

Scanning Microradiography: Measurement of X-ray Attenuation Coefficient of Fissure Sealants Containing Tin Methacryloxytri-n-butyltin (SnM) as Radio-opaquer

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Received November 08, 2013; Revised November 27, 2013; Accepted December 10, 2013

Abstract Objective: Radio-opacity is a fundamental requirement for fissure sealants in order to be clearly visible radiographically and may be well differentiated from caries. Many attempts have been made to make resin based fissure sealants radio-opaque. Researchers achieved radio-opacity of the sealants by blending heavy metals as fillers into the polymeric matrix. Keeping the demerits of using heavy metals in view, a novel fissure sealant was developed by chemically incorporating Tin (Sn) in monomer of methacrylate. This study has been done to assess under scanning micro-radiography (SMR), the radiopacity of the indigenously prepared fissure sealants containing increasing amount of SnM until the consistency of the mix remains workable and clinically useful. **Design:** Experimental study. **Place of study:** Biophysical Lab Queen Mary University of London, United Kingdom. **Methodology:** An organo-tin compound – Methacryloxytri-n-butyltin (SnM) and Ethylene Glycol Dimethacrylate (EGDMA) in varying quantities were mixed to prepare fissure sealant indigenously. Camphorquinone (CQ) and N, N-Dimethyl-P-Toluidine (DMPT) were added to above mixture. The prepared sealants were polymerized in cuvettes and mounted on Scanning microradiography machine. The machine was run for 100 seconds repeatedly for 60 runs. Obtained results were processed and calculated in Microsoft Excel software. **Results:** It was observed that increasing the weight of Tin (Sn) content in a sealant increases the radio-opacity of the sealant but SnM more than 9.5 gm renders the material unworkable. **Conclusion:** This study will not only help in enhancing the radio-opacity of fissure sealants but also the radio-opacity of other clinical composite materials maybe enhanced by this method.

Keywords: linear attenuation coefficient, tin methacrylate, radio-opacity of fissure sealant

Cite This Article: Aftab Ahmed Khan, Asaad Javaid Mirza, Adel Zia Siddiqui, and Maaz Asaad, "Scanning Microradiography: Measurement of X-ray Attenuation Coefficient of Fissure Sealants Containing Tin Methacryloxytri-n-butyltin (SnM) as Radio-opaquer." *International Journal of Dental Sciences and Research* 1, no. 3 (2013): 56-59. doi: 10.12691/ijdsr-1-3-4.

1. Introduction

The **attenuation coefficient of X-rays**, also called linear attenuation coefficient (LAC) is defined as a quantity of absorbed X-rays that shows how easily a medium can be penetrated by a beam of X-rays. It measures the fraction of beam energy absorbed or scattered per unit thickness of an absorbing medium [1]. A large attenuation coefficient means that the beam is quickly "attenuated" (weakened) as it passes through the medium, and a small attenuation coefficient means that the medium is relatively easier to pass through to the beam. It is represented using the symbol μ , and measured in cm^{-1} . The linear attenuation coefficient of a substance varies with density and X-ray energy, depending on size of its atoms and spacing between the atoms [2].

Like other dental restorative materials, polymeric based dental composite material should have high value of radiopacity so that the clinician may identify the filling in contrast to the tooth and carious lesions radiographically. The attenuated beam will be unable to pass through fillers of the fissure sealant and thus the sealant appears radio-opaque [3].

The fissure sealant is a dental resin applied to deep pits and fissures of posterior teeth that may be susceptible to caries [4].

Though all the short and long term studies have proved the significance of the use of sealants on pits and fissures of teeth [5,6,7], fissure sealants are not applied in dental practices as readily as they should have been [8]. The logical reason behind their underuse is that almost all of the commercially available sealants are made up of Bisphenol A and Glycidyl Methacrylate which is radiolucent [9] It becomes very difficult for a dental clinician to distinguish radiologically between an applied

sealant and caries. One study has shown that an error rate among dentists to identify the presence or absence of dental sealants is 1.4% for opaque sealants and 22.8% for clear sealants [10].

Radio-opacity of fissure sealants is dependent on percentage and type of their fillers [11]. The researchers achieved radio-opacity of the sealants by blending heavy metals as fillers into the polymeric matrix. Although these materials provide radio-opacity to some extent but use of the heavy metals have many disadvantages; they are toxic and sometimes radio-active, oxides of the rare earths like ytterbium fluoride can lead to undesired discoloration. Heavy metal organic compounds such as Triphenyl bismuth easily wash out of the matrix [12]. Commercially available fissure sealants have inclusion of heavy metals so that the sealants may appear radiopaque. Although inclusion of heavy metals makes the sealants radiopaque but their presence in fissure sealants have deleterious effect if the weight percent of these heavy metals increases in polymeric fissure sealants.

A novel fissure sealant has been developed by chemically incorporating Tin (Sn) in monomer of methacrylate [3]. This monomer mixture (SnM) has many merits. It is liquid at room temperature and prevents increase in viscosity of the sealant. It is homogeneously mixable with matrix and has contrasting radio-opacity. Moreover, it does not hinder polymerization and acts as filler to improve wear resistance and decreases microleakage [13].

Ahmed *et al* [3], 2005 measured LAC of fissure sealants containing maximum 50% SnM to assess their radio-opacity. They observed that increasing the content of SnM increases the radiopacity of the resultant sealant due to the presence of Tin in methacrylate formulation for being heavy element having a molecular weight of about 118 u. Moreover Tin being chemically incorporated with the chain of Methacrylate, has an advantage of not being washed out or leached out of the polymeric chain.

It is hypothesized that incorporation of more than 50% SnM may further enhance the radio-opacity which will facilitate the dental clinicians in accurate diagnosis. This study has been done to assess, under scanning microradiography (SMR), the radio-opacity of the indigenously prepared fissure sealants containing increasing amount of SnM until the consistency of the mix remains workable and clinically useful.

2. Objectives

To assess the radio-opacity of experimental fissure sealants containing 7.5, 8.5 and 9.5gm SnM using scanning microradiography.

3. Methods

An organo-tin compound – Methacryloxytri-n-butyltin (SnM 6488, Lot# 2155-706, ABCR GmbH & Co. KG, ImSchleher 10 D-76187 Karlsruhe) and Ethylene Glycol Di-methacrylate (EGDMA), 90% inhibited with 40-150 ppm HQ (Aldrich chemical Co. Ltd, Gillingham Dorset, England, Lot no. 97-90-5) in varying quantities were mixed to prepare fissure sealant indigenously.

Camphorquinone (CQ)97%(Aldrich Chemical Company Inc, Riedstr 2,Steinberg, Germany, Lot no.S12442-513)in a constant quantity of 0.05gm and N, N-Dimethyl-P-Toluidine (DMPT),99% (Aldrich Chemical Company Inc, Riedstr 2, Steinberg, Germany, Lot no. AQ05027HN)in a constant quantity of 0.05ml were added to above mixture.

3.1. Preparation of Experimental Sealant Containing 7.50 gmSnM

2.50 gm Ethylene Glycol Dimethacrylate (EGDMA) was poured on a weighing boat with the help of a pipette and the precise weight was measured on Explorer® (OHAUS Europe GmbH Heuwinkelstrasse 3 CH-8606 Nanikon Switzerland).The measured weight was poured into amber-colored bottle (amber-colored bottles were used to stop accidental polymerization) with cap on it. 7.5 gmSnM, 0.05 gmCamphorquinone and 0.05 ml N, N-Dimethyl-P-Toluidine were added to the EGDMA in the bottle.

To mix all the constituents homogeneously, a magnetic stirrer (IKA LABORTECKNIC® Janke & Kunkel-Str.10, 79219 Staufen, Germany) set at 500 rpm was used. The material was mixed for 10 minutes in a dark room having a room temperature set at 25 °C.

3.2. Preparation of Experimental Sealant Containing 8.5gm & 9.5 gmSnM

Mixtures of the same ingredients were prepared in the same manner with increasing weight of SnM as 8.5gm and 9.5gm and decreasing quantity of EGDMA as 1.5gm and 0.5 gm respectively.

Following three compositions were prepared:

7.5 gmSnM + 2.5 gm EGDMA + 0.05 gm CQ + 0.05 ml DMPT (75% SnM)

8.5 gmSnM + 1.5 gm EGDMA + 0.05 gm CQ + 0.05 ml DMPT (85% SnM)

9.5 gmSnM + 0.5 gm EGDMA + 0.05 gm CQ + 0.05 ml DMPT (95% SnM)

With the help of a disposable pipette, 01 ml of each experimental sealant from amber colored bottle was placed in a cuvette measuring 12.5x12.5x45 mm Figure 1 and light-cured for 20 seconds on each side using a curing light (Heliolux DLX by VIVADENT).



Figure 1. Experimental Sealant in a Cuvette

After polymerization, the cuvettes (filled with experimental dental sealants) were mounted on the SMR machine and XY step-scanned past a $15\ \mu\text{m}$ X-ray beam using a precision computer-controlled micro-positioning system [Figure 2](#). The SMR machine used had a high power supply generator (ENRAF NONIUS FR590) run at 5.0mA and 45KeV. The machine was run for 100 seconds repeatedly for 60 runs. Obtained results were processed and calculated in Microsoft Excel software.



Figure 2. Scanning Microradiography machine

For visual analysis the samples were analyzed using Amira (TGS Template Graphics Software Inc., USA). It is a computer program which gives 3-D imaging. It allowed visualization of every single slice as well as viewing of the samples from each angle as shown in [Figure 3](#).

4. Results

The result shows linear absorption coefficient for the varying amount of SnM in the experimental sealants. It was observed that increasing the weight of Tin (Sn) content in a sealant increases the radiopacity of the sealant. This is due to the fact that Sn is a heavy metal atom with a high X-ray LAC. Increasing the SnM content in sealant increases the fraction of X-rays that are absorbed and cause "attenuation" of the X-Ray beam.

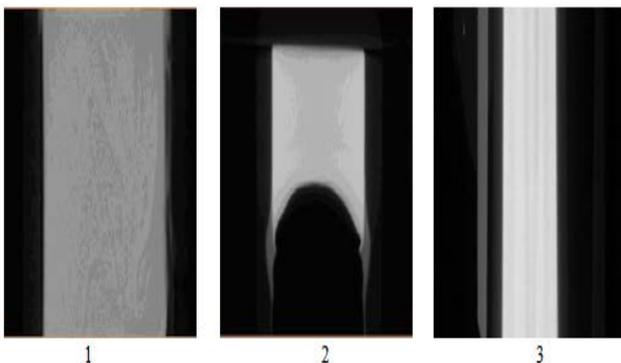
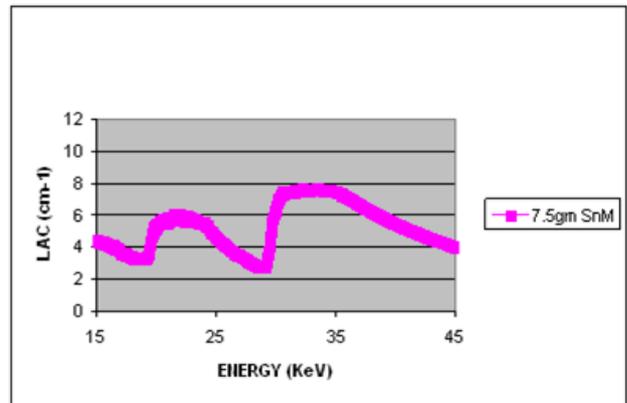
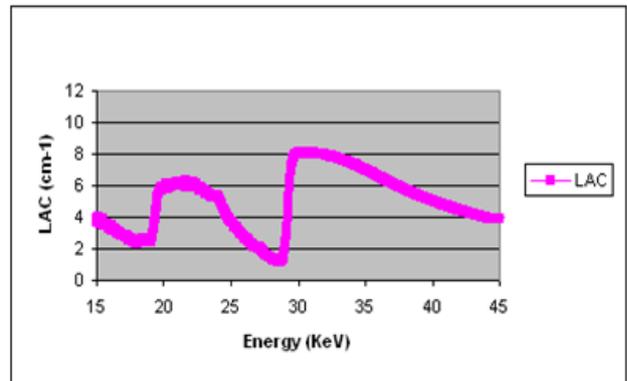


Figure 3. 1: sealant with 7.5 gmSnM. 2: sealant with 8.5 gmSnM. 3: sealant with 9.5 gmSnM

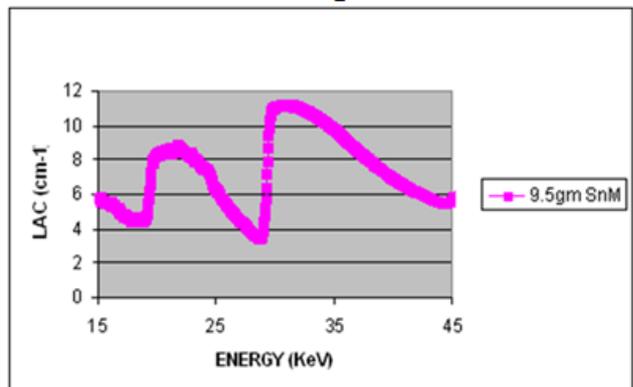
The graphs shown in [Figure 4](#) reveal that at around 30 KeV, Linear attenuation coefficient of the sealant containing 7.5, 8.5 and 9.5 gm is $7.617\ \text{cm}^{-1}$, $8.07\ \text{cm}^{-1}$ and $11.18\ \text{cm}^{-1}$ respectively. It is also observed that beyond 30 KeV the LAC decreases.



1



2



3

Figure 4. 1: Graph showing LAC of 7.5gm SnM 2: Graph showing LAC of 8.5gm SnM 3: Graph showing LAC of 9.5gm SnM

5. Discussion

Scanning Micro radiography (SMR) is a digital form of microradiography in which the photographic emulsion is replaced by a solid-state X-ray detector [\[14\]](#). It was developed to overcome problems associated with contact microradiography [\[15\]](#).

It is an accurate method of measuring x-ray attenuation coefficient because transmitted intensity can be measured at each scan point by an electronic x-ray detection system. The X-ray detection system includes a multi-channel analyzer to obtain X-ray spectra [\[14\]](#).

Radio-opacity is a fundamental requirement for restorative dental materials [16]. ISO standard 4049 stipulates that the minimum radiopacity for a restorative material should be equal or higher than its equivalent thickness in aluminum. Amalgam, Gold, Cobalt Chromium, Stainless Steel are the metallic dental materials that have good radio-opacity [17]. The high radiopacity of a fissure sealant gives clear differentiation between restoration and tooth structure.

We used Methacryloytri-n-butyltin (SnM) as a radiopaque material to make the sealants radio-opaque. The rationale behind choosing this material was SnM's physical and chemical properties. SnM is a liquid at room temperature and its melting point is 17-20°C. When this liquid radio-opaque material was mixed with diluent EGDMA, it was completely soluble in EGDMA. As both SnM and EGDMA were in liquid form, so a homogenized mixture was formed. Although a magnetic stirrer was used to mix these materials but there was no need to give temperature to solubilize SnM sealant with EDGMA.

In addition to Methacryloytri-n-butyl tin use as dental sealant, the copolymer mentioned in this experimental work may also be used in other medical and dental applications. This SnM compound maybe copolymerized with other acrylic resin to form artificial radio-opaque teeth or maybe used for making other radio-opaque restorative materials as well as medical cements.

The general toxicity of organo tin compounds have been discussed by Blunden *et al* [3]. Minor skin lesions have also been observed by the application of butyle tin compounds on the back of the hand by Lyle [13]. Further work is required to establish the proportion of residual monomer in the polymerized sealant in order to carry out initial risk assessment [3].

Viscosity is a very important property of the fissure sealants. Sealants with a high penetration coefficient and low viscosity achieve optimum penetration [18]. It is therefore recommended that low viscosity sealants should be used [19]. Increasing % of Sn in copolymer increased the radio-opacity but its effect on the viscosity of the resultant sealants has not been assessed in this study. Although the viscosity observation when done through naked eyes, it was found that the sealants were easily flowing but interpretation done without any scientific calculation cannot be relied.

6. Conclusion

It was observed that in all the experimental sealants, the absorption edge was around 30 KeV but with the increasing energy level, linear attenuation coefficient decreased.

This Study has proven the radio-opacity of SnM based sealants that can be comparable to pure metallic materials. This study will not only help in enhancing the radio-opacity of fissure sealants but also the radio-opacity of

other clinical composite materials maybe enhanced by this method.

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