EM value of all (2.25 ± 0.40 GPa). Statistically significant variations were noted among the study groups ($p < 0.05$).

Table 1 provides information on the water solubility and water sorption characteristics of the study groups. Among the water solubility test, the control group (GC) exhibited the lowest values (0.81 ± 0.02%) while G3 demonstrated the highest water solubility among the groups, with a value of 1.37%. For the water sorption test, all groups exhibited water loss and negative values. The highest water sorption was observed in GC (−0.43 ± 0.26%) while the lowest water sorption was observed in G3 (−1.73 ± 1.02%).

TABLE 1. Comparison of water solubility and water sorption (in %) for different groups (GC, G1, G2, G3).

Group	Water solubility (%)	Water sorption (%)
GC	$0.81 \pm 0.02^{A,B}$	−0.43 ± 0.26
G1	1.02 ± 0.17	−1.21 ± 0.84
G2	1.32 ± 0.37^{A}	−0.73 ± 0.61
G3	1.37 ± 0.41^{B}	−1.73 ± 1.02

Key: The same uppercase alphabets depict significant differences between the groups.

4. Discussion

This investigation aimed to synthesize and characterize a new resin-based pit and fissure sealant by incorporating it with different bioactive glass fillers for enhanced mechanical and physical attributes. The hypothesis put forth was partially validated, as various formulations of the experimental sealant exhibited enhancements in specific tested properties.

Surface roughness holds significant importance in the evaluation of material properties. Elevated surface roughness has been linked to the accumulation of biofilm and plaque, which can exacerbate caries lesions and periodontal disease [15]. Interestingly, the surface roughness of GC was unexpectedly higher when compared to the experimental formulations. Seal-Rite, a pit and fissure sealant based on urethane dimethacrylate, contains approximately 34.4% amorphous silica particles. While the precise particle size remains undisclosed by the manufacturer, the observed increase in surface roughness may be attributed to larger particle dimensions. In contrast, the experimental formulations utilized Bis-GMA and TEGDMA

monomers with fillers comprising both 5 μm silica particles (at 10 wt.%) and 20 wt.% bioactive glass particles (ranging from 5 to 25 μm). Notably, the lower surface roughness among experimental groups may be linked to the size and uniform distribution of filler particles, favouring a smoother surface finish [16]. Geometric properties, fraction area, and particle size distributions constitute significant components of surface morphology study. Higher granularity, usually linked to bigger particles, appears in surface imperfections or deeper surface fissures. The smaller particles yield finer textural characteristics. Roughness measures are often higher on surfaces with a wider range of particle sizes and a higher proportion area assigned to bigger particles [17].

The ability of dental materials to wet or adhere to tooth substrate is crucial for their functionality. Evaluating the water contact angle can assist in predicting how effectively a material will wet the tooth surface [18]. Though all the groups exhibited a contact angle less than 90°, however, lower contact angle amongst experimental groups compared to control (GC) indicates that silica and bioactive glasses were uniformly dispersed in the polymer network, filling the interstitial spaces among polymer particles without extending beyond the polymer surface [19]. Another factor could be the presence of TEGDMA monomer. TEGDMA is hydrophilic because it contains polyethylene and glycols in its chemical composition. TEGDMA is primarily used as a cross-linking agent, however, it tends to make the contact angle decrease significantly [20]. In addition, the bioactive glasses' hydroxyl groups and the supplementary salination process applied to both the bioactive glasses and silica particles likely introduced additional hydrophilic functional groups onto the filler surface [21] and therefore overall reduced water contact angles were observed amongst the experimental groups.

Assessing a material's microhardness is a vital aspect of its characterization. Yet, nanoindentation proves to be a valuable method for measuring the unit area of the indented surface on a nanoscale [22]. The statistically higher nH of the G1 group compared to other experimental groups, *i.e.*, G2 & G3 might be attributed to the hardness of Biomin F particles, *i.e.*, 4.5 GPa [23]. The reduced nH of G2 & G3 could be related to the incorporated powdered with decreased hardness. Whereas, the highest nH of the control group was because of the urethane dimethacrylate-based polymer (UDMA), which has a greater affinity for both chemical and physical crosslinking, and is entirely aliphatic. In contrast, Bis-GMA used in experimental groups is distinguished by its minimal crosslink density and the lowest degree of conversion within the polymer [24].

Similar trends in EM results were identified, aligning closely with the patterns observed in the nH data. The achieved control group (GC) result with the highest EM is justified as it contained a UDMA-based polymer matrix that has a higher crosslinking density due to its chemical structure, leading to a stiffer and more rigid polymer network compared to Bis-GMA. The complete composition formulation of Seal-Rite is not explicitly disclosed in the material safety data sheet, and information regarding additional copolymers utilized in its formulation is not available. The manufacturers commonly safeguard proprietary information pertaining to the precise composition of their products. The control group with UDMA monomer has a molecular weight of 470 g/mol, which might have contributed to increased intermolecular forces and higher modulus [25]. In contrast, Bis-GMA's molecular weight is 512 g/mol, though higher than UDMA, however the use of 60 wt.% TEGDMA monomer (286.32 g/mol) in experimental formulations might have lowered the overall stiffness of the experimental compositions [26].

The increase in water solubility from Gc to G3 could be due to differences in composition, notably the presence of Bis-GMA and TEGDMA in G1, G2 and G3, as opposed to UDMA monomer in Gc. The experimental formulations utilized monomers characterized by a 2:3 weight ratio of Bis-GMA and TEGDMA. TEGDMA is commonly incorporated into formulations with a twofold purpose: reducing the initial viscosity of the resin and assisting in the polymer's cross-linking process. The reduction in viscosity serves to enhance wetting characteristics during the application of the resin, while the cross-linking process contributes to the enhancement of physical properties, including resistance to solvents [27]. However, the water sorption of TEGDMA monomer is reported to be higher than that of Bis-GMA [28]. The inclusion of 60 wt.% TEGDMA in the polymeric formulation enhances water sorption and concurrently diminishes mechanical properties [29], specifically nH and EM.

The overall achieved results suggest that Biomin F powder can be a potential reinforcing agent for synthesising experimental pit and fissure sealants. However, diminutive reduction in nH and EM properties and insignificant elevation in water solubility and sorption could be attributed to the polymeric content of the experimental formulation in which 60 wt.% of TEGDMA was used. Although the current formulation achieved optimal physical properties in all the experimental compositions, however, optimal mechanical properties might be achieved by reducing the weight ratio of TEGDMA.

One of the limitations in this laboratory investigation pertains to the uniform resin-based composition utilized in all experimental formulations with variations in filler composition only. For scientific understanding or practical applications of experimental formulation, different aspects of the material under investigation are mandatory. This laboratory study was conducted in a simplified and controlled environment with relatively short observation periods, which might not fully replicate the dynamic oral conditions. For future work, it is postulated that with the variation of TEGDMA wt.% in the Bis-GMA matrix, the optimal composition with enhanced nH and EM can be achieved. Additionally, the laboratory studies related to the adhesion aspect and rheological properties of experimental sealants would be interesting to evaluate.

5. Conclusions

Although, the experimental formulations of G2 and G3 demonstrated reduced nH and EM with increased water solubility and sorption compared to the control group (GC). However, the experimental formulation with Bimon F (G1 group) demonstrated an insignificant difference compared to the control group (GC) in terms of nH, EM, water solubility and sorption. Overall, the surface roughness and water contact angle proper-

ties were significantly improved in G1 and most of the experimental formulations compared to Gc. The observed promising results of this study suggest further validation through biological properties evaluation and clinical trials.

AVAILABILITY OF DATA AND MATERIALS

The data presented in this study are available on reasonable request from the corresponding author.

AUTHOR CONTRIBUTIONS

AAK and AAAK—conceived and designed the research study; wrote the manuscript. MAM and IAM—performed the research. MAM and SA—analyzed the data. IAM and SA—prepared figures and tables. All authors read and approved the final manuscript.

ETHICS APPROVAL AND CONSENT TO PARTICIPATE

The study has received an ethical approval exemption from the King Saud University Institutional Review Board (IRB).

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CONFLICT OF INTEREST

The authors declare no conflict of interest.

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