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Physicomechanical Properties of Resin-Based Pit and Fissure Sealants Reinforced with Rice Husk Derived Nano Silica and Nanohydroxyapatite

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ABSTRACT

Resin-based pit fissure sealants (RBS) are used to prevent occlusal caries in children. The success of RBS in preventing dental caries is largely influenced by its retention on the tooth surface, which is also affected by its physicomechanical properties. The physicomechanical properties of RBS can be enhanced through the addition of fillers. With the advent of nanofillers, the physicomechanical properties were improved without altering RBS flowability. The present study developed an RBS with a 70 wt% resin matrix and 30 wt% nanofillers. The resin matrix consisted of urethane dimethacrylate (55 wt%), triethylene glycol dimethacrylate (45 wt%), camphoroquinone (0.3 wt%), and 2-(dimethylamino) ethyl methacrylate (0.7 wt%). Silane-treated rice husk-derived nanosilica (20 wt%) and nanohydroxyapatite (10 wt%) were added as fillers. Clinpro, Fissurit FX, and an unfilled sealant were controls. All RBS were tested for surface roughness, Vickers hardness, flexural strength, and flowability. Statistical analysis with one-way ANOVA revealed significant differences between groups in surface roughness, hardness, flowability ($p < 0.001$), flexural strength, and flexural modulus ($p < 0.05$). Experimental sealants had higher flexural strength (78 MPa) and flow distance (29.05+1.16 mm) than commercial controls. However, the surface roughness of experimental sealants (0.25+0.08 μm) was higher than Clinpro (0.087+0.027 μm) but lesser than Fissurit FX (0.35+0.19 μm). The Vickers hardness of experimental sealants (23+1.63 VHN) was less than Fissurit FX (28.80+1.69 VHN) but higher than Clinpro (21.74+1.68 VHN). This novel RBS had physicomechanical properties comparable to commercial sealants. The use of nanosilica from rice husk makes this pit and fissure sealer sustainable and environmentally friendly in dentistry.

Keywords: *Dental sealants; nanohydroxyapatite; physicomechanical properties; pit and fissure sealants; rice husk nanohybrid silica*

INTRODUCTION

According to the most recent Global Oral Health Status Report by the World Health Organisation, 3.5 billion people suffer from oral diseases, including 2.5 billion with untreated dental caries. Two billion adults suffer from caries of their permanent teeth, while 514 million children have caries of their primary teeth (Jain *et al.*, 2023). Biofilm formation that causes dental caries is a natural occurrence (Baelum *et al.*, 2008). However, inhibiting its metabolic activity can prevent the onset and progression of caries. Particularly important is the prevention of occlusal caries on permanent molars, as the initiation of caries occurs shortly after their eruption in the oral cavity (Carvalho *et al.*, 1989; Ekstrand *et al.*, 2000; Ferreira Zandoná *et al.*, 2012). The occlusal surfaces are considered one of the stagnation areas of the tooth that promote plaque accumulation and microbial development (Fejerskov & Thylstrup, 1994). During the first eruption of permanent molars, children have limited mechanical oral function for plaque elimination, which multiplies the risk of dental caries (Carvalho, 2014). The sealing of stagnant areas, which are the occlusal pit fissures, acts as a mechanical barrier for plaque and detritus collection, thereby preventing the initiation and progression of non-cavitated initial carious lesions.

The most common pit and fissure sealants are resin-based pit fissure sealants (RBS) and glass ionomer sealants. RBS is the recommended material because of its durability and longer retention. According to data from randomised controlled trials reported in the American Academy of Pediatric Dentistry Guidelines, glass ionomer sealant retention was five times lower than RBS after two years of follow-up (Wright *et al.*, 2016). The retention of sealant is critical to the prevention of caries. Aside from operator skill, fissure shape, and tooth position, the sealant material also has an impact on sealant retention (Muller-Bolla *et al.*, 2006; Azarpazhooh & Main, 2008; Hevinga *et al.*, 2010). Since the advent of pit and fissure sealants six decades ago, sealant materials have made remarkable progress in improving their retention on tooth surfaces. Sealant retention is influenced by physicomechanical factors such as polymerisation shrinkage, water sorption, occlusal force deflection, and wear characteristics (Osorio *et al.*, 2007).

Fillers are added to RBS to improve its physico-mechanical characteristics. Recently, there has been a lot of interest in using nanomaterials as fillers. Their physicomechanical characteristics are said to be improved by their reduced size and high surface area (Utneja *et al.*, 2018; Bohns *et al.*, 2019; Hesaraki *et al.*, 2020). Nanofilled RBS was recently the subject of a randomised clinical experiment that showed superior retention compared to traditional sealants (Kamath *et al.*, 2022). According to a recent systematic review, the intrinsic nature, morphology, volume, and concentration of the nanomaterial employed, as well as their interaction with the resin matrix, all had an impact on how well nanoparticle fillers enhanced the physicomechanical properties of RBS (Yassin *et al.*, 2023). Adequate flow of RBS is essential for adequate sealing at the margins of pits and fissures. Low-viscous RBS can penetrate deeper than the etched depth to create a resin-filled layer in the enamel with enough marginal adaptation (Irinoda *et al.*, 2000; Prabhakar *et al.*, 2011). Furthermore, it is reported that the in-depth flow of low-viscous RBS tends to retain itself even though the occlusal part of the sealant is lost after a certain period. This quality of low-viscous sealant is thought to prevent caries even in cases of partial loss of sealants (Kakaboura *et al.*, 2002). Earlier, fillers were added to enhance the physicomechanical properties of sealants, but this affected their flow. However, with the advent of nanofillers and resin sealants, physicomechanical properties are now enhanced without compromising their flow. Fillers in currently available sealants include fumed silica, silicate glass, inorganic glass ionomers, zirconia, and surface pre-reacted glass ionomers (Yaşa *et al.*, 2023). None of

these are made from bio-based components. The use of bio-derived substances has unique benefits as they are inexhaustible and environmentally safe (Zulkifli *et al.*, 2013; Yusoff *et al.*, 2019). Rice husk-derived nanohybrid silica is one such biobased material that has displayed promising results as a filler in earlier studies (Noushad *et al.*, 2016; Yusoff *et al.*, 2019). A recent review of rice husk-based dental composite resin recommended the incorporation of regenerative and antibacterial agents to prevent secondary caries and increase the longevity of restoration (Lin *et al.*, 2022). Hence, nanohydroxyapatite (nHA) was used as a bioactive filler in the present study. The bioactive and biocompatible nature of nHA has led to an interest in including it in various dental biomaterials. The use of nHA in dental resin composites and RBS is found to improve the mechanical properties besides being bioactive (Memarpour *et al.*, 2019; Yadav & Kumar, 2020; Yadav & Gangwar, 2020; Netalkar *et al.*, 2021). Thus, combining biobased and bioactive fillers in a RBS might enhance physicomechanical properties. Therefore, the aim of the present study was to develop an RBS comprising rice husk-derived nanohybrid silica and nHA and compare its physicomechanical properties to commercial pit and fissure sealants.

MATERIAL AND METHODS

Materials

In the present study, the materials used were urethane dimethacrylate (UDMA) (Aldrich, USA) as a base monomer, triethylene glycol dimethacrylate (TEGDMA) (Aldrich, USA) as a diluent monomer, camphoroquinone (CQ) (Merck, Germany) as a photo initiator, 2-(dimethyl amino) ethyl methacrylate (Merck, Germany) as an accelerator, and 3-(trimethoxysilyl) propyl methacrylate (γ-MPS) (Merck, Germany) as a coupling agent. Nanohybrid silica and nHA (SIGMA-ALDRICH) were used for reinforcement. Nanohybrid silica was synthesised as described by Noushad *et al.* (2016). The commercial sealants Clinpro (3M ESPE, USA) and Fissurit FX (VOCO, Germany) were used as commercial controls (positive controls), while the experimental RBS without fillers (EUS) was used as an experimental control (negative control). The details of the study group are presented in Table 1.

Table1 Details of pit and fissure sealants studied

Types of pit and fissure sealant studied	Composition
Clinpro (3M ESPE, USA) (positive control)	Bis-GMA; TEGDMA; CQ; Iodonium salt; Titanium di oxide; Silane-treated fumed silica; Organic fluoride salt; Rose Bengal dye
Fissurit FX (VOCO, Germany) (positive control)	UDMA; TEGDMA; Bis-EMA; Bis-GMA; Fillers 55 wt%; Sodium fluoride
Experimental RBS without fillers (EUS) (negative control)	UDMA; TEGDMA; CQ; DMAEMA
Experimental filled sealant (EFS)	UDMA; TEGDMA; CQ; DMAEMA Rice husk nanosilica; Nanohydroxyapatite

Notes: Bis-GMA: bisphenol A-diglycidyl methacrylate; UDMA: urethane dimethacrylate; TEGDMA: triethyleneglycol dimethacrylate; Bis-EMA: bisphenol A diglycidyl methacrylate ethoxylated; CQ: Camphoroquinone; DMAEMA: 2-(Dimethylamino) ethyl methacrylate).

Characterisation of Filler Particles

The morphology of nanosilica and nHA fillers was studied using a field emission scanning electron microscope (Quanta FEG 450, FE) operating at 5 kV in low vacuum.

Fabrication of Resin Based Pit and Fissure Sealant

The experimental filled sealant (EFS) was made of 70% resin matrix, 20% nanohybrid silica from rice husks, and 10% commercial nHA. Nanohybrid silica and nHA fillers were silanized, as previously reported (Noushad *et al.*, 2016). Initially, UDMA (55 wt%) was manually combined with TEGDMA (45 wt%) in a container. Camphoroquinone (0.3 wt%) and 2-(dimethyl amino) ethyl

methacrylate (0.7 wt%) were added to the resinous mixture to initiate and accelerate the polymerisation process. To prevent pre-polymerisation, we placed an aluminium foil cover over the container. The mixture was then agitated in a magnetic stirrer (VELP Scientifica Srl, Italy) for 1 h. Silanised nanohybrid silica and nHA were added to the resinous mixture in increments at room temperature and well stirred with a plastic spatula to make a homogeneous mix. The sealant paste was then transferred to plastic tubes and coated in aluminium foil before being kept in a cool, dry place.

Flexural Strength and Flexural Modulus

Five bar-shaped specimens (n = 5) measuring 25 × 2 × 2 mm were made using a spilt stainless-steel mold in accordance with ISO 4049 requirements. A glass slab and a mylar strip were placed beneath the stainless-steel mold, and the sealants were extruded into the mold. Another mylar strip and glass slab were placed on top and pushed to remove any air bubbles. The commercial sealants were light-cured according to the manufacturer's specifications. The experimental sealants were cured for 20 sec. The light curing unit head diameter was 8.5 mm; therefore, the samples were subjected to three overlapping irradiations on both top and bottom surfaces using the light curing unit (BLUEDENT LED Smart). Using a radiometer (CURE RITE, Dentsply Caulk, USA), the output light intensity was maintained at 1200 mW/cm2 throughout the course of the experiment. The cured specimens were kept in deionised water for 24 h. The specimen's dimensions were then measured with a digital calliper (Mitutoyo, Japan). Flexural strength and modulus were assessed using a three-point bending test with a span of 20 mm (SHIMADZU, AG-X plus) at a crosshead speed of 1 mm/min. The flexural strength and modulus were calculated based on the following equations:

$$\text{Flexural strength} = \frac{3PL}{2bt^2}$$

$$\text{Flexural modulus} = \frac{L^3m}{4bt^3}$$

Whereby P = maximum force applied, L = Span length, b = width, t = thickness, m = slope of initial straight line deflection curve.

Surface Roughness

Five specimens (n = 5) were prepared in a 5-mm-diameter, 2-mm-thick acrylic mold. A two-dimensional surface profilometer (SURFCOM FLEX-50A, ACCRETECH, Japan) was used to measure surface irregularity. The cut-off value, evaluation length, and measure speed were respectively set to 0.8 mm, 2 mm, and 0.15 mm/s. The roughness parameter was calculated as the arithmetic mean of the roughness profile values, Ra.

Vickers Hardness

Five specimens (n = 5) were prepared using a 5 mm diameter and 2 mm thick acrylic mold and preserved in distilled water for 24 h at 37^0C. The hardness was measured using a Vickers hardness tester (Model VM 50, FIE, India) with a 1 kg load and a dwell duration of 15 sec. Each specimen received three indentations.

Flow Distance

The flow distance of each test sealant was determined by dispensing 0.1 mL of sealant onto a glass plate that was held at 90^0. The dispensed sealant was allowed to flow for one minute. Following that, the resin was instantly cured for 20 sec to stop the flow. The distance flowed was measured using a digital calliper (Mitutoyo, Japan).

Statistical Analysis

The results of each test were statistically analysed using one-way ANOVA. A post-hoc Tukey test was followed for a multiple comparison. The test of significance was set at 5%.

RESULTS

Characterisation of Filler Particles

Field emission scanning electron microscopy (FESEM) image of nanohybrid silica is shown in Fig. 1. The micrographs confirmed the spherical shape of the particles. The diameter of the particles ranged from 36 nm to 501 nm with the mean diameter being 253±180 nm. These findings were in agreement with previous studies (Noushad *et al.*, 2014; 2016). The nHA were rod shaped with diameter ranging from 56 nm to 174 nm with mean diameter being 110± 37 nm (Fig. 2).

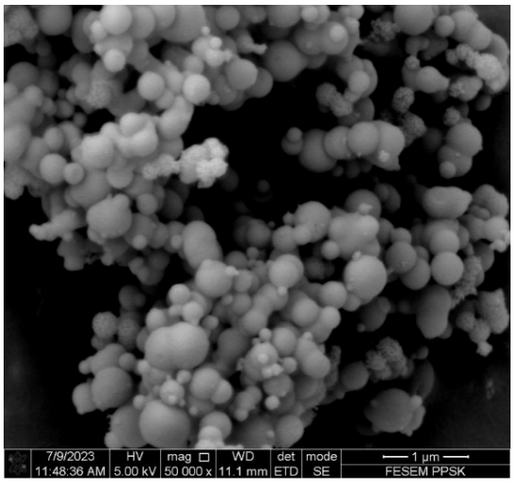

Fig. 1 Scanning electron microscopic image of nanohybrid silica.

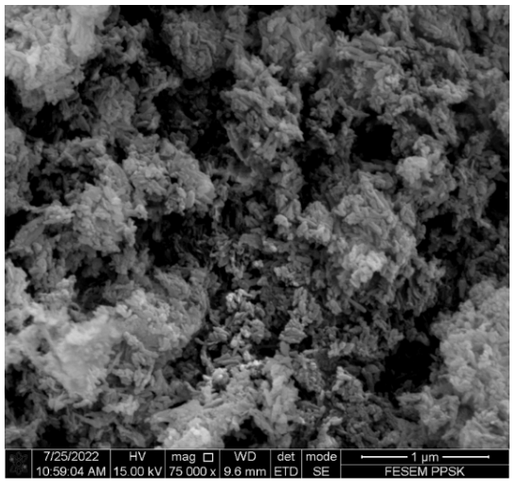

Fig. 2 Scanning electron microscopic image of nanohydroxyapatite.

Flexural Strength and Flexural Modulus

EFS had the highest flexural strength of the sealants evaluated and was similar to the heavily filled commercial sealant, Fissurit FX, with no significant difference (Fig. 3). Clinpro, on the other hand, had considerably lower flexural strength than EFS ($p < 0.05$). The flexural strength of EUS and other tested sealants did not differ significantly. Clinpro and EUS had considerably lower elastic moduli than EFS and Fissurit FX ($p < 0.05$) (Fig. 4).

Surface Roughness

One-way ANOVA analysis revealed significant difference in surface roughness of studied sealants (Fig. 5). Clinpro and EUS had the lowest roughness, with no statistically significant difference between them. However, the roughness of EFS, Clinpro, and Fissurit FX differed significantly ($p < 0.001$). EFS had much less roughness than Fissurit FX ($p < 0.001$).

Vickers Hardness

Fig. 6 shows the hardness of studied RBS. Fissurit FX had a considerably greater hardness value than the other sealants ($p < 0.001$). However, no substantial difference was found between Clinpro and EFS. The hardness value of EUS was significantly lower than that of other sealants ($p < 0.001$).

Flow Distance

EFS had a significantly greater flow distance (mm) than Fissurit FX ($p < 0.001$) (Fig. 7). EFS had a longer flow than Clinpro, but there was no significant difference between them. EUS had the greatest flow distance and was considerably greater than the other sealants tested ($p < 0.001$).

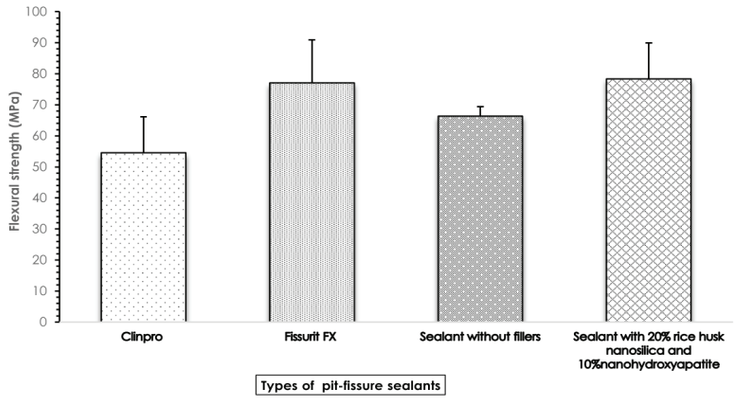

Fig. 3 Flexural strength of studied pit and fissure sealants.

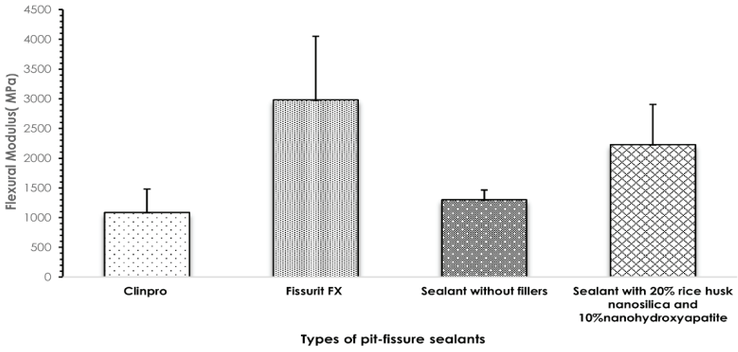

Fig. 4 Flexural modulus of studied pit and fissure sealants.

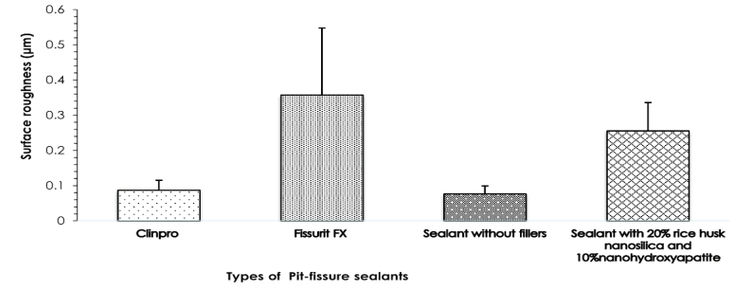

Fig. 5 Surface roughness of studied pit and fissure sealants.

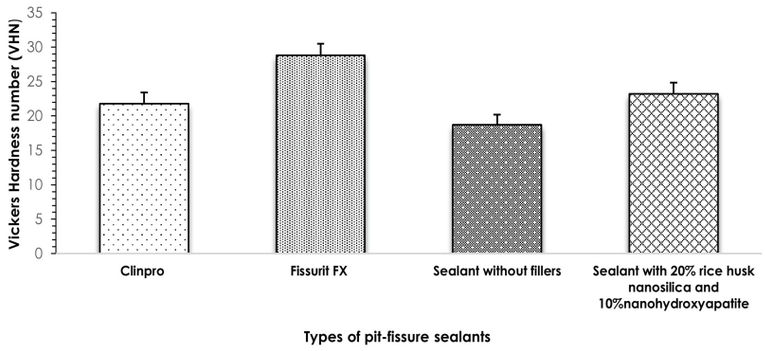

Fig. 6 Vickers hardness of studied pit and fissure sealants.

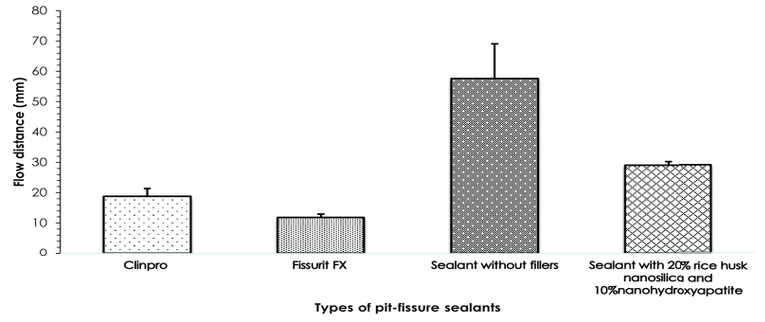

Fig. 7 Flow distance of studied pit and fissure sealants.

DISCUSSION

Resin sealants reinforced with nanofillers have displayed better flexural strength and are comparable to commercial sealants. Ibrahim *et al*. (2018) observed using nano-amorphous calcium phosphate at 20 wt% increased flexural strength up to 79.5±8.4 MPa, which was similar to or higher than commercial sealants Fluoroshield and Virtuoso, a flowable composite. The EFS in the present study displayed the highest flexural strength among the studied sealants and were comparable to commercial controls. Fissurit FX containing 55 wt% of fillers displayed lesser flexural strength than EFS, though the difference was not significant. The exact filler used in Fissurit FX is not very clear; it is reported that inorganic glass ionomers are used as fillers (Yaşa *et al*., 2023). The high flexural strength of EFS could be due to the filler morphology as well as the resin matrix employed. The smaller the filler size, the higher the surface area, thus leading to increased surface energy at the filler matrix contact and enhanced flexural strength of the sealant (Elfakhri *et al*., 2022). Moreover, spherical-shaped nanosilica would have allowed for more dense packing of fillers, increasing the filler volume of the RBS. Furthermore, the difference in resin monomers used in commercial sealants and EFS could also have affected flexural strength. Asmusen and Peutzfeldt observed that replacing Bis-GMA with UDMA improved the flexural strength (Asmussen & Peutzfeldt, 1998). The improved flexural strength resulting from the replacement of Bis-GMA with UDMA was observed in the present study. UDMA, being the base monomer in EFS, possesses strong hydrogen bonding and a better degree of conversion than Bis-GMA, which might have contributed to higher flexural strength (Asmussen & Peutzfeldt, 1998; Barszczewska-Rybarek, 2019).

A high flexural modulus is desirable in load-bearing areas where the resin is subjected to significant masticatory stress. Occlusal surface pits and fissures do not experience high occlusal forces (Dejak *et al.*, 2003). However, the modulus of pit and fissure sealant is unimportant, especially if the pits and fissures are narrow (Beun *et al.*, 2012). In the present study, the flexural modulus of EFS was comparable to that of control sealants, with no significant difference. Fissurit FX sealant, on the other hand, had a much higher modulus than the other two control sealants. Besides filler load, the use of three monomers, Bis-GMA, UDMA, and TEGDMA, may account for Fissurit FX's greater flexural modulus. The combination of three distinct monomers results in strong intermolecular interactions, resulting in a stronger polymer network and a stiffer resin (Szczesio-Wlodarczyk *et al.*, 2021).

The surface roughness of resin-filled material depends on filler structure, percentage of surface area occupied by filler, filler-resin matrix interaction, and coupling agent. Willems *et al.* (1991) concluded that the enamel surface roughness value of 0.64 μm to 0.25 μm at enamel-to-enamel occlusal contact areas is the comparative standard for resin composites. In the present study, the surface roughness of EFS was 0.25 μm, which was well within this range. The surface roughness of EFS was significantly less than that of Fissurit FX but higher than that of Clinpro and EUS. The higher values in the present study could be due to a higher load of nanohydroxyapatite. Klimek *et al.* (2023) reported that the use of hydroxyapatite above 5 wt% makes the surface rougher and more heterogeneous, leading to increased wear of the resin, while Moradian *et al.* (2019) observed increased surface roughness of composite resin even with the use of 2 wt% nanohydroxyapatite. The other reason for increased roughness could be the tendency for agglomeration of hydroxyapatite (HA), along with voids in the aggregates with feeble embedment with the resin, which can increase the surface roughness (Zhang & Darvell, 2012). Increasing HA concentration increases void content due to the increased surface area-to-volume ratio of HA. This increased surface area might result in improper wetting of HA with the resin matrix, leading to clustering and non-uniform dispersion of nanoparticles (Yadav & Gangwar, 2020). Moreover, they also observed that using 3-aminopropyl triethoxysilane (APTES) as a coupling agent reduced the percentage of voids compared to 3-methacry-trimethoxysilane (MPTS), particularly with a higher load of 12% nanohydroxyapatite (Yadav & Gangwar, 2020).

Hardness is a crucial component of a material's ability to withstand indentation or permanent penetration and prevent long-term material breakdown. The Vickers hardness of the EFS increased as the filler content increased, like in earlier studies (Noushad *et al.*, 2016; Yadav & Gangwar, 2020). The EFS had a substantially greater hardness value than the EUS. The higher filler volume fraction causes closer filler contact within the matrix, resulting in stress transfer across the material, mostly through particle-particle interactions (Alshabib *et al.*, 2019). Moreover, a higher filler volume creates an optimal matrix zone that promotes greater attachment of fillers to the resin matrix, thereby enhancing mechanical properties (Jager *et al.*, 2016). The composition and structure of the organic matrix are also important factors in determining hardness (Marghalani, 2016). In the present study, EFS was developed using UDMA as the base monomer. According to a recent study by Levya del Rio and Johnston, using UDMA as a base monomer has a higher microhardness than Bis-GMA (Leyva del Rio & Johnston, 2023). This observation is supported by other published data (Barszczewska-Rybarek, 2009). UDMA's inherent flexibility and strong hydrogen bonding potential may account for its increased microhardness (Lemon *et al.*, 2007).

Effective marginal adaptation is critical for sealant longevity. The rheological behaviour

of a resin heavily impacts its marginal adaptation. According to capillary flow dynamics, the lower the viscosity, the greater the penetration coefficient, allowing RBS to reach deep pits and fissures by penetrating through uneven tooth surfaces (Washburn, 1921). RBS formulations with 20% nano-amorphous calcium phosphate or 20% nano-calcium fluoride as fillers had no effect on sealant flow and were equivalent to commercial controls (Ibrahim *et al.*, 2018; Fei *et al.*, 2020). However, using clay nanotubes or chitosan fluoride with silica nanoparticles in RBS considerably limited their flow (Feitosa *et al.*, 2021; Lai *et al.*, 2022). In the present study, EFS flow distance was comparable to Clinpro sealant and much greater than Fissurit FX. Despite containing 30% filler loads, the EFS flowed similarly to Clinpro, an unfilled sealant. Prior research has found that the rheological behaviour of RBS has little correlation with filler load and is predominantly determined by monomer content (Beun *et al.*, 2012; Ku *et al.*, 2015). According to published data, the viscosity of UDMA is 800,000 centipoise and that of TEGDMA is 5–30 centipoise (Anusavice *et al.*, 2013). UDMA is less viscous than Bis-GMA because the imino groups in UDMA create weaker hydrogen bonds than the hydroxyl groups in Bis-GMA (Sideridou *et al.*, 2002). TEGDMA increased UDMA viscosity more than 2-hydroxyethylmethacrylate (HEMA) or 3-hydroxypropyl methacrylate (HPMA) monomer (Silikas & Watts, 1999). The larger molecule size of TEGDMA compared to HEMA or HPMA was responsible for the observed increase in viscosity (Silikas & Watts, 1999). Thus, the type of monomer utilised, as well as their ratio, has a significant impact on the flow of the RBS.

The present study had certain limitations. This study assessed only hardness, strength, roughness, and flowability. Further studies are needed to assess the degree of conversion, depth of cure, polymerisation shrinkage, colour stability, cytocompatibility, and antibacterial effect of the new sealant.

CONCLUSION

The present study conducted the synthesis of nanohybrid silica derived from rice husk and subsequently formulated a novel pit and fissure sealant including both nanohybrid silica and nanohydroxyapatite. Rice husk continues to be an economically viable source of silica that can be utilised as filler. The incorporation of nanohybrid silica and nanohydroxyapatite resulted in enhanced physicomechanical characteristics of the novel sealant. The experimental RBS demonstrated similar flexural strength, flexural modulus, and hardness as commercially available sealants while maintaining its flow characteristics. The surface roughness of the developed sealant was lower compared to the commercial control, Fissurit FX. The inclusion of a biobased filler and a bioactive filler in this novel sealant makes it a viable substitute for the commercially employed sealants investigated in this research.

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