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Influence of $\text{Al}_2\text{O}_3/\text{Y-TSZ}$ mixture as filler loading on the radiopacity of PMMA denture base composites

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Abstract

The radiopacity of alumina/yttrium stabilizer zirconia ($\text{Al}_2\text{O}_3/\text{Y-TSZ}$) particles with nitrile butadiene rubber (NBR) dispersed in PMMA denture base material has been investigated. PMMA matrix without filler was prepared from PMMA powder with 0.5% benzoyl peroxide (PBO) as the control material. The similar PMMA matrix was mixed with $\text{Al}_2\text{O}_3/\text{Y-TSZ}$ (1:1) together with NBR particles as the reinforcement. The amount of NBR was fixed at 7.5 wt %, however, $\text{Al}_2\text{O}_3/\text{Y-TSZ}$ varied from 1 to 10 wt %, respectively. Samples with 4 mm thickness for each composition were irradiated using 60 KV, 10 mA, 0.4 s to examine their radiopacity. This radiopacity was compared to radiopacity of aluminum plate which having the same thickness. The result shows that the radiopacity (i.e. the lower optical density the higher radiopacity) of reinforced PMMA matrix slightly increased from 1.40 to 1.05, respectively, with the increased of filler loading compared to unreinforced PMMA matrix.

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1. Introduction

The denture base should be ideally radiopaque and capable to be detected using normal diagnostic radiographic techniques and measurement of the ability of electromagnetism to pass through a particular material. Radiopacity is an essential property for all restorative materials¹. The patient may ingest (swallow) these fragments of denture fracture. Therefore, pure denture base is necessary to be radiopaque for easy radiological detection. In a previous study, by a review about 123 cases of swallowed or aspirated dentures or fragments of denture base demonstrated that need for radiopaque for detection these particles, the difficulties and time involved in locating these fragments of the denture bases². Incorporation of opaque filler such as metal or ceramic could be the way out. There were attempts to increase radiopacity by adding barium sulphate by ratio 8% to acrylic resin and however, this did not yield sufficient levels of radiopacity. Increasing the barium sulphate content by ratio 20% gives sufficient radiopacity but unfortunately it has a deleterious effect on the mechanical properties of the resin³.

The basic problems of the denture based to be easily radiologically detected and radiolucent because of C, O and H atoms are poor X-ray absorbers. The patient occasionally swallows dentures and may even inhale fragment of denture if involved in a violent accident such as car crash. Therefore, radiological detection fragment of denture is used to help the treatment of the patient^{3,4}. Early method for detection was through the incorporation of heavy-metal salts as physical mixtures with the polymers⁵. Another approach is through the incorporation of glass containing such as barium, aluminum, strontium, zinc and zirconia, respectively, to produce composites that are radiopaque (impenetrable by X-rays), thus enhancing their visibility on a

radiographic film⁶. This is important for diagnostic purposes and radiographs that can be used to differentiate between various specimens based on their radiopacities. Radiopacity of dental resin composites usually resulted from the incorporation of elements of relatively high atomic numbers such as Ba, Zr, Sr, V and La into the resin matrix⁵.

Many attempts were tried to achieve higher radiopacity values for PMMA as denture base material as well as bone cement. Dental materials were developed using heat-curing PMMA, prepared by mixing liquid and powder components. The liquid component contains, MMA as monomer, an inhibitor for preventing the premature polymerization of the monomer and promoter of initiator decomposition. The powder component contains, PMMA as polymer, a mineral powder such as barium sulfate (BaSO_4) or zirconium dioxide (ZrO_2) acting as radiopacity and free radical initiator (typically benzoyl peroxide, BPO)^{7, 8}. Abboud et al.⁷ investigated reinforcement using Al_2O_3 particles treated by γ -MPS. The experiments showed that 4% of Al_2O_3 in cement is sufficient for providing a good opacity for X-rays. A few of denture base materials contain heavy metal compounds or elements such as barium or radiopaque glass filler added to improve the radiopacity. It is necessary to add up to 20% by weight of these compounds to give sufficient radiopacity and this result in a reduction in the strength of the material and change in the appearance of the denture. The weight fraction of the filler and matrix in resin composites influenced the scattering and diffraction of the X-ray interaction⁹. Most of the prostheses investigated are radiopaque though the removable prostheses most likely to be radiolucent. For this reason, the removable prostheses, full acrylic denture were not visible while the upper partial acrylic denture was moderately visible¹⁰. For example, the PMMA denture base easily broken during accidents or when the patient applies high mastication force and also the patient may even accidentally ingest (swallow) these particles. Besides that, it is also radiolucent and cannot be different from soft tissues.

Therefore, the purpose of this study is to prepare heat curing PMMA denture base composites containing micron-size ceramic fillers particles ($\text{Al}_2\text{O}_3/\text{Y-TSZ}$) together with 7.5 wt% NBR (as an impact modifier) in PMMA composite and investigate their radiopacity compared to aluminum plate (reference standard), commercial PMMA and PMMA matrix. In dental application, reinforcement by $\text{Al}_2\text{O}_3/\text{Y-TSZ}$ as filler loading is possible provide to this property in PMMA denture base.

2. Materials and methods

The materials that used in this research are PMMA powder (Mw, 996.000 g/mol; Sigma Aldrich USA), benzoyl peroxide (Merck chemical, Darmstadt, Germany), and MMA monomer "Fluka, UK" with EGDMA (Sigma-Aldrich, USA), 99% purity Al_2O_3 particles (SulzerMetco, Westbury, NJ, 4.4 μm average particle size and density 2.70 g/cm³), Y-TSZ particles (containing 6.38 of Y_2O_3 stabilizer, GoodFellow Cambridge Limited, USA, 1.05 μm , average particle size and density 5.90 g/cm³), and NBR particles (Genzo Scientific Ent., Malaysia, less than 150 μm and density 0.98 g/cm³).

The PMMA denture base material was prepared using powder components mixed with liquid component. The powder components were comprised of PMMA powder, NBR as impact modifier particles, $\text{Al}_2\text{O}_3/\text{Y-TSZ}$ powder was added each other by 1:1 ratios as mixture filler and then, it was mixed with 7.5 wt % NBR particles using 1, 3, 5, 7 and 10 wt %, respectively. Each mixture was mixed for 30 min by using internal mixer (599957-K model, MS Instruments, Malaysia). The rotor speed was 50 rpm and heated at 65 °C. The mixing chamber was cleaned before next mixing process to avoid contamination. The liquid was comprised of 10 % EGDMA as cross-linking agent and 90% MMA as an activator containing 0.025 % hydroquinone as inhibitor (liquid component)¹¹.

Each of the powder mixture components were mixed with liquid component by hand mixing, respectively. The mixing of powder mixture to liquid medium (P/L) ratio was set at 2.5:1, according to standard dental laboratory usage³. The composite reached the dough stage (working stage) for easy forming of the paste around 15 min, the mixture was packed into the specific mold. After that, the mold was pressed under 14 MPa using a hydraulic press (Mestra 48150 Sondika-Bilbao, Spain) maintained under pressure for 30 min at room temperature. The curing process is carried out by placing the mold in a water bath. The temperature of water bath was kept at 78 °C for 90 min to complete the heat polymerization. The mold was removed from the water bath and then left to cool slowly to room temperature. The samples were removed from the mold, then, trimmed and polished by using emery paper 240. This procedure is in accordance to ISO 1567:2001 dentistry-denture base polymer standard method for preparing a conventional denture base in a dental laboratory³.

Radiopacity test was assessed according to ISO 4049–2009 standard specifications. The experimental procedure was carried out according to the following produces; the specimens, the aluminum plate and the film were irradiated with X-ray at 60 kV, 10 mA and the exposure time of 0.4 s using an X-ray machine (Toshiba KXO-15R, Japan). The aluminum plate had compositions of, at least 98% of aluminum, 0.1% of copper and 0.1% of iron present in plate. While, dimensions of aluminum plate, the length 50 mm \times 20 mm width, having a thickness range from 0.5 mm to 5.0 mm. The film density of each formulation's image was measured by optical density (OD) using a densitometer machine (RMI, USA). The OD of the test samples were then expressed in terms of equivalent aluminum thickness (mm) by reference to the calibration curve for the OD of the aluminum step-wedge (Fig.1)¹².

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