FILM THICKNESS, DIAMETRAL TENSILE STRENGTH AND FRACTURE SURFACE OF ALUMINA REINFORCED DENTAL CEMENT

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Abstract. An ideal film thickness and optimum diametral tensile strength are important for the longevity of dental cement. This study aims to investigate the effects of different concentrations of alumina on the properties of dental cement derived from silica obtained from rice husk. Alumina reinforcements (3 and 10 wt%) were used in the formulation. Negative control specimen (0 wt% alumina) and commercial dental cement Rely-X (3M ESPE) were also prepared concurrently for comparison purpose. All the specimens were analysed for film thickness (FT), diametral tensile strength (DTS) and fracture surfaces. One-way ANOVA and post-hoc Tukey's test were used to analyse the data. Results showed that FT was significantly increased with the addition of alumina reinforcement (3 and 10 wt%) ($p < 0.05$). DTS values increased with the addition of alumina compared to negative control (0 wt% alumina) ($p > 0.05$). However, the differences were not statistically significant for 3 wt% and 10 wt% alumina ($p > 0.05$). Fractographic analysis showed the presence of crack branches with reinforcement of alumina which indicates the deviation of stress distribution to support force applied.

Keywords: dental cement, rice husk, alumina, tensile strength, fracture surface

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Introduction

Dental composite cement is a synthetic resin cement consisting of resin acrylic base, a filler such as natural silica or glass, and a silane coupling agent as the main components. In dentistry, resin-based dental cement has been used widely for multi dental specialties. Dental cement supports most dental prostheses in place of the tooth structure. Hence, the dental cement must be stable physically and mechanically. Alumina has been used since 1970 to strengthen dental materials and its improvement of particles grows soon after for better performance. Previous studies report the reduction support of force distribution of alumina incorporated with dental composite due to agglomeration [1-2]. Nevertheless, hybrid alumina and silica enhance mechanical strength [1, 3-4].

The addition of hybrid fillers results in better filler dispersion within the resin matrix and enhance mechanical strength such as hardness, compressive strength, and diametral tensile strength [1, 5-7]. Besides, the hybrid fillers affect the film thickness which is one of the essential properties of dental cement [8]. The film thickness is the height between two flat surfaces separated by cement. The film thickness influences the usage of dental cement in the dental clinic to avoid excessive material affecting cost and longevity [9]. Use of silaned hybrid fillers is reported to increase the film thickness and bonding in the matrix resin [10, 12-13].

Due to the significance of the hybrid fillers, other studies also have investigated a variety of filler types depending on their content percentage, shape, and size [3, 12-13]. Mesoporous natural or synthetic silica can be produced with a wide range of sizes. In addition, the manufacturer used other fillers such as alumina and zirconia to improve the physical and mechanical properties with no or minimal cytotoxicity of dental resin cement [5, 14]. It is important to avoid agglomeration with the higher concentration of filler loading [4, 15]. The addition of alumina has improved the diametral tensile strength but agglomeration occurred at higher filler loading [4]. However, an optimum concentration of silane coupling agent used to modify surface of alumina filler have shown homogeneity of dental composite [1, 16].

An ideal film thickness of dental cement is essential to ensure the accurate seating for dental restoration to tooth structure [17]. While diametral tensile strength can be used to investigate the brittleness of dental composite, Noushad et al. have shown that silica rice husk improves hardness, compressive strength and flexural strength of dental composite [3]. Modification of the existing dental composite with alumina may enhance its mechanical properties. To the best of our knowledge, studies of film thickness and diametral tensile strength of dental cement using silica rice husk reinforced with alumina are not found in any published literature. In this study, silica extracted from rice husk and alumina have been treated with a silane coupling agent and used to produce a hybrid dental cement. This study aimed to investigate the effect of adding alumina with different percentages into the newly developed experimental dental cement. Film thickness, diametral tensile strength, and fracture surface of the dental cement composites were measured. Fractographic analysis after mechanical testing such as compressive strength, diametral tensile strength and flexural strength were analysed.

Materials and Methods

Materials. Composite resins used in this study were bisphenol A-glycidyl methacrylate (Bis-GMA) (Esstech, Inc., Essington, PA, USA), urethane diamethacrylate (UDMA) (Sigma-Aldrich, USA), and triethylene glycol dimethacrylate (TEGDMA) (Sigma-Aldrich, USA). Alumina powder (Al_2O_3, < 50 nm) (Sigma-Aldrich, USA) and silica rice husk were the fillers. For the surface treatment, γ-methacryloxypropyltrimethoxysilane (γ-MPS) (Sigma-Aldrich, USA) was used as a silane coupling agent. DL-champhorquinone (CQ) (Merck, Schuchardt OHG, Germany) acts as a photoinitiator. The amide co-initiator used was (2-dimethylaminoethyl) methacrylate (DMAEM) (Merck, Schuchardt OHG, Germany). Rely-X U200 (3M ESPE, USA) was used for comparison purposes.

Fabrication of experimental dental cement. Silica and alumina were treated with γ-MPS. Characterisation of the treated silica and alumina was done with the field emission scanning electron microscopy (FESEM) (Quanta 650 SEM, FEI, Germany) and the accelerated surface area and porosimetry system (ASAP 2020, Micromeritics Instrument Corporation, USA). The sample was degassed at 50 °C for 24 hours. Dental cement was prepared using treated silica obtained from rice husk by following the steps reported in the previous study [3] with a modification on the ratio of resin used and the addition of UDMA. The use of UDMA was to promote the adhesion of the experimental dental cement. Besides, UDMA forms both chemical and mechanical bonds to the dentin [18]. Then, the treated alumina was added into the dental cement for improvement of physical and mechanical properties.

Table 1 shows the formulation of experimental dental cement. Bis-GMA, UDMA, TEGDMA, 0.5 wt% CQ, and 0.5 wt% DMAEM were mixed into a porcelain dish. Then, treated silica and treated alumina were added into the mixture. This new cement was vortexed for 2 minutes and covered with aluminum foil. The process of preparation of the dental cement is summarized in the Figure 1 A. This dental cement was newly developed as an adhesive cement for bonding.

Table 1. Formulation of experimental dental cement

Groups	Filler/Resin (30/70)		
	Filler loading (%)		Resin
	Silica	Alumina	
CTR	30	-	BisGMA/UDMA/TEGDMA (40/20/40)
3 wt% alumina	30	3	
10 wt% alumina	30	10	
Rely-X U200	N/A	N/A	Bis-phenol-A-bis(2-hydroxy-3-methacryloxypropyl)ether (BisGMA), TEDGMA [19]

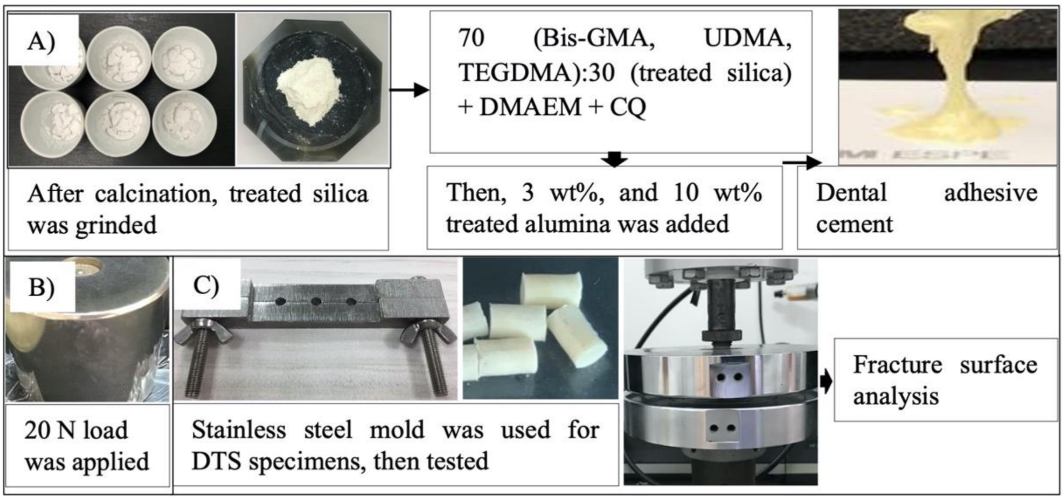

Figure 1. A) Preparation of experimental dental cement B) Load applied before measurement of FT and C) DTS specimens prepared and tested

The experimental dental cement were divided into 2 groups based on the percentage of alumina filler reinforcements: 3 wt % alumina and 10 wt % alumina. Experimental dental cement without alumina reinforcement (CTR) as negative control and commercial dental composite resin, Rely-X U200 (3M ESPE, USA) were used. The selection of the Rely-X U200 was due to the fact that this luting cement is the newest generation of self-adhesive cement and dual-cure clinically approved for multiple indications. The experimental dental cements were subjected to the test for the evaluation of their physical and mechanical properties: Film thickness (FT), diametral tensile strength (DTS), and fracture surface as shown in Figure 1 B and C. Descriptive statistics of FT and DTS were calculated, and Tukey's post hoc multiple comparison test (one-way analysis of variance) with significance level at $p<0.05$ was carried out.

Film thickness. A total of 40 specimens (n=10/group) were prepared for the FT test at 25 °C. Two flat square glass plates 2 cm in width and 5 cm in length were used [9, 20]. All groups of experimental dental cement were taken out from the storage were kept at room temperature for at least 1 hour before the film thickness test. The Rely-X U200 was mixed according to the manufacturer's instruction before the test measurement. Based on the International Organisation of Standardisation (ISO) 4490:2019, 0.1 ml of each sample was placed centrally between the two plates [20-21]. A vertical load of 20 N was applied on the top plate for 60 seconds. Then, the load was removed and the thickness of the two glass plates, with the interposing cement, were measured with a digital micrometer (Mitutoyo, Japan) which was recalibrated after each measurement. An average of three values was recorded for each specimen. The difference in thickness of the two plates before and after cement placement was calculated as the FT.

Diametral tensile strength test. A total of 40 specimens (n=10/group) were tested for DTS test using the Instron Universal Testing Machine (3366, USA) at a crosshead speed of 1mm/min. The samples comprised of 10 cylindrical specimens from each study group with a measurement of 4 mm in diameter x 6 mm of height using a split stainless steel mold and cured 2 mm incrementally using the light-emitting diode curing unit (SmartLite iQ2, Dentsply, USA) with the wavelength range between 450 to 475 nm. Each increment was cured for 20 seconds. The formed specimen was additionally cured for 20 seconds. The specimens were polished with aluminum oxide (coarse, medium, fine, and superfine) disc (Sof-Lex, 3M ESPE, USA) with a slow-speed handpiece in a sequence of 60 seconds. All specimens were kept in distilled water at room temperature for 24 hours before the DTS test.

Fracture surface analysis. Based on the results of the DTS test, the fracture surface of a specimen with the closest mean value was analysed using the FESEM and energy dispersive spectrometry (EDX). A sputter coating was done to the each specimen before testing with the FESEM and EDX.

Results and Discussion

***Filler characterisation*.** Figure 2 shows FESEM micrographs of mesoporous spherical silica and irregularly flake-shaped alumina particles at a magnification of 10,000x. From the FESEM results, the size of treated silica and treated alumina particles ranged between 53-356 nm and 3.34-9.85 µm, respectively. Representative FESEM micrographs revealed a homogenous distribution pattern of the filler particles.

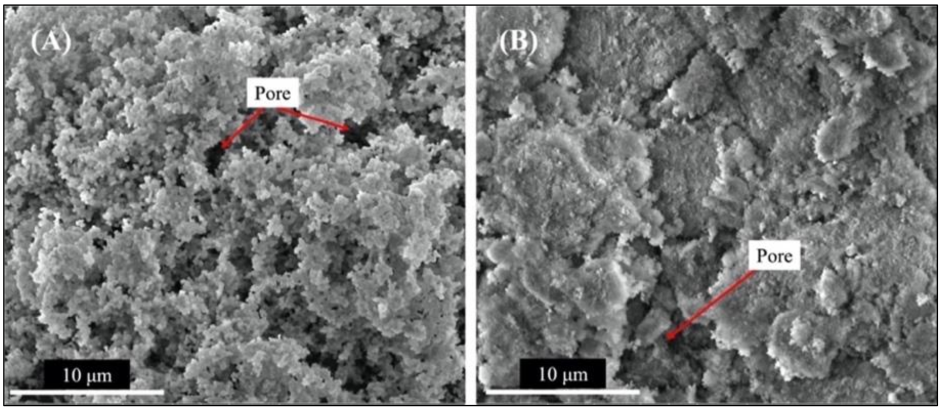

Figure 2. FESEM micrographs on magnification of 10, 000x: treated (A) silica and (B) alumina

By using the porosimetry system (ASAP 2020, Micromeritics Instrument Corporation, USA), silica particles were found to be 31 nm for average pore size larger relative to 8 m^2/g, the Brunauer, Emmet and Teller (BET) surface area. Previous studies showed that a silica filler with a small surface area is produced for the best mechanical property in a composite [10, 22], whereas, alumina was found to be 126 nm for average pore size larger relative to 21 m^2/g, the BET surface area. Figure 3 shows the isotherm curves and pores size distribution of treated silica and treated alumina fillers. The pore size distribution of the fillers were plotted using Barrett Joyner Halenda (BJH) method. The finding showed that the pore size distribution of treated silica was narrower than treated alumina. The FESEM results approved that the treated alumina with flaked shape and a larger size produces larger pore size than the treated silica. The treated alumina with large size has more extended time to achieve equilibrium contributing to large pore size, which is an agreement with Lyu et al. [23].

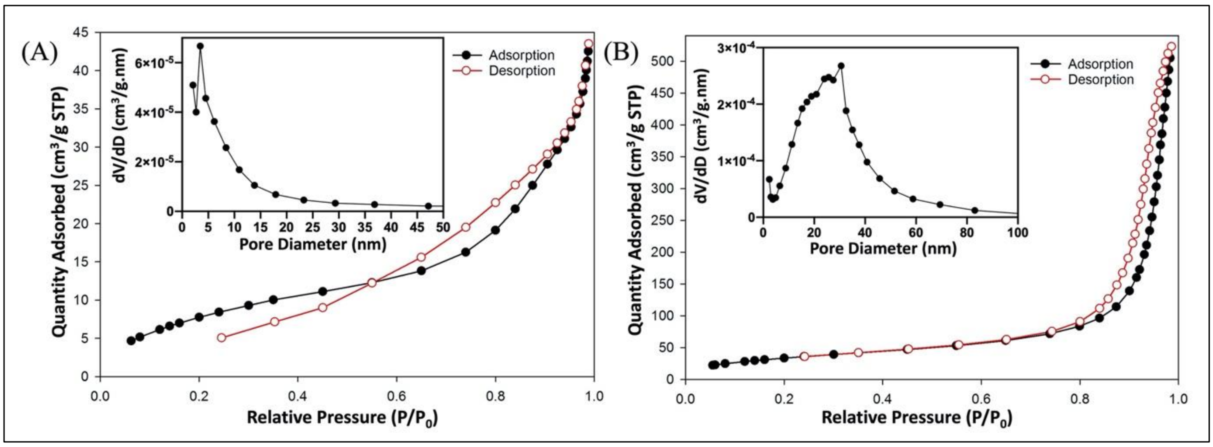

Figure 3. Nitrogen adsorption-desorption isotherms and pore size distribution of treated (A) silica; and (B) alumina

Based on the IUPAC classification, the BET graphs suggest that it belongs to the type IV isotherm and hysteresis loop type H1 for treated silica. The findings confirm that silica is mesoporous (2-50 nm) and cylindrical pore that is in agreement with Noor Sheeraz et al. [22], whereas a type III isotherm and hysteresis loop type H3 for treated alumina that is close to slit-shaped pore result found by Long et al. [24]. From this study, it showed that the variety of size, shape, and porosity range enhance the physical and mechanical strength of the hybrid dental material, correlating with previous studies [25, 26].

Film thickness of specimens. Table 2 shows the mean (SD) values of film thickness (FT) for each group of specimens. For the FT test, the result shows significant difference among study groups. However, there was no significant increase with the addition of 3 wt% alumina while there was a significant increase with 10 wt% alumina compared to the CTR group. The outcome shows FT improvement by addition alumina, although it is inferior to the Rely-X U200 group. Besides, the FT results were 15.5 to 17.8 µm which is less than 50 µm, the maximum FT allowable according to the ISO 4049:2019 [20] to achieve the function of successful dental adhesive cement.

Table 2. Film thickness of the experimental dental cement

Dental Luting cement	Film thickness (µm) Mean (SD)	F statistic^a (df)	*p* value^a
CTR	15.505 (0.522)^b		
3 wt% alumina	15.564 (0.570)^c	38.270	0.000
10 wt% alumina	16.886 (0.744)^{bc}	(3, 36)	
Rely-X U200	17.818 (0.391)^{bc}		

Statistical analysis was carried out using One-way ANOVA^a, followed by post-hoc Tukey's test. Significance level set at $p = 0.05$. The same letter indicates a statistically significant difference ($p < 0.05$).

Diametral tensile strength of specimens. An optimal filler concentration and dispersion are important to improve the physical and mechanical properties. Table 3 shows the diametral tensile strength (DTS) of the study groups. Alumina reinforcement in this study showed an increase DTS of dental cement. The possible reason for this finding could be that hybrid treated silica and alumina strengthen the microstructure of the resin matrix and reduce the volume of pores, which is close to the study by Souza et al. [27] added alumina into glass ionomer cement.

However, a decreased DTS when loaded with 10 wt% alumina despite not being statistically significant to 3 wt% alumina. Ma et al. also found a decreased DTS for 10 wt% alumina due to agglomeration [4], while, the Rely-X U200 was significantly lower than 3 wt% alumina. Previous studies also found that the Rely-X U200 resulted in lower DTS compared to UDMA contained self-adhesive cement [19, 28]. In contrast, Borges et al. [17] found a higher DTS value for Rely-X U200.However, further explanation remained due to limited information of Rely-X U200. Nevertheless, the feed rate of the mixture when increasing filler content is standardized to avoid any discrepancy of DTS value [29].

Table 3. Diametral tensile strength of the experimental dental cement

Groups	Diametral tensile strength (MPa) Mean (SD)	F statistic[a] (df)	*p* value[a]
CTR	76.655 (28.680)		
3 wt% alumina	85.722 (28.742)[b]	2.989	0.044
10 wt% alumina	83.160 (13.634)	(3, 36)	
Rely-X U200	57.582 (18.472)[b]		

Statistical analysis was carried out using One-way ANOVA[a], followed by post-hoc Tukey's test. Significance level set at $p = 0.05$. The same letter indicates a statistically significant difference ($p < 0.05$).

Fractographic images of specimens. Fractographic images (arrows indicate crack propagation) of dental cement after DTS were obtained using FESEM, as shown in Figure 4. The secondary electron (SE), backscattered electron (BSE) and EDX mapping showed different elements compared to the dental cement. CTR group showed a pulled-out fracture behaviour. Whereas, crack branches were seen with alumina reinforcement for 3 wt% alumina, 10 wt% alumina, and Rely-X U200. The findings are in agreement with previous studies that crack branches associated with alumina reinforcement contributing an increased DTS values [1, 5, 16]. The possible reason would be the silanisation of silica and alumina that enhance interface adhesion and distribute forces to strengthen the resin matrix, which is in accordance to with earlier studies [1, 30]. In this study, the higher filler content of flaked shape and larger size of alumina is another possible factor affecting the DTS values. Due to these reasons, the alumina agglomeration has occurred resulting in reduction of the stress distribution as reported earlier [1, 4]. For the Rely-X U200 group, the cracks tend to deviate between weak matrix-filler interfaces, leading to lower DTS than experimental groups. This could be due to different diluent and characteristics of hybrid fillers of Rely-X U200 that increases stress concentration within resin matrix interface [26, 29, 31]. These findings give an idea that the hybrid silica and alumina reinforced dental dement may potentially act as an alternative to commercial products. Furthermore, improving film thickness and diametral tensile strength gives an advantage to long-term clinical success to support dental prosthesis during cementation and withstand the mechanical forces, respectively.

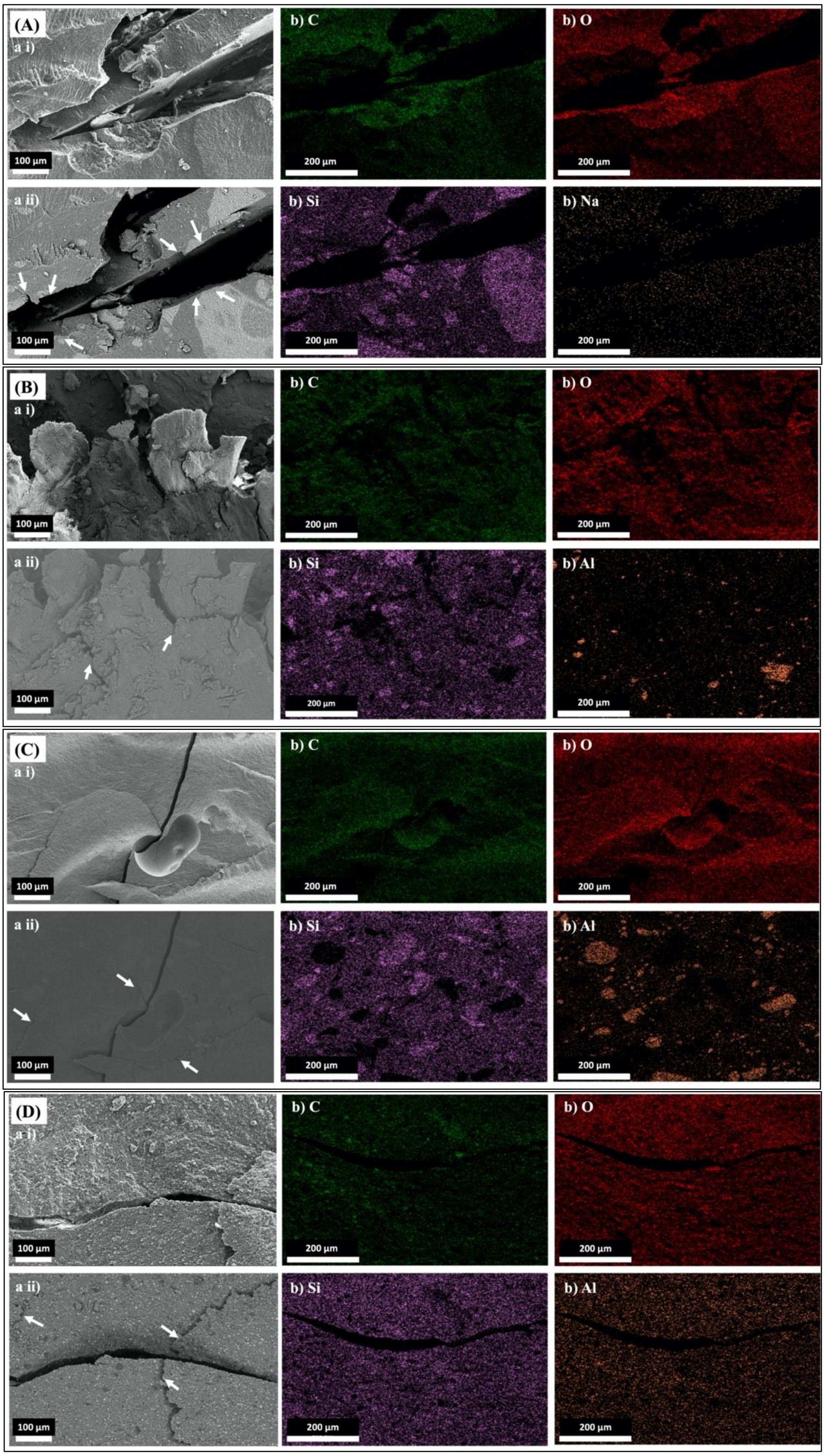

Figure 4. FESEM micrographs on magnification of 150x: a i) SE and a ii) BSE; and b) EDX mapping (C: Carbon, O: Oxygen, Si: Silicon, Na: Sodium and Al: Aluminium) of fractured (A) CTR, (B) 3 wt % alumina, (C) 10 wt% alumina and (D) Rely-X U200 dental cements after diametral tensile strength test

Conclusion

Alumina filler increases the film thickness and the diametral tensile strength compared to that of experimental dental cement. The treated alumina filler supports the force distribution within the dental cement before fracture diametral tensile strength. However, the optimum alumina filler content mixture is crucial to minimise agglomeration that could affect the physical and mechanical properties.

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Author Contributions

All authors contributed toward data analysis, drafting and critically revising the paper and agree to be accountable for all aspects of the work.

Disclosure of Conflict of Interest

The authors have no disclosures to declare

Compliance with Ethical Standards

The work is compliant with ethical standards

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