



Silver nanoparticles incorporated hydrophilic pit and fissure sealant- its preparation, characterization and assessment of shear bond strength

P.Rahmath Meeral¹, Jayashri Prabakar^{2*}, Meignana Arumugham Indiran³, Ganesh J⁴, Rajesh Kumar. S⁵

¹First Year MDS, Department of Public Health Dentistry, Saveetha Dental College & Hospital, Saveetha Institute of Medical and Technical Sciences, Saveetha University, Chennai, Tamil Nadu

²Reader, Department of Public Health Dentistry, Saveetha Dental College & Hospital, Saveetha, Institute of Medical and Technical Sciences, Saveetha University, Chennai, Tamil Nadu

³Professor and Head, Department of Public Health Dentistry, Saveetha Dental College & Hospital, Saveetha Institute of Medical and Technical Sciences, Saveetha University, Chennai, Tamil Nadu

⁴Reader, Department of Pedodontic and Preventive Dentistry, Saveetha Dental College & Hospital, Saveetha Institute of Medical and Technical Sciences, Saveetha University, Chennai, Tamil Nadu

⁵Professor, Department of Pharmacology, Saveetha Dental College & Hospital, Saveetha Institute of Medical and Technical Sciences, Saveetha University, Chennai, Tamil Nadu.

***Corresponding author:** Jayashri Prabakar, Reader, Department of Public Health Dentistry, Saveetha Dental College & Hospital, Saveetha Institute of Medical and Technical Sciences, Saveetha University, Chennai, Tamil Nadu, No.162, Poonamallee High Road, Chennai-600077, India, Email: jayashri.sdc@saveetha.com

Submitted: 17 February 2023; Accepted: 28 March 2023; Published: 02 April 2023

ABSTRACT

Background: Occlusal fissure dental caries are more common in children due its complex morphology resisting the biofilm clearance by routine tooth brushing. Pit and fissure sealants are utilized for the sealing function of deep fissures making its morphology shallow that can reduce the risk of caries development. Ultraseal is the hydrophilic pit and fissure sealant that was developed to overcome the technical difficulties of salivary moisture contamination during sealant application procedure.

Aim and Objectives: The present study was done to evaluate the newly synthesized, silver nanoparticles incorporated hydrophilic pit and fissure sealant in terms of its shear bond strength when compared to the conventional hydrophilic Ultraseal XT Hydro sealant.

Materials and Methods: Green synthesis of Ag nano particles were prepared from the beetroot followed by the Scanned electron microscope analysis, EDAX analysis and FTIR analysis for the characterization of extracted silver nanoparticles. Twenty premolars extracted for the orthodontic purpose were randomly allocated equally (n = 10) into two groups with two different sealants to evaluate shear bond strength at sealant space. Group 1 (n=10) had ultraseal XT hydro and Group 2 with Ag nanoparticles incorporated ultraseal XT hydro. The shear bond strength was evaluated with the INSTRON Universal testing machine. The values obtained were comparatively analysed with independent t test.

Results: Ag nanoparticles incorporated hydrophilic sealant recorded the highest shear bond strength with mean value 175 MPa and the difference was statistically significant (p value = 0.000) with Ultraseal sealant which has mean value of 165 MPa.

Conclusions: Ag nanoparticles incorporated hydrophilic sealant recorded the highest shear bond strength and the difference was statistically significant with Ultraseal sealant.

Keywords: *Hydrophilic sealant, pit and fissure sealant, Silver nanoparticles fissure sealant, Silver nanoparticles hydrophilic sealant*

INTRODUCTION

Approximately 90 % of Dental carious lesions and cavities commonly occur in pits and fissures of the occlusal surfaces in primary and permanent posterior teeth¹. This is attributed to the fact that, the complex morphology of this surfaces defended against the remineralizing flow of saliva and routine brushing². It is evident from the results of various previous study that 56–70% of the carries in children of 5–17 years of age occurs in pit and fissures^{3,4}. Research in the field of dentistry had developed a preventive agent, pit and fissure sealant, which has shifted the treatment philosophy from “Drill and Fill” to that of “Seal and Heal”⁵.

The World Health Organization considers the pit and fissure sealants as the primary preventive measure, one of the most effective and least invasive means available to ensure the complete protection and the preservation of the total occlusal from the carious phenomenon^{6,7}. Apart from the functioning of prevention from the initiation of dental caries, there is evidence that sealants also can inhibit the progression of non cavitated carious lesions⁸.

Buonocore in 1955 described acid etch bonding to enamel as new technology and it was employed in the form of resin sealants for the first time in the prevention of pit and fissure caries⁹. Simonsen illustrated pit and fissure sealant as a material that is introduced into the occlusal pits and fissures of caries-prone teeth, thus forming a micromechanically-bonded, protective layer cutting access of caries-producing microorganisms from their source of nutrients¹⁰.

Based on a systematic review, a 2016 guideline panel convened by the American Dental Association Council on Scientific Affairs and the

American Academy of Pediatric Dentistry recommended the following evidence-based clinical proposal for the use of pit and fissure sealants on the occlusal surfaces of primary and permanent molars in children and adolescents¹¹.

On this basis, over the past few years, a wide spectrum of resin-based and glass ionomer sealants has been introduced in the dental merchandise. These materials are mostly hydrophobic that bond to the enamel surface via micromechanically interlocking tags¹². The presence of moisture and saliva-contamination during the placement of the sealant compromise the quality of adhesion at the sealant-enamel interface, which impacts the ongoing resistance to microleakage of microorganisms.

Recently introduced hydrophilic sealant, UltraSeal XT® hydro™ is a new moisture tolerant, self-adhesive, light-cured, acrylate-based, hydrophilic pit, and fissure sealant which has which bond effectively to moist enamel surfaces, present a distinct advantage in paediatric dentistry where patient-compliance, isolation, and moisture-control can be particularly challenging. It has been reported that this material “chase” moisture and works efficiently¹³. Also it has 53% highly filled resin with thixotropic (ideal viscosity), and advanced adhesive technology which allows it to flow into pit and fissures and bond effectively without a drying agent to the tooth¹⁴.

To improve the property of resin-based sealants, previously manufacturers have added filler particles, fluoride, and fluorescence to the material. Recently, Nanotechnology has been applied to dental materials as an innovative concept for the development of materials with better properties and anticaries

potential. Functional materials or structures at the nanometer scale (0.1–100.0 nm) can be used to control the formation of cariogenic oral biofilms: nanoparticles can deliver antibiotics and bioactive compounds. Dental plaque contains a vacuum and channels that occasionally extend completely through the biofilm biomass to the underlying dental surface, influencing the transfer of particles through biofilms. Within this context, the nanoparticles are potentially useful because their surface charge, degree of hydrophobicity, the ratio of surface area to biofilm mass, and the ability of the particles to adsorb or be collected on the surface of the biofilm can be changed¹⁵.

Nanomaterials provide superior antimicrobial activity and display comparable physical properties when compared with conventional materials, this is probably because of the small size and high surface area of the nanoparticles, capable of releasing high levels of ions at a low filler level, thereby enabling the incorporation of reinforcing (but nonreleasing) fillers in the same material. nanoparticles and nanoclusters with a broad particle distribution provide a higher filler load, desirable handling characteristics, and better optical and/or physical properties in the restoration material¹⁶. From this aspect, nanoparticles can be utilized for enhancing the effectiveness of the current sealant.

One of the prerequisites for a sealant to be effective as suggested by Brauer is that it should have good and prolonged adhesion to enamel¹⁷. The retention rate of a sealant is directly related to the micromechanical bonding between the sealant and tooth enamel. Shear bond strength measures the ability of sealant to bond to tooth structure. Higher shear bond strength is equated with enhanced performance¹⁸. This necessitates

the rationale for the study. Therefore, the effectiveness of resin-based sealants depends on sealant retention which could be directly attributed to the shear bond strength of the material.

Laboratory in vitro tests play a key role in providing the necessary information regarding the efficacy of new products in a short period of time. Several in vitro studies have been conducted to evaluate the shear bond strength different sealant material, but no studies have compared the effect of this two pit and fissure sealant.

Since there exist no nanoparticles incorporated fissure sealant that are hydrophilic, the objectives of this study is to prepare silver nano particles reinforced pit and fissure sealant followed by its characterization and in vitro comparison and evaluation of the shear bond strength of conventional and silver nano particles reinforced hydrophilic fissure sealants applied to the enamel on the buccal surface of permanent molars.

METHODOLOGY

Green synthesis of silver nanoparticles

Beetroot extract was obtained by boiling 20.3 grams of beetroot in 100ml of distilled water for 4 hours. This was followed by further adding of 50 ml of distilled water and 0.016 gram of silver nitrate powder. This final mixture was kept in orbital shaker for 32 hours followed by centrifugation at 10000 rpm for 20 minutes. The supernatant solution was separated and the concentrated solution was transferred to petri dish. The Solution in petri dish was kept at hot air oven for 24 hours which yield Ag nano particles in powder form. This Ag nanoparticles powder are mixed with the hydrophilic pit and fissure sealant in the ratio of 1:4. (Fig 1a – 1f)

Silver nanoparticles incorporated hydrophilic pit and fissure sealant- its preparation, characterization and assessment of shear bond strength

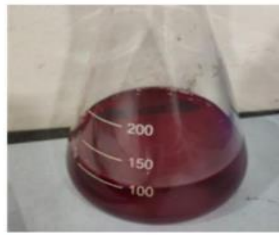


Fig 1a. Beetroot extract with Silver nitrate



Fig 1b. Orbital Shaker



Fig 1c. Centrifugation

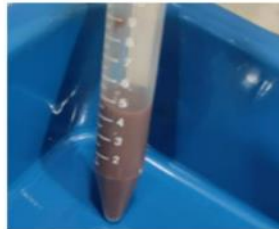


Fig 1d. Precipitated Ag nanoparticles

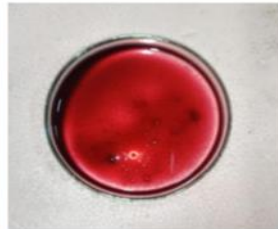


Fig 1e. Dried Ag nanoparticles to be scrapped

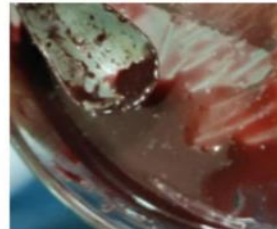


Fig 1d. Final Ag nanoparticles to be incorporated into Ultraseal XT Hydro Sealant

Characterization of silver nano particles

Silver nanoparticles presence was confirmed by Scanned electron microscopy (SEM) Fig 2, which shows Spherical and rectangular nanoparticles of size less than 100nm in the background of sugars

of green origin. Phytochromes responsible for nanoparticles synthesis are shown in Fourier-transform infrared spectroscopy Fig 3. Further elemental analysis by EDAX shows the presence of AG nanoparticles Fig4.

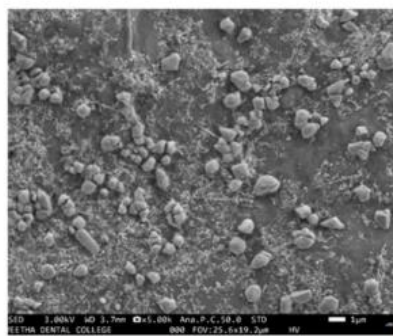


Figure 2: SEM analysis

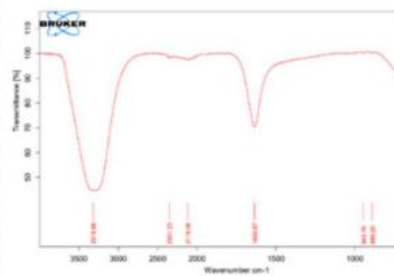


Figure 3: FTIR image



Figure 4: EDAX image

Shear bond strength tests

Twenty sound premolar teeth extracted for orthodontic purpose or extracted due to periodontitis were selected for the study. Sample size was calculated with ± 0.5 of standard deviation with minimum expected difference of 0.74 and 0.05 of significance criterion at 90% statistical power. Roots were cut off 2 mm below the cemento-enamel junction with diamond saw at low speed under water-coolant. The crowns were embedded in acrylic resin blocks with exposed sound buccal or lingual surfaces. Enamel surface was cleaned on each tooth with a using a low-speed hand piece with brush under water coolant. Twenty teeth were randomly categorized in to two equal groups with 10 samples per group based on the sealant type used for shear bond strength testing. Group 1 – Etchant Gel + Ultraseal. Group 2 – Etchant Gel + Ag nano

particles incorporated ultraseal hydrophilic sealant. In both the group specimens, enamel surface was etched with 37.5% phosphoric acid for 60 s followed by washing and drying. Ultraseal and Ag nanoparticles incorporated hydrophilic sealant were applied in respective groups in a 2-mm thick layer over the conditioned enamel substrate and cured using ultraviolet light. The bonded specimens were then left undisturbed at 37°C for 24 h in 100% humidity before shear bond strength testing. Shear load was applied using a universal testing machine (Instron) in a direction parallel to the bonded interface Fig 5. The load at failure was recorded in Newtons (N). The diameter of the debonded composite cylinder was measured with a digital caliper. Bond strength was then calculated in Mega Pascals (MPa).

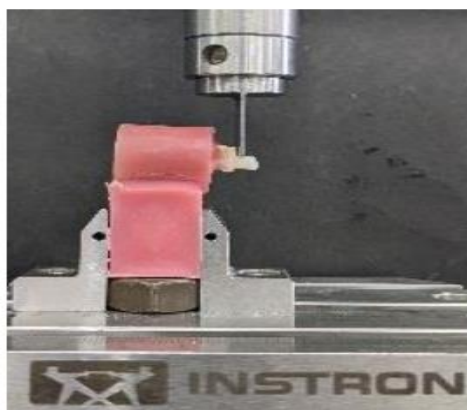


Fig 5. Shear bond strength measurement

RESULTS

Test sealant. (n=10)	Mean shear bond strength.	P Value
Group 1	157 \pm 3.55 MPa	<0.05
Group 2	175 \pm 3.13MPa	

The Independent t test revealed that the shear bond strengths of the tested materials differed significantly with p value 0.000 and Ag nano particles reinforced hydrophilic sealant recorded the highest mean shear bond strength of 157 \pm 3.13. (p value = <0.05)

DISCUSSION

The complex morphology of the occlusal surface of the molars makes a more susceptible area for dental caries initiation and development as it is difficult to perform a proper oral hygiene practice there. The complex types of fissures such as K type, Y type makes it a caries prone area by cariogenic biofilm build up. Also, since

permanent molars are having long eruption phase and were not in occlusion for longer time, they are in risk of developing caries by colonization of cariogenic, acidogenic and aciduric bacteria.

From the previous literature and various number of research studies we can find a wealth of evidences that pit and fissure sealants are an excellent material that can be place in the deep pits and fissures which were at the risk of developing caries. Most of the pit and fissure sealant materials used by the dental practitioners are resin based which were hydrophobic in nature, that requires strict isolation from salivary moisture contamination. To overcome this issue hydrophilic fissure sealant Ultraseal XT hydro were introduced into the market. In our study we aimed to introduce the various benefits of nanotechnology into this recently introduced hydrophobic Ultraseal XT hydro sealant. Although there are many methods practiced for the synthesis of nanoparticles in nanotechnology, biogenic reduction of metal precursors is less expensive, eco-friendly and free of chemical contaminants for medical and biological applications¹⁹. Hence our study used the green synthesis of silver nanoparticles from beetroot pulp extract.

According to B Nevue et al, on the basis of salivary contamination, newer resin-based hydrophilic sealant such as Ultraseal XT hydro with its characteristic thixotropic property, chases moisture deep into pit and fissures on a microscopic level and its advanced adhesive technology allows it to flow into pit and fissures, and bond effectively without a drying agent to the tooth⁷.

From the previous literature evidence, invitro investigation of shear bond strength is very important tool for determining the retention ability on enamel to predict its clinical effectiveness for the dental restorative material. According to Buonocore MG et al, the retention rate of a pit and fissure sealant is directly related to the micromechanical bond between the sealant and enamel which can be evaluated by measuring the shear bond strength of sealants which provide a guide for clinical use of these materials⁹. Hence the objective of the current study focuses on the evaluation of shear bond strength of the test

material which can reflects its clinical efficacy. As we are synthesising the novel material with modification by adding silver nano particles in existing Ultraseal XT hydro sealant, the primary objective of our study is to determine the shear bond strength of synthesized Ag nanoparticles incorporated hydrophilic fissure sealant and the secondary objective is to comparatively evaluate it with the conventional hydrophilic sealant.

Previous study by Prashant Babaji et al, 2016 evaluated the shear bond strength of different pit and fissure sealants in vitro and found that the hydrophilic resin sealant, Tetric flow recorded the highest shear bond strength among them and the difference was statistically significant with Enamel loc20. This result is partly in accordance with the result of our current study which produces the mean shear bond strength of 157 ± 3.55 MPa for the Ultraseal Hydro XT, but it is statistically lesser than the Ag nanoparticles incorporated Ultraseal XT hydro which has shear bond strength of 175 ± 3.13 MPa. The main reason for the higher shear bond strength of Ag nanoparticles incorporated Ultraseal XT Hydro sealant is mainly due to its nano filler particles of Silver. This reason is strengthened by the results of the previous study by Azam A et al in 2013, which shows that incorporation of silver nanoparticles containing 5% and 1% silver maintains and increases the shear bond strength of orthodontic adhesives²¹.

According to the results of HM Pushpalatha et al in 2014 on comparison of shear bond strength of different pit and fissure sealant, we can find that unfilled sealant possess higher shear bond strength than filled sealant. This result is not in accordance with the results obtained from the current study¹⁷.

The limitation of this current study is comparing the shear bond strength of the test material only with one group i.e, Ultraseal XT hydro. There might be a difference in the obtained result of higher shear bond strength by Ag nanoparticles incorporated sealant when comparing to other commercially available sealant. So the future study should focus on comparative evaluation with other different types of sealants and also the assessment of shear bond strength should be done

after certain time period which could have some difference.

CONCLUSION

Within the limits of this in vitro study and according to the methodology and the results drawn, the following statements were concluded that Ag nanoparticles incorporated sealant has maximum shear bond strength.

Financial Support and sponsorship

Nil.

CONFLICTS OF INTEREST

There are no conflicts of interest.

REFERENCE

1. Splieth CH, Ekstrand KR, Alkilzy M, Clarkson J, Meyer-Lueckel H, Martignon S, et al. Sealants in dentistry: Outcomes of the ORCA Saturday afternoon symposium 2007. *Caries Res* 2010;44:3-13.
2. Gooch BF, Griffin SO, Gray SK, Kohn WG, Rozier RG, Siegal M, Fontana M, Brunson D, Carter N, Curtis DK, Donly KJ. Preventing dental caries through school-based sealant programs: updated recommendations and reviews of evidence. *The Journal of the American Dental Association*. 2009 Nov 1;140(11):1356-65.
3. Kaste LM, Selwitz RH, Oldakowski RJ, Brunelle JA, Winn DM, Brown LJ, et al. Coronal caries in the primary and permanent dentition of children and adolescents 1-17 years of age: United states, 1988-1991. *J Dent Res* 1996;75 Spec No:631-41.
4. Meneghim MC, Saliba NA, Pereira AC. Importance of the first permanent molars in the determination of DMFT Index. *J Brasi Odontop Odontol Bebê* 1999;2:37-41.
5. Singh S, Adlakha V, Babaji P, Chandna P, Thomas AM, Chopra S. A comparative evaluation of the effect of bonding agent on the tensile bond strength of two pit and fissure sealants using invasive and non-invasive techniques: An in-vitro study. *J Clin Diagn Res* 2013;7:2343-7.
6. Hiiri A, Ahovu-Saloranta A, Nordblad A, Mäkelä M. Pit and fissure sealants versus fluoride varnishes for preventing dental decay in children and adolescents. *Cochrane Database Syst Rev* 2010;17:CD003067.
7. Beslot-Neveu A, Courson F, Ruse ND. Physico-chemical approach to pit and fissure sealant infiltration and spreading mechanisms. *Pediatr Dent* 2012;34:57-61.
8. Splieth C, Förster M, Meyer G. Additional caries protection by sealing permanent first molars compared to fluoride varnish applications in children with low caries prevalence: A 2-year results. *Eur J Paediatr Dent* 2001;2:133-7.
9. Buonocore MG. A simple method of increasing the adhesion of acrylic filling materials to enamel surfaces. *J Dent Res* 1955;34:849-53.
10. Simonsen RJ. Pit and Fissure Sealants. In: *Clinical Applications of the Acid Etch Technique*. 1st ed. Chicago, IL: Quintessence Publishing Co, Inc.; 1978. p. 19-42.
11. Wright JT, Crall JJ, Fontana M, Gillette EJ, Nový BB, Dhar V, et al. Evidence-based clinical practice guideline for the use of pit-and-fissure sealants: A report of the american dental association and the american academy of pediatric dentistry. *J Am Dent Assoc* 2016;147:672-8200000000000.
12. Sezinando A. Looking for the ideal adhesive – a review. *Rev Port Estomatol Med Med Dent Cir Maxilofac*. 2014;55:194-206.
13. Güçlü ZA, Dönmez N, Hurt AP, Coleman NJ. Characterisation and microleakage of a new hydrophilic fissure sealant-UltraSeal XT® hydro™. *Journal of Applied Oral Science*. 2016 Jul;24:344-51.
14. Ultradent Products, Inc. *Product Guide for Ultraseal XT Hydro*; 2013.
15. Allaker RP. The use of nanoparticles to control oral biofilm formation. *Journal of dental research*. 2010 Nov;89(11):1175-86.
16. Melo MA, Guedes SF, Xu HH, Rodrigues LK. Nanotechnology-based restorative materials for dental caries management. *Trends in biotechnology*. 2013 Aug 1;31(8):459-67.
17. Pushpalatha HM, Ravichandra KS, Srikanth K, Divya G, Done V, Krishna KB, Patil V. Comparative evaluation of Shear bond strength of different Pit and fissure Sealants in Primary and Permanent teeth-An In-Vitro Study. *Journal of international oral health: JIOH*. 2014 Apr;6(2):84.
18. Dhillon JK, Pathak A. Comparative evaluation of shear bond strength of three pit and fissure sealants using conventional etch or self-etching primer. *Journal of Indian Society of Pedodontics and Preventive Dentistry*. 2012 Oct 1;30(4):288.
19. Hussain I, Singh NB, Singh A, Singh H, Singh SC. Green synthesis of nanoparticles and its

Silver nanoparticles incorporated hydrophilic pit and fissure sealant- its preparation, characterization and assessment of shear bond strength

- potential application. *Biotechnology letters*. 2016 Apr;38(4):545-60.
20. Babaji P, Vaid S, Deep S, Mishra S, Srivastava M, Manjooran T. In vitro evaluation of shear bond strength and microleakage of different pit and fissure sealants. *Journal of International Society of Preventive & Community Dentistry*. 2016 Aug;6(Suppl 2):S111.
 21. Akhavan A, Sodagar A, Mojtahedzadeh F, Sodagar K. Investigating the effect of incorporating nanosilver/nanohydroxyapatite particles on the shear bond strength of orthodontic adhesives. *Acta Odontologica Scandinavica*. 2013 Sep 1;71(5):1038-42.