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Development and Characterization of New AR Glass Fiber-Reinforced Cements with Potential Medical Applications

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ABSTRACT: The aim of this study was to obtain and investigate new biomedical glass fiber resin cements with improved mechanical properties and radiopacity, using alkaline-resistant (AR) glass fibers. New light-curing, self-curing, and dual-curing resin cements were obtained from AR glass fibers and $BaSO_4$ powder mixed with 2,2-bis[4-(2-hydroxy-3-methacryloyloxypropoxy)-phenyl]propane and triethyleneglycol dimethacrylate monomers at different weight ratios, such as 5/20/75, 10/20/70, 20/20/60. The newly obtained cements were investigated for mechanical properties—compressive yield strength (CYS), compressive modulus (CM), diametral tensile strength (DTS)—as well as for radiopacity. Slight increases in CYS, CM, DTS and improved radiopacity with increasing amount of glass fibers were observed. The mechanical properties were found to increase in the order light-curing < self-curing < dual-curing. SEM images support the reinforcement of resin cements by glass fibers. © 2012 Wiley Periodicals, Inc. J. Appl. Polym. Sci. 000: 000–000, 2012

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INTRODUCTION

Fiber-reinforced composites (FRCs) have been introduced in dentistry as metal-free esthetic alternatives for purposes including prosthodontic bridges, single crowns, full coverage fixed partial dentures,[1] periodontal splints,[2] orthodontic retainers,[3] post core systems,[4] and implant prostheses.[5] There are also studies in which biocomposites are reinforced with bioactive glass,[6–8] radiopaque glass,[9] E-glass fibers,[10,11] ceramic fibers,[6] or glass cloth.[12] Fiber-reinforced polymer composites can also be found to be highly biocompatible for applications in orthopedics.[13] Marcolongo et al.[7] showed that bioactive glass fiber/polysulfone composites significantly increase the interfacial bond strengths after 6 weeks of implantation, when compared with polymer samples. E-glass fiber composites based on 2,2-bis[4-(2-hydroxy-3-methacryloyloxypropoxy)-phenyl]propane and triethyleneglycol dimethacrylate polymer matrix showed good biocompatibility under cell culture conditions, indicating that these materials have reasonable potential to promote bone–cell interactions and bone bonding.[14] A preliminary osteoblast culture studies on the S520 fiber surface were promising, showed proliferation, nodule formation, and mineralization.[15] To improve mechanical properties glass fibers were inserted in glass ionomer cement (GIC).[16–21]

In joint replacement surgery or in dentistry it is very important for the cement to be radiologically discernible from the surrounding bone or dental tissue. The radiopacity of dental restorative materials allows for detection of secondary caries,[22] the recognition of faulty proximal contours, voids, marginal adaptation, and interfacial gaps on a radiograph.[23] Polymers based solely on covalent bonds between carbon, hydrogen, and oxygen are not visible using typical medical imaging techniques (such as X-rays). The use of alkaline-resistant (AR) glass fibers instead of E-glass fibers may be advantageous by increasing the composite radiopacity, because of their Zr content. Alkaline-resistant (AR) glass fibers have a tensile strength of 3241 MPa and a modulus of 73.1 GPa, which are close to the values seen in E-glass fibers, 3445 MPa and 72.4 GPa, respectively.[24] All these properties make the AR glass fibers a promising alternative for biomedical applications.

Dual-curing resin cements have been used in dentistry for luting indirect esthetic restorations, metal castings (e.g., with crowns and fixed partial dentures), or as an alternative to zinc phosphate and GIC.[25] Recently, Schneider et al.[26] reported that a high-curing calcium phosphate nanocomposite could offer new possibilities to bone surgery of complex defects. In bone surgery, light-curing composites might be used for bone

replacement or as a glue for bone defect treatment,[26] provided that one could transmit light through the cement. Light-cured cement offers many clinical advantages over chemically cured cement, in terms of duration of working and of setting time. Dual-curing resin cements could be characterized by a longer self-curing reaction than self-curing resin cements and advantage of light-curing resin cements. In addition dual-curing resin cements present an advantage in clinical manipulation, allowing sufficient time to place the restoration in the right position and to remove the excess material before curing; or, when using a light-curing lamp, allowing a shortening of the time of manipulation and hardening of the cement. Dual-curing materials could be cured by chemical reaction if light-curing is not possible or is only partially transmitted in the materials. Until now, in none of the known biocomposites AR glass fibers have been used in combination with dual-curing system.

In this work, we aim to develop a new biomedical resin cements with improved mechanical properties and radiopacity, using AR glass fibers. Thus, several light-curing, self-curing, and dual-curing resin cements were designed and obtained, based on a bisphenol A-glycidyl methacrylate/triethyleneglycol dimethacrylate polymer matrix mixed with barium sulfate powder and reinforced with AR glass fibers as fillers. These new materials could have potential applications in dental and orthopedic fields.

EXPERIMENTAL

Materials

The reagent grade chemicals barium sulfate powder (Aldrich Chemical, Milwaukee, WI), 2,2-bis[4-(2-hydroxy-3-methacryloyloxypropoxy)-phenyl]propane (bis-GMA, Aldrich Chemical, Milwaukee, WI), and triethyleneglycol dimethacrylate (TEGDMA, Sigma Chemical, St. Louis, MO). *N, N*-dihydroxyethy-p-toluidine (DHEPT) and benzoyl peroxide (POB) were purchased from Merck–Schuchardt, Germany. Camphorquinone (CQ) and *N, N*-dimethylaminoethyl methacrylate (DMAEMA) constituted the photoinitiator system and were supplied by Merck–Schuchardt, Germany. Silane A-174 (γ-methacryloxypropyl-1-trimethoxysilane) was purchased from Aldrich Chemical, Milwaukee, WI, and used as an adhesion promoter for glass fibers. All materials were used as received without any further purification.

Glass Fibers

AR glass fibers having the length of about 1 mm and the diameter of about 23 μm were treated with 1 wt % γ-methacryloxypropyl-1-trimethoxysilane (silane A-174).[27] This silane is used as a coupling agent in dentistry and contains an ester functional group on one end, for bonding to the glass fiber surface, and a methacrylate group on the other end in order to make the filler compatible with the resin before curing, and able to connect to the polymer matrix when polymerization reaction starts.

Preparation of Glass Fiber-Reinforced Resin Cements

An experimental organic matrix (monomer mixture) was obtained from bis-GMA (60% by weight) and TEGDMA (40% by weight). From the experimental monomer mixture light-curing liquids were prepared by disolving DMAEMA (1% by weight) and CQ (0.5% by weight) in the monomer mixture. Two liquids (liquid A and liquid B) based on the same bis-GMA/TEGDMA mixture were employed in order to obtain self-curing liquids and dual-curing liquids. The self-curing system was based on POB (initiator) and DHEPT (activator). POB was disolved in liquid A (1.5% by weight) and DHEPT (1.5% by weight) were introduced in liquid B. Dual-curing liquids were obtained by dissolving POB in liquid A (1.5% by weight) and DHEPT (1.5% by weight), DMAEMA (1% by weight) and CQ (0.5% by weight) in liquid B. Light-curing, self-curing, and dual-curing liquids were mixed with the same ratio of inorganic fillers (glass fibers and barium sulfate powder). In the case of light-curing liquid the resulted paste is hereafter referred to as light-curing resin cement. From the self-curing and dual-curing liquids two pastes were obtained for each material (paste A and paste B). By hand-mixing at room temperature the components of paste A with paste B from self-curing or dual-curing was obtained self-curing or dual-curing resin cements. The final composition of resin cements is given in Table I.

Mechanical Tests

Samples for compressive strength (CS) tests were prepared by inserting the resin cement paste in a cylindrical Teflon mould (internal diameter of 4.0 $\pm$ 0.01 and length of 8.0 $\pm$ 0.01 mm) and compressing the composite material in mould with two glass plates at top and bottom of the mould. The light-cured resin cement samples were cured with a halogen curing lamp Optilux 501 (Kerr/Demetron) for 40 s from top and bottom and for 40 s from both sides in length in order to assure that the entire material was cured. Self-curing resin cements and respectively dual-curing resin cements prepared by hand-mixing of paste A with paste B were inserted in the same mould, and due to the radical polymerization of the monomeric phase samples, they solidified within 7–9 min. Dual-curing resin cements were additionally cured using the same protocol as for the light-cured resin cement samples. Samples for diametral tensile strength (DTS) were prepared by inserting the unpolymerized material (light-curing resin cement, self-curing resin cement and dual-curing resin cements) inside of the mold having 3 mm in depth and 6 mm in diameter and compressing the material in the mould with two glass plates. Light-curing and dual-curing samples were light-cured for 40 s at top and 40 s at the bottom, respectively.

After curing, the specimens were removed from the mould and those displaying voids were excluded from this investigation. Samples were measured with a micrometer and those samples with a thickness higher than the chosen value were sanded, using # 800 and 1200 SiC abrasive papers until their thickness was reduced to the selected values ($\pm$ 0.01). The test specimens (n = 8–10) were stored in water at 37°C for 24 h before the mechanical tests. The diameters and heights of the samples were measured using an electronic digital caliper (Vogel, Germany) with an accuracy $\pm$ 0.01 mm. Mechanical testing of the samples was carried out in a universal testing machine (LLOYD LR5Kplus) at a loading rate of 0.75 mm/min until fracture. The load deflection curves were recorded with a computer software (Nexygen; Lloyd Instruments, England). The compressive yield strength (CYS) in MPa was calculated at 2% offset strain[28] by eq. (1). Compressive modulus (CM) in GPa was determined from the slope in the elastic portion of the stress–strain curve.

Table I. Composition of Resin Cements

Code	Composition of filler (wt %)P	Organic phase composition (wt %) L	P/L (wt %)
P		Polymer (100%)	0/100
C10Ba	$BaSO_4$ (10%)	Polymer (90%)	10/90
C20Ba	$BaSO_4$ (20%)	Polymer (80%)	20/80
C5F	Glass fibers (5%), $BaSO_4$ (20%)	Polymer (75%)	25/75
C10F	Glass fibers (10%), $BaSO_4$ (20%)	Polymer (70%)	30/70
C20F	Glass fibers (20%), $BaSO_4$ (20%)	Polymer (60%)	40/60

$$CYS = F/\pi r^2 \quad (1)$$

where F is the applied load (N), and r is the radius of the cylindrical sample measured before testing (2 mm).

DTS expressed in MPa[29–31] was calculated using eq. (2).

$$DTS = 2F/(\pi d t) \quad (2)$$

where F is the applied load (N), d is the diameter of the cylindrical sample measured before testing (6 mm), and t is the thickness the cylindrical sample measured before testing (3 mm).

The Film Thickness

The film thickness of composite materials ($n = 5$) were made in accordance with ISO 4049 : 2000.[32] Amount of 0.05 g of cement materials were dispersed between two glass plates. The samples were compressed until 150 N (± 0.01) for 180 s using the universal testing machine (LLOYD LR5Kplus). After 180 s the load system was released and the samples were irradiated through the centre of the upper glass plate for 80 s. After irradiation the plates were removed from the loading device and the film thickness of cement was measured using a micrometer (Vogel, Germany) with an accuracy ± 0.001 mm (± 1 μm).

Scanning Electron Microscopy (SEM)

The sample morphology was investigated by scanning electron microscope (SEM, JEOL, JSM 5510 LV), using the secondary electron imaging (SEI) technique. After the DTS test, the structure of fractured surfaces of samples of C20Ba, C5F, C10F, and C20F composites was explored using a JEOL, JSM 5600 LV instrument, equipped with an EDS (energy dispersive X-ray spectrometer, Oxford Instruments), using the backscattered electron (BSE) imaging technique. Surface of a glass fiber from C20F composites was investigated using EDS analysis in order to evaluate the quantity of major oxides from glass fiber composition.

Radiopacity

Four disks of each resin cement, measuring 8 mm in diameter and 1 mm (± 0.01) in thickness, were selected for investigation. Samples were measured with a digital caliper and those samples with higher thickness than 1 mm were sanded until their thickness was 1 mm (± 0.01). Two freshly extracted human molars and one premolar tooth extracted for orthodontic purposes, all of which on visual examination were free from caries, hypoplastic defects or cracks, were selected for the current study. The teeth were embedded in acrylic resin and one mesiodistally section with a 1 mm (±0.01) thickness was obtained from each of the three teeth, using a rotary cutting machine (IsoMet Buehler, Lake Bluff, IL). In addition to these samples, pure aluminum samples consisting of 1–8 mm thick step were prepared. The resin cement samples, teeth slices and the aluminum step wedges were placed on an intraoral sensor. The images were taken using an intraoral sensor system XIOS Plus (Sirona) and a dental X-ray machine Intraoral X-Ray Soredex (Minray) at 70 kV, 7 mA, 0.04 s with a target- sensor distance at 30 cm. The mean gray value of each aluminum stepwedge and selected materials were measured by outlining a region of interest by using the equal-density area tool of the Image J software (version 1.37 V). The regions were selected by avoiding areas containing air bubbles inside the material and the average gray value was recorded for every sample. For each radiograph image a calibration curve generated by the gray scale values as a function of the aluminum thickness was calculated. The radiopacity values of the samples were expressed in terms of the equivalent thickness of aluminum per 1 mm unit thickness of material.

Statistical Analyses

Data were statistically analyzed by one-way analysis of variance (ANOVA) SPSS (Version 11.5, SPSS Inc.) software package, with Tukey's test with the level of significance set at 0.05 in order to determine the significant differences between the mean values of the tested materials.

RESULTS AND DISCUSSION

In vivo, the forces applied to a material used for dental restoration, bone defect or bone replacement could develop a complex stress distribution in the materials with various modes of fracture. Strength tests such as CYS, CM, and DTS were proposed to determine the limit of performance of a material to stress. $BaSO_4$ powder has been used in many bone cement products on the market. This was the reason for us to choose and test the C10Ba and C20Ba resin cements as references in our study. CYS [Figure 1(a)] ranged from 80.38 to 107.72 MPa for light-cured resin cements, from 88.16 to 120.12 MPa for self-cured resin cements and 92.30 to 128.89 MPa for dual-cured resin cements. Addition of glass fibers in the cement composition led to an increase in CYS and CM values, in the following order: C5F < C10F < C20F. Thus, the CM values given in Figure 1(b) showed values between 1.39 and 1.71 GPa for light-cured resin cements, 1.54 and 1.98 GPa for self-cured resin cements and 1.60 and 2.08 GPa for dual-cured resin cements. The trends in

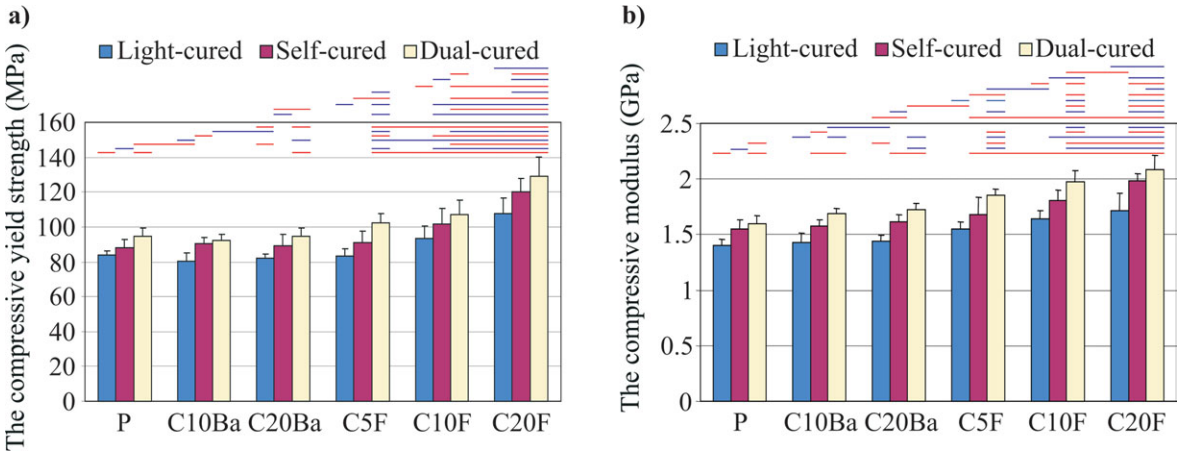

Figure 1. (a) The results of CYS of resin cements; (b) The results of CM of resin cements. (Horizontal bars indicate mean values statistically significant different from each other, when compared using the Tukey test, $P < 0.05$). [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com].

these CYS values were similar to those seen in the CM [Figure 1(b)], and they may all be related to the increases in filler content and in fiber amount within composite materials. A lower mechanical strength (CYS, CM, and DTS tests) for C10Ba and C20Ba composites could be explained by the absence of chemical bond between the polymer and the inorganic particles. CYS and CM tests allowed for an assessment of the mechanical behavior of the materials in the elastic regime. If the force load will increase up to the yield point, the material will have a plastic deformation. According to the literature CYS and CM of different bone cement could be in range of 72.6–113.3 MPa and 1.95–3 GPa.[33] Krause et al.[28] investigated the compression, uniaxial tension and fracture toughness of dental composites based on bis-GMA/TEGDMA with 40, 50, 60, and 70% glass fibers, finding that the elastic modulus, tensile strength and CS are dependent on the filler content in composites. The fracture toughness values showed an increase with increasing glass fiber content up to 50%, while at 60% and higher it remained constant. The authors[28] concluded that a 50% of silanized glass fibers would provide the optimum mechanical and handling properties. Our results showed higher CYS values and lower CM values than those obtained by Krause et al.,[28] and this could explained by the difference in the composition of the materials. The addition of barium sulfate powder from 10 wt % to 36 wt % was found to improve bone cement radiopacity; however, it also creates local stress concentrations or fatigue crack initiation sites and leads to a decrease in tensile and fatigue properties.[34] This change in the amount of barium sulfate content showed a small increase of elastic modulus and a maximum compressive of only 13 wt % and 5 wt %, respectively.[34]

DTS testing is a common method for measuring tensile strength of brittle materials. The diametral compressive strength test is also known as the the diametral tensile test, Brazilian disk test, indirect tensile test, compact crushing test, or compact hardness test.[35] Addition of glass fibers to glass–ionomer cements led to increases in DTS values to 18 MPa, 1.8 time higher than those of glass–ionomer cements without glass fibers.[18] Obtaining of glass ionomer cements with addition of glass fibers in 3 wt % and 5 wt % glass fibers (1 mm length) sled to increased DTS, flexural strength, flexural modulus, and fracture toughness.[29] DTS values (Figure 2) were registered between 27.99 and 39.78 MPa for self-cured resin cements and between 28.43 and 40.98 MPa for dual-cured resin cements. There was a small increase in the DTS values for dual-cured resin cements compared with self-cured resin cements. Addition of $BaSO_4$ powder showed a smaller increase of DTS values (Figure 2) for C10Ba and C20Ba resin cements than for polymer samples. The dual-curing system used in this work, improved mechanical properties of the experimental materials. The DTS results were in agreement with other results for direct core build-up materials of 27.2–51.2 MPa,[30] 19–55.1 MPa,[31] or the limits imposed by ADA specifications for direct filling resins Type I and II materials (24 and 34 MPa).[36] Kim et al.[9] showed that DTS of dental composites could be increased if bis-GMA was replaced by bis-M-GMA (2,2-bis[4-(2-methoxy-3-methacryloyloxy propoxy)phenyl] propane). The higher values of DTS obtained by Kim et al. compared with the values obtained in our study could be explained by the higher amount of glass fillers (75 wt %) used in dental composites.[9] The mechanical properties increase in the following order: light-cured resin cements < self-cured resin cements

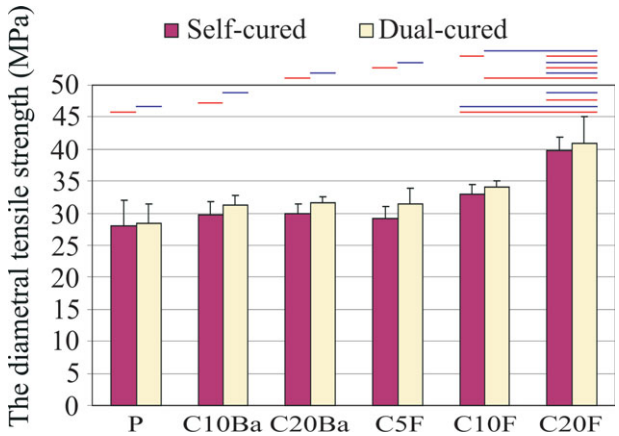

Figure 2. The results of DTS of resin cements. (Horizontal bars indicate mean values statistically significant different from each other, when compared using the Tukey test, $P < 0.05$). [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com].

< dual-cured resin cements. Studies indicate that the use of dual-curing systems (photo initiation and chemical initiation) increases the conversion of double bonds within dual-curing resin cements.[37–39] This could explain in our study increasing mechanical properties for dual-curing composites.

The resin cements showed the mean film thickness from 26.6 μm (C10Ba), 37.2 μm (C20Ba), 47.1 μm (C5F), 98.6 μm (C10F), and 203.2 μm (C20F). According to ISO standard 4049 : 2000[32] the film thickness can be acceptable up to 50 μm, but it may be even more depending on different clinical situations.[40] Some article reported the film thickness of luting materials from 7 to 152 μm, depending on the material tested.[41,42] The highest bond strength values for glass fiber posts luted with different cements were obtained when cement thickness was from 0.1 to 0.3 mm[43] and a thickness more than 2.0 mm could on the masking of various types of opaque posts.[44] Several studies in cemented total hip arthroplasty (THA) have emphasized a minimum thickness of 2–3 mm provide to yield a better long-term radiographic outcome,[45,46] thickness from 2.4 to 3.7 mm caused substantial strain reductions in the distal cement (40–49%) and may increase the fatigue life of a bone-implant system by reducing peak strains within the cement.[47]

SEM images (Figure 3a) showed some heterogeneity in the materials, which could be explained by the lack of a chemical bond between the $BaSO_4$ powder and the polymer matrix. The highest DTS values were obtained for the C20F FRCs. This behavior could be explained by the larger filler load in the composites and also by presence reinforcing effect of the glass fibers. The fibers will delay initiation and propagation of cracks in the composite materials, by bridging through the cracks that appear in the material when a force is applied. The fibers that bridge the cracks will increase the resistance of the material for propagation of the fissures in the direction of fracture. This was in agreement with other publications.[21] The SEM images [Figure. 3(b–f)] illustrate an increased percentage of glass fibers in the order C5F < C10F < C20F, in good agreement with the increasing content of glass fiber employed in preparing the samples. In the images, the fibers are found to be orientated in various directions. The fractured surface of fiber composites was intact, without cracks in the polymer matrix. Regarding the fractured surface of composites (Figure 3), one may observe the fibers broken across their entire diameter at the level of the fracture surface (red arrows), the spaces left within the surface after pulling out glass fibers (light yellow arrows) and the fibers that remained after fracturing material (tan arrows); additionally, small patches of polymer composite adhered to the glass fibers surface after pulling out may also be observed (green arrows). Lohbauer et al.[20] showed that pull-out fibers in fractures from composite materials correlate well with increased fracture toughness and work-of-fracture. The SEM images [Figure 3(g)] show that the polymer matrix adheres to the fiber surfaces, indicating a good interfacial interaction.

Materials such as polymeric, quartz, or silica are not radiopaque and in consequence it is difficult for restorations to be detected on radiographs. To improve radiopacity, bone/dental cements have been prepared by incorporating radiopacifying agents, which contain elements with a high atomic number.[23,48–50] These radiopaque elements may be present in a wide range of concentrations and combinations in composites. A higher percentage of fillers with high atomic numbers in composites lead to increased radiopacity.[23,48–50] The commercial bone cements are usually made radiopaque by the addition of an inorganic compound, such as barium sulfate or zirconium dioxide.[49] Kobayashi et al.[50] showed that 30% $BaSO_4$/PMMA was more clearly visualized by micro-CT images than the 10% $BaSO_4$/PMMA in all vertebrae. Addition of barium sulfate (10, 20, 30, and 40%) to polymethylmethacrylate (PMMA) bone cement increased radiopacity but at 40% barium sulfate concentration the cement was significantly more fragile than the cement with lower barium concentrations.[51] The enamel is the hardest and most highly mineralized substance of the body and consists of 97 wt % hydroxyapatite.[51] The dentine has a composition very similar to that of the bone tissue. The content in minerals is 70 wt % in dentin and 65 wt % in bone.[52] The radiopacity of materials investigated (Figure 4) showed values between 0.31 (C10Ba), 0.93 (C20Ba), 1.30 (C5F), 1.76 (C10F), and 2.05 (C20F) mm Al. The radiopacity value of 0.97 mm Al (Figure 4) obtained in our study for 1 mm dentin could be used as reference for bone. C10Ba composites had the lowest radiopacity and were significantly different in this respect enamel and dentin. Increasing addition of $BaSO_4$ to 20 wt % showed a radiopacity higher than human dentin. While the $BaSO_4$ content was the same in composites C5F, C10F, and C20F (Table I), the increase in glass fiber content led to an improvement of radiopacity (Figures 4 and 5) in the order: C10Ba < C20Ba < C5F < C10F < C20F. This could be explained by the increased amount of reinforcing filler and zirconium content in the glass fiber [Figure 3(g)]. The EDS analysis of glass fiber [Figure 3(g)] showed the inorganic oxides: SiO_2 (63.23 wt %), Na_2O (16.08 wt %), Zr_2O (15.71 wt %), CaO (4.00 wt %), and Al_2O_3 (0.97 wt %). From all these inorganic oxides, zirconium oxide (15.71 wt %) improves the radiopacity of materials because of the high atomic number of Zr. Besides improving radiopacity, AR glass fibers are less susceptible to degradation compared with E glass fibers because of their 15–20% ZrO_2 content.[53] The zirconium content from the AR glass fibers may allow one to lower the amount of extra radiopaque agent added to the cements for increasing the radiopacity. E-glass fibers have been used in dentistry.[1–5,18] and orthopedic fields[10] but this glass did not have radiopaque elements in its composition. The improvement of the radiopacity of resin cements using AR fibers was for the first time mentioned in our work for possible biomedical applications, including possible replacement of E-glass fibers. The incorporation of too much metal oxide in fillers used in dental composites may be a disadvantage because barium or strontium ions can disrupt the alumino-silicate network[54] and can increase the solubility and degradation of dental composites.[54,55] Also, using radiopaque oxide powders ($BaSO_4$, ZrO_2, Yb_2O_3, and so on) in bone cements could limit mechanical properties, as opposed to the case when the radiopaque element is in an AR glass structure. The radiopacity values for the experimental glass fiber resin cements tested were influenced of fibers content in cement, and were higher than 1 mm Al, a limit requested by ISO 4049 : 2000[32] for a dental cement product to be used clinically. Composites with glass fibers evaluated in this study had radiopacity value comparable with other dental cements[56] or dental composites[23] from the market.

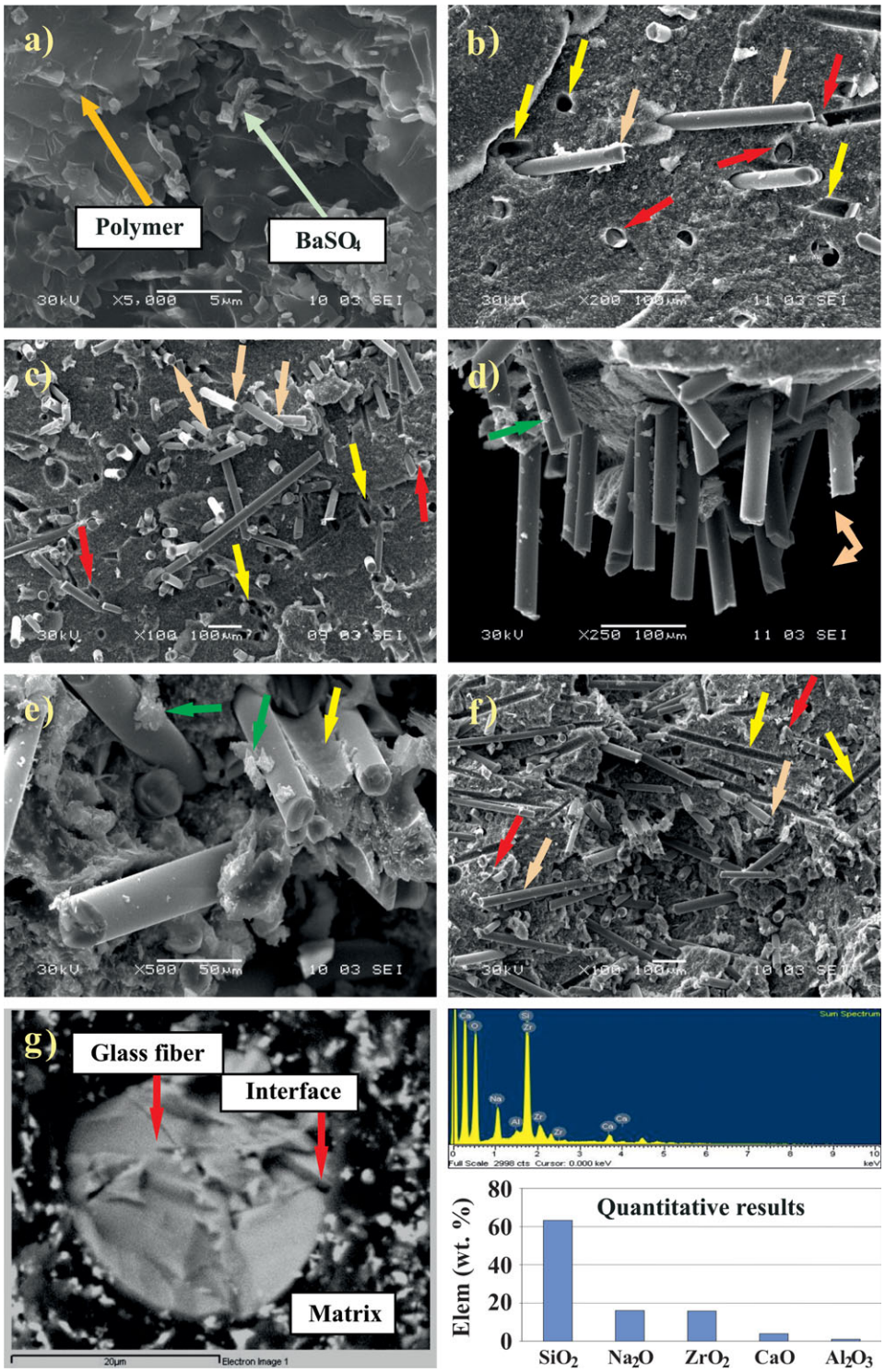

Figure 3. SEM micrographs of fractured surface of resin cements after DTS test. a) C20Ba composites; (b) C5F glass fiber-reinforced resin cement; (c) C10F glass fiber-reinforced resin cement; (d), (e), and (f) C20F glass fiber-reinforced resin cement; (g) SEM micrographs and EDS analysis of section of glass fiber from glass fiber-reinforced resin cement. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com].

The self-curing resin cements proposed in this study could be used as materials in bone surgery and, if the light-curing is possible, as glue for bone defect treatment.[26] Increasing the elastic modulus of bone cements with reinforcements has important implications in contributing toward an improved load transfer from the prosthetic stem to bone[57] and toward reducing the fracture of cements and the prosthesis failure. Improving mechanical properties had a positive effect to prevent bone loss at the proximal end of the prosthesis, implying an improvement in implant life.[57] Because the AR glass fibers are white, the AR

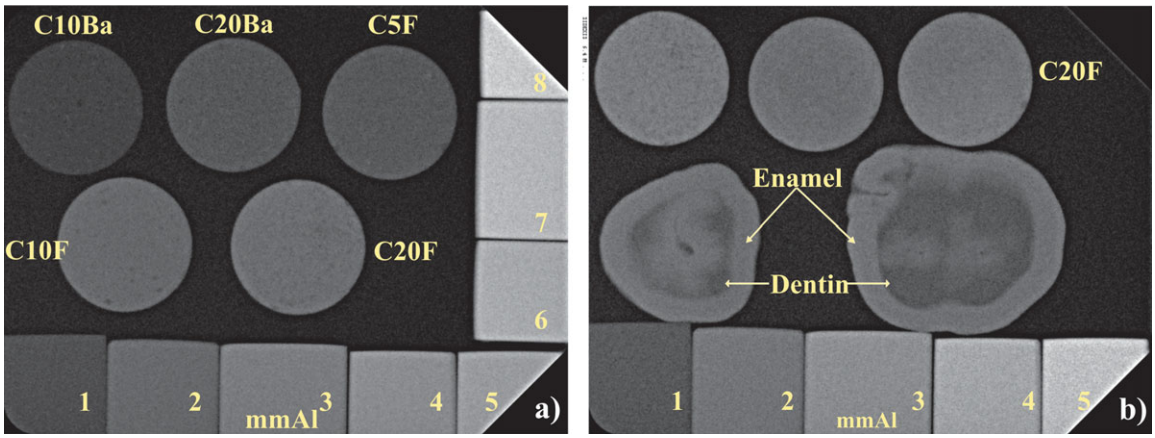

Figure 4. Radiopacity of resin cement. (Horizontal bars indicate means values not statistically significant different from each other compared using the Tukey test, $P > 0.05$). [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com].

glass fiber-reinforced resin cement can be considered an aesthetical one. A high conversion degree can be achieved because of the light transmission properties of this kind of glass fibers. The AR glass fiber-reinforced resin cement could be used as luting agent for inlays, onlay, crowns (C5F composites), postluting into the root canal (C5F and C10F composites), and for core build-up or post build-up (C10F and C20F composites). The materials studied here could have the potential for use in biomedical application. However, other studies regarding the physical properties and clinical studies are needed.

CONCLUSIONS

The CYS, CM, DTS, and radiopacity of AR glass fiber-reinforced resin cements show increasing values with increasing glass fiber content. The mechanical tests reveal that the mechanical properties increase in the following order: light-curing < self-curing < dual-curing resin cements. The new glass fiber resin cements with improved mechanical strength (CYS, CM, and DTS) and radiopacity could be useful in places in the body with high mechanical stress. SEM micrographs confirm the reinforcement resin cement with glass fibers. These results encourage us to pursue further investigation *in vitro* and *in vivo* for clinical application of these materials.

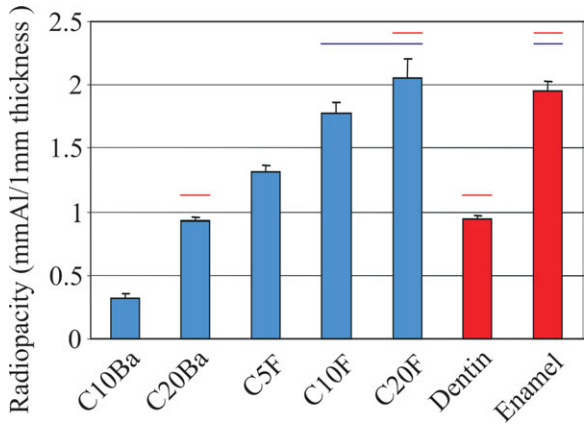

Figure 5. Radiopacity of resin cements and Al step wedges. (a) one sample of C10Ba, C20Ba, C5F, C10F, and C20F resin cement, (b) C20F resin cement samples and human dentin and enamel. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com].

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