EVALUATION OF MECHANICAL PROPERTIES OF PROVISIONAL FIXED PARTIAL DENTURE PMMA MATERIAL CONTAINING ALUMINA NANOFIBERS

by

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DEDICATION

To my beloved wife, Maram T. Alghabbashi,
and my angels, Lana and Layan.

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TABLE OF CONTENTS

TABLES AND FIGURES

INTRODUCTION

Provisional restorations are an essential part of fixed prosthodontic treatment. They are designed to enhance the esthetics, the stability, and the function of teeth for a limited period of time, after which the provisional restorations are replaced by definitive restorations.[1,2] While CAD-CAM technology now allows the delivery of a simple dental prosthesis in one visit,[3,4] most prostheses are still made in the dental laboratory. In such a case, the patient must be provided with a provisional restoration from the time of initial tooth preparation until the definitive prosthesis is delivered. The provisional item is also used to enhance the effectiveness of a treatment plan or the form and function of the intended definitive prostheses.[1,2]

The terms provisional, interim, and transitional have been used interchangeably in the literature.[2,5] However, it is controversial and inappropriate to use the term temporary,[6,7] because provisional restorations serve many functions. The term "temporary treatment" may be interpreted as treatment of lesser importance or value. Provisional restorations should be the same as definitive restorations in all aspects, except for the material from which they are fabricated.[5,6]

There are several functions of provisional restorations. A properly constructed provisional restoration should protect the pulp of prepared teeth, protect the teeth from dental caries, maintain periodontal health, provide a method for immediate replacement of missing teeth, stabilize mobile teeth during periodontal treatment, and provide positional stability of the prepared teeth in relation to adjacent and opposing teeth. It could also serve as a trial prosthesis for evaluation of esthetics, phonetics, and patient acceptance for the final prostheses.[6]

Extensive prosthodontic treatment often requires fabrication of long-term provisional restorations. Provisional restorations in full-mouth rehabilitation cases act as diagnostic tools for functional and esthetic try-in. These cases almost always involve changes in vertical dimension of occlusion and anterior guidance.[7] Also, abutment teeth may require adjunctive periodontal, orthodontic, or endodontic treatment, which require an extended period of provisional restoration.[8] Long-term provisional fixed restorations are also indicated for patients undergoing implant therapy when teeth adjacent to the edentulous area are restored with complete crowns. In these cases, fixed provisional restorations prevent loading of submerged implants or tissue grafts during the healing phase, which is likely to occur with transitional removable partial dentures.[9] Other indications that may require stronger provisional restorations include long-span edentulous areas, above-average muscle strength as seen with bruxism, or a history of frequent breakage.[10]

REQUIREMENTS OF PROVISIONAL RESTORATION

The requirements of provisional restorations can be divided into biological, biomechanical, and esthetic requirements.[5,11,12]

BIOLOGICAL REQUIREMENTS

Provisional restorations should be biocompatible and non-irritating to the pulp and periodontal tissues, and these restorations should have good marginal integrity and adaptation to protect the prepared teeth against bacterial microleakage, and chemical and thermal irritation.[10] It should have proper proximal contour for maintaining the interdental papillae and promoting periodontal health.[13] In endodontically treated teeth, interim restorations help to maintain the biologic seal and prevent coronal leakage.[14] They should have low exothermicity, pleasant odor and taste, and a low incidence of localized allergic reactions. They should also provide high polishability to maintain gingival health, conducive to routine oral home-care cleaning procedures.[1,6,15,16]

BIOMECHANICAL REQUIREMENTS

Fixed provisional restorations must be strong, durable and hard,[16] wear resistant,[11] and able to withstand the functional forces of mastication without fracture or displacement.[5] Materials used should be easy to mix and load in a matrix, fabricate, adjust, reline, and repair, be nonporous and dimensionally stable, easy to remove and re-cement by the dentist, and relatively inexpensive.[15,17]

ESTHETIC REQUIREMENTS

The appearance of an anterior provisional restoration usually has higher esthetic demands than those for the posterior region.[18] For esthetic clinical purposes, appropriate emergence profile, contour and pontic design,[13] initial accurate color shade match, color stability, and stain resistance are very important factors over the course of provisionalization.[1,5] Color changes are influenced by surface quality and porosity in conjunction with the oral hygiene habits of the patient.[5,19]

There have been several problems of using poly (methyl methacrylate) acrylic resin for fabrication of provisional restorations. Despite its popularity, it has not fulfilled all the requirements in terms of optimal mechanical properties. Difficulties may be expected with extensive prosthodontic treatment where provisional restorations must be used for longer time or with long-span provisional bridges. Crack propagation may occur with these materials because of insufficient long-term flexural strength or fatigue resistance.[20] There is a tendency for occlusal wear and fracture failure that leads to unnecessary repair.[21] Time and expense involved with fabrication of heat-polymerized or metal casting re-inforced provisional restorations make them less cost effective.[6]

The technique of reinforcing different polymers with alumina nanofibers has been reported in the literature.[22-24] Therefore, it is possible to use this technique to reinforce the resin for a provisional prosthesis. Dispersion homogeneity has been identified as a key aspect of successful nanocomposite preparation.[25]

PURPOSE OF THE STUDY

The objective of this investigation was to study the effects of alumina nanofibers reinforcement on the mechanical properties of a commercially available provisional fixed partial denture PMMA material.

The ultimate goal of the study is to find the optimal combination of alumina nanofibers and dispersant to reinforce provisional PMMA resin.

NULL HYPOTHESIS (H_0)

The null hypothesis was that the incorporation of alumina nanofibers (in 2.5 percent or less by weight) does not alter the flexural strength, fracture toughness, and microhardness of provisional PMMA resin.

ALTERNATIVE HYPOTHESIS (H_A)

The alternative hypothesis was that the incorporation of alumina nanofibers into a PMMA matrix will significantly improve the flexural strength, fracture toughness, and microhardness of the provisional PMMA resin.

REVIEW OF LITERATURE

Custom fabrication of provisional restorations provides close contact between a provisional restoration and the prepared tooth while allowing the material to be contoured and shaded to meet various anatomic, esthetic, or occlusal needs.[6] A commonly used material for custom fabrication of fixed partial denture provisional restorations is an acrylate-based resin such as poly(methyl methacrylate).[6] The material has several advantages including adequate strength and good esthetics.[1,26] It is capable of obtaining a high polish, easy to add to, and relatively inexpensive.[27] The disadvantages of this material are insufficient long-term mechanical properties[20] and poor wear resistance.[26] Also, when used in a direct technique, it produces high exothermic reaction; excess monomer that leads to pulpal irritation; strong odor, and volumetric shrinkage during polymerization[1,26] (Table I).

Long-term strength of the material is another concern. Physical properties of strength, density, and hardness may predict the longevity of provisional restorations.[6] One classical method to evaluate the material's ability to withstand functional loads is to evaluate the material's flexural strength, a critical physical property in the scenario of the failure of a three-unit provisional bridge. Fracture is normally initiated from the side of the specimen where the material is subjected to tensile stress.[28]

Fracture toughness is a mechanical property that describes the resistance of brittle material to the catastrophic propagation of flaws under an applied stress. It is considered an appropriate parameter for predicting the clinical performance of dental materials.[29] Most resins used for provisional restorations are brittle.[30] Crack propagation and fracture failure may occur with these materials because of inadequate transverse strength or fatigue resistance.[20] Failure often occurs suddenly, and repairing or replacing failed provisional restorations is a concern for both the clinician and the patient because of the additional cost and time associated with these complications.[31]

Hardness is the resistance of a material to plastic deformation under indentation load.[28] It is also indicative of the ease of finishing of a material and its resistance to in-service scratches.[32] It may also be used to predict the wear resistance of a material and its ability to abrade opposing dental structures.[28]

To deal with this long-term mechanical property issue, fibers have been used extensively to reinforce provisional restorations, including glass,[33,34] polyethylene,[33,35] or carbon-graphite.[36,37] The effectiveness of fiber reinforcement is dependent on the quantity of fibers in the resin matrix,[38,39] fiber length,[39] the form of the fibers,[40] the orientation of the fibers,[41] the adhesion of fibers to the polymer matrix,[42] and the degree of impregnation of fibers with the resin.[43]

Hamza et al.[33] compared the effects of six different types of fibers (Construct, Fibrestick, Ribbond normal, Ribbond THM, Ribbond triaxial, and Fibrenet) on the fracture toughness and flexural strength of different provisional resins (Jet, Trim, and Temphase). Fibrestick (glass fibers) and Construct (polyethylene fibers) reinforcements showed a significant increase in mean fracture toughness over unreinforced controls for all resins tested. He concluded the use of glass and polyethylene fibers may be an effective way to reinforce resins used to fabricate fixed provisional restorations.

Vallittu compared the fracture resistance of provisional three-unit fixed partial dentures made from methacrylate resins that were reinforced either with one, two, or three unidirectional glass fiber reinforcements and one woven glass fiber reinforcement.

He showed that unidirectional and woven glass fibers significantly improved fracture resistance and prevented catastrophic failure.[34]

Samadzadah et al. evaluated fracture strength of different provisional restorative materials after adding plasma-treated polyethylene fibers. Polyethylene fibers increased the fracture strength for the bis-acryl material, but not for PMMA prostheses. In both types of resin, the incidence of catastrophic failures has been reduced with the incorporation of polyethylene fibers.[35]

Larson et al. compared elastic moduli of three different provisional resins (Jet, Splintline and Trim) after the addition of carbon graphite fibers. These fibers were supplied in braided strands that were cut into specified lengths and soaked in the appropriate monomer before placing them between two poured layers of resin in the mold compartments. For all the resins, the modulus of elasticity increased significantly with fiber incorporation, and water storage over time did not have a significant effect on the modulus of elasticity of the three resins tested.[36]

Yazdanie and Mahood evaluated the flexural strength of acrylic resin reinforced with two types of carbon fibers; strands and woven mats. They found that carbon fiber-acrylic resin composites were stronger and stiffer than unfilled controls.[37]

Solnit tested the flexural strength of PMMA resin after the addition of silane-treated and untreated glass fibers. He found that samples with untreated fibers tested weaker than unreinforced controls. The samples with treated fibers tested stronger, but the difference was not statistically significant.[44]

The use of short glass fibers (3 mm in length) to reinforce laboratory composites produced higher compressive and flexural strength when compared with unreinforced controls.[45] Carbon-graphite fibers are strong but may have limiting esthetic properties.[36] Greets et al. showed glass fibers and stainless steel wire reinforcements produced significantly higher fracture toughness for PMMA, but polyethylene fibers did not.[46]

Diaz-Arnold et al. evaluated the microhardness of three bisacryl resin composites (Integrity, Protemp Garant, Temphase) and two methyl methacrylate acrylic resins (Jet, Temporary Bridge). Samples were tested after storage in artificial saliva for 24 hours and 14 days. They found that hardness of most materials (Integrity, Protemp Garant, Jet) decreased over time. All of the bis-acryl resin composite materials exhibited superior microhardness over traditional methyl methacrylate (Jet, Temporary Bridge) resins.[47]

Hernandez et al. compared the hardness of four methyl methacrylate-based resins (Jet, Acralon, titanium dioxide filled PMMA and IPN) by using various processing techniques and conditions. He concluded that Acralon (heat cure) has an advantage for long-term fixed provisional restorations and showed higher microhardness than Jet acrylic.[21]

There are challenges in using these traditional fibers. Difficulty in incorporating a high concentration of fibers into the resin matrix, mechanical irritation from fiber protrusion, lateral spread of fibers under compression, and difficult handling are drawbacks of using long fibers.[48] The use of dark carbon-graphite fibers caused esthetic problems.[36]

Although it has been reported that polymer-fiber composites have good mechanical properties, none of these composites are commonly used in prosthodontics because of these complications.[48] With the limitations seen in traditional fibers, researchers are exploring the use of nanofibers. A few studies showed that nanofibers have the potential to provide reinforcement without the drawbacks seen in the use of long fibers.[49,50] The challenge of using nanofibers lies in the dispersion of the nanofibers. Dispersion is the process of separating bundles or agglomerates into individual particles within a matrix. Dispersion homogeneity has been identified as a key aspect of successful nanocomposite preparation. Enhanced dispersion improves mechanical properties by allowing more polymer-filler interactions and greater polymer chain restriction. At the same time, the presence of fiber agglomerates acts as a structural defect that detrimentally affects the mechanical properties.[25,50]

Several dispersion techniques have been reported in the literature. Brown and Ellyin mixed acidic deionized water (pH = 4) and silane agent with alumina nanofibers in a magnetic stirrer to aid in surface modification of the fibers, and then they used ultrasonic energy to achieve proper dispersion of the nanofibers in epoxy resin.[23] Wang et al. used dispersant and ultrasonic vibration to disperse short carbon fibers into cement-based composite.[51] Ultrasonication and high shear mixing have been used as mechanical methods to separate carbon nanotubes from each other and reduce their tendency to agglomerate.[52] Chemical methods such as the use of solvents or surfactants have been used to obtain high-weight fraction of individually dispersed nanotubes.[52]

Despite preliminary success through the use of several dispersion techniques, including the use of solvent, an important classical fiber dispersion technique, the use of bipolar dispersant has not been explored with provisional resin. Dispersant aids in fiber dispersion through breaking the van der Waals forces between the fibers. Dispersant also might get absorbed into the surface of the fibers to create repellent surface charges to break fiber agglomerates (electrostatic repulsion).[53] Fibers need to be dispersed to achieve proper reinforcement.

MATERIALS AND METHODS

The fixed partial denture provisional resin used was Jet Tooth Shade (Lang Dental, Wheeling, IL) (Figure 1). Jet was chosen because it is a commonly used resin to fabricate provisional restorations, has adequate mechanical properties, and is relatively inexpensive.[6]

Alumina nanofibers (Argonide Cooperation, Sanford, FL) are 2 nm in diameter and 200 nm to 300 nm in length. Alumina nanofibers were chosen because they have high mechanical properties. These nanofibers have high surface area (500-700 m^2/g) with an aspect ratio of 100 to 150, which permits significant polymer-filler interaction and improves mechanical properties of the polymer at low particle loading.[23] Also, to the best of our knowledge, they have never been used to reinforce PMMA resin before. They should provide no esthetic complication and are available at reasonable cost.

A quaternary ammonium acetate dispersant (CC-59, Goldschmidt, Janesville, WI) was added to the acrylic monomer to aid in fiber dispersion (Figure 2). A quaternary ammonium acetate was chosen as the dispersant based on a study by Chu et al.[54] They used a similar dispersant to disperse alumina powder in acrylates.

SPECIMEN PREPARATION

The specimens were fabricated using the aforementioned resin, Jet (Lang Dental, Wheeling, IL, USA), following manufacturer’s instructions with a powder: liquid ratio of 2:1. This ratio proved through trial to provide the consistency necessary to incorporate the highest percentage of fibers (2.5 percent) and as much polymer powder as possible.

The powder and liquid were weighed using an electronic balance (model SC-2000, Ainsworth Co, Denver, CO). A quaternary ammonium acetate (CC-59, Goldschmidt, Janesville, WI) dispersant was added to the acrylic monomer at 0.0 percent, 1.0 percent, 2.0 percent and 5.0 percent of nanofibers weight (Table II).

Alumina nanofibers were added as received from the manufacturer (Argonide Cooperation, Sanford, FL) at 0.0 percent, 0.5 percent, 1.0 percent, and 2.5 percent, of total material weight, to the solution of a quaternary ammonium acetate (CC-59, Goldschmidt, Janesville, WI) dispersant and methyl-methacrylate monomer in a glass beaker. A total of 13 groups were tested (1 control, 12 test group) as shown in Table II. Each fiber-dispersant combination was assigned a group name. The group names will be used in the result and discussion sections in this thesis. The goal of using the dispersant was to aid in the dispersion of nanofibers. Therefore, the use of dispersant without fibers will be irrelevant to the actual application of this project, and the dispersant was added as a percentage of the fiber present as stated above. The fiber concentration levels were chosen based on the study by Tian et al.[50] They investigated the reinforcement of Bis-GMA/TEGDMA dental resins with various mass fractions of nanofibrillar silicate.

A magnetic stirrer machine (Figure 3) was used to mix the solution (CC-59+ methyl methacrylate monomer + alumina nanofibers) for 24 hours. The mixture was allowed to air cool. The provisional resin was fabricated by adding the powder to the solution and the material was then mixed for 15 seconds to ensure the wetting of all powder particles. The mix was packed into a stainless steel mold and polymerized at room temperature of 22 ± 2 ºC for 10 minutes.

Flexural Strength Specimens

Specimens (2 x 2 x 25 mm) were made (13 groups of fiber-dispersant combinations, n = 24 for each group) using a split stainless steel mold (Figure 4, 5) between transparent Mylar sheets according to the ISO standard 10477 dimensional specifications. After complete polymerization of the resin, the specimens were separated from the mold, and flash was removed using a sharp scalpel. Specimens were examined visually for any void and defective specimens were discarded. Then, specimens were polished with SiC paper (600-grit) to achieve smooth edges. For each fiber-dispersant combination, 12 specimens were tested after storage in distilled water for 24 hours at 37 ± 1 ºC according to the ISO specification 10477 for chemically polymerized provisional restoration. Another 12 specimens were tested after storage in distilled water at 37 ± 1 ºC for 7 days to evaluate the effect of short-term storage in water on the mechanical properties.

Fracture Toughness Test Specimens

Specimens (2 x 5 x 25 mm) were prepared (13 groups of fiber-dispersant combinations, n = 24 for each group) using a split stainless steel mold (Figure 6, 7) between transparent Mylar sheets for the single-edge notch 3-point-bending test according to ISO 13586 specification. The depth of the precrack was 2.150 mm. After complete polymerization of the resin, the specimens were separated from the mold and flash was removed using a sharp scalpel. Specimens were examined visually for any void and defective specimens were discarded. Then, specimens were polished with SiC paper (600-grit) to achieve smooth edges. Twelve specimens were tested after storage in distilled water for 24 hours at 37 ± 1 ºC. Another 12 specimens were tested after storage in distilled water at 37 ± 1 ºC for 7 days.

Microhardness Test Specimens

Twenty-four specimens for each group of fiber-dispersant combinations were made within aluminum rings with dimensions of 10 mm in diameter and 1.5 mm in height (Figure 8, 9). Both planar surfaces of all specimens were polymerized against glass plates. Twelve specimens were tested after storage in distilled water for 24 hours at 37 ± 1 ºC. Another 12 specimens were tested after storage in distilled water at 37 ± 1 ºC for 7 days.

MECHANICAL TESTING

Flexural strength, flexural modulus, fracture toughness, and surface microhardness (Knoop test) for all 13 groups were measured using the testing methods listed below.

Flexural Strength Test

The flexural strength and flexural modulus were determined using the flexural strength test as specified by the ISO standard 10477. The tests were performed using a universal testing machine (Sintech Renew 1121, Instron Engineering Corp., Canton, MA) (Figure 10). A standard three-point bending jig (Figure 11) was attached to the machine and connected to a computer with a specifically designed program (Test-Works 3.0 MTS Systems Co., Eden Prairie, MN). This software controlled the testing machine and recorded the breakage load and beam deflection. Before each test, the specimen thickness and width were recorded with a digital micrometer (Mitutoyo Corporation, Japan), to measure the dimensions after polishing, and the measurements were stored in the computer. The specimens were then placed on the jig and the test carried out using a crosshead speed of 1mm/min and a span length of 20 mm.

The flexural strength (S) was calculated using the following formula:

$$S = 3FL / 2bd^2$$

where (S) Flexural strength in MPa,

(F) load at break or yield in N.

(L) span of specimen between supports = 20mm

(b) width of the specimen =2mm

(d) thickness of the specimen =2mm

The flexural modulus (E) = MPa, was calculated using the following formula:

$$E = F_1 L^3 / 4bd^3 D_1,$$

where (F_1) is the force at deflection,

(L) is the span of specimen between supports = 20 mm

(b) specimen width = 2 mm

(d) specimen thickness = 2mm

D_1 the deflection at linear region of load deflection curves.

Fracture Toughness (K_{IC}) Test

The fracture toughness was determined using the single-edge notch 3-point-bending test according to ISO 13586 specification. The test was performed using a universal testing machine (Sintech Renew 1121, Instron Engineering Corp., Canton, MA). Before each test, the specimen thickness and width were recorded with a digital micrometer (Mitutoyo Corporation, Japan) and stored in the computer. The specimens were placed on the jig (Figure 12) and the test carried out using a crosshead speed of 0.2 mm/min and a span length of 20 mm.

Fracture toughness was calculated from the following equation:

$$K_{IC} = f(a/w)(F/h\sqrt{w})$$

Where, K_{IC} Fracture toughness (MPa $m^{0.5}$)

f(a/w) Fracture geometry factor

$$6\alpha^{1/2} [1.99 – \alpha(1- \alpha)(2.15 – 3.93\alpha + 2.7\alpha^2)] / [(1+2\alpha)(1-\alpha)^{3/2}]$$

(F) Force at begin of crack propagation in N.

(a) Crack length = 2.150 mm

(h) Specimen thickness = 2 mm

(w) Specimen width = 5 mm.

Surface Microhardness (Knoop Test)

The PMMA nanocomposites were tested using Knoop microhardness testing (M-400 Hardness Tester, Computing Printer ACP-94, LECO, Knoop Diamond Indenter 860-538) (Figure 13). Five indentations, at least 1 mm apart, were made on the surface of each sample. The load of the indenter was set at 100 g and the indentation time at 15 s.

The resulting impression was observed under a microscope and the length of the long diagonal was measured optically at X20 magnification. This measurement was converted into a hardness number. The Knoop hardness number (KHN) is the ratio of the load applied to the area of the indentation calculated from the following formula:

$$KHN= L/l^2C_p,$$

Where, (L) is the load applied in Kgf,

(l) is the length of the long diagonal of the indentation in mm, and

(C_p) is a constant relating l to the projected area of the indentation. The units for KHN are kg/mm^2.

ENERGY DISPERSIVE SPECTROMETRY (EDS) ANALYSIS

Three fracture toughness sample groups were randomly selected for Energy Dispersive Spectrometry (EDS). Samples were polished and dried in a vacuum desiccator for 24 hours. After that, samples were sputter coated (Denton Vacuum Desk II, Moorestown, NJ) using gold target. The coat thickness was ~200 Å. The samples were mounted on aluminum stubs using double-sided carbon tape. One side of each sample was painted with carbon paint and allowed to dry for 1 hour. Sections outside of the support area were observed under low-vacuum scanning electron microscope (JEOL 5310LV, JEOL USA, Peabody, MA). One image of the sample was taken for EDS analysis at acceleration voltage of 10 kV, 128 pixels resolution and X500 magnification with aluminum being the element of interest.

STATISTICAL TESTS

Two-way analysis of variance (ANOVA) was used to test the effects of storage time and combinations of alumina nanofiber level and quaternary ammonium acetate dispersant level on the flexural strength, fracture toughness and microhardness of the provisional PMMA resin. Pair-wise comparisons among groups were performed using Tukey’s multiple comparisons procedure to control the overall significance level at 5 percent.

RESULTS

A total of 13 groups were tested in this study with two storage conditions, 1 day and 7 days. Descriptive statistics for all groups are available in Table III.

FLEXURAL STRENGTH

The mean flexural strength values for one-day storage conditions were as follows: for control group A, 73.48 MPa; test group B, from 70.00 MPa to 74.78 MPa; group C, from 67.51 MPa to 73.83 MPa, and for group D, from 66.44 MPa to 68.83 MPa.

The mean flexural strength values for seven-day storage conditions were: control group A, 76.84 MPa; test group B, from 71.11 MPa to 75.96 MPa; group C from 70.06 MPa to 74.3 MPa, and group D from 63.53 MPa to 65.46 MPa (Figure 13).

Statistically, the storage condition (1 day vs. 7 days) had no effect on the flexural strength of the provisional PMMA resin. There was no significant difference in flexural strength values within groups under the two storage conditions (Table VI).

Among groups in the one-day storage condition, statistically significant differences were observed between groups B3-D1 and B3-D3 (Table VII). In the seven-day storage condition, statistically significant differences were observed between groups B1-D1; B1-D2; B1-D3; B1-D4; B2-D3; B3-D1; B3-D2; B3-D3; B3-D4; B4-D2; B4-D3; C1-D1; C1-D2; C1-D3; C1-D4; C2-D1; C2-D2; C2-D3; C2-D4; C3-D2; and C3-D3 (Table VIII).

FLEXURAL MODULUS

The mean flexural modulus values for one-day storage conditions were: control group A, 2203.37 MPa; test group B, from 2136.47 MPa to 2263.42 MPa; group C, from 2178.67 MPa to 2200.03 MPa; and group D, from 2255.76 MPa to 2330.33 MPa.

The mean flexural modulus values for seven-day storage conditions were: control group A, 2059.29 MPa; test group B, from 2096.91 MPa to 2211.91 MPa; group C, 2137.22 MPa to 2202.41 MPa; and group D, from 2040.18 MPa to 2176.85 MPa (Figure 14).

Statistically, the storage condition (1 day vs. 7 days) had no effect on flexural modulus of the provisional PMMA resin except groups D2, D3, and D4. Flexural modulus of the aforementioned groups under one-day storage was significantly higher than their counterparts in the seven-day group (Table VI).

Between groups, in the one-day storage condition, statistically significant differences were observed between groups B2-D1 and B2-D2 (Table VII). In the seven-day storage condition, statistically significant differences were observed between groups B3-D3, C2-D3 and C4-D3 (Table VIII).

FRACTURE TOUGHNESS

The mean fracture toughness values for one-day storage conditions were: control group A, 1.66 MPa.m$^{0.5}$; test group B, from 1.49 MPa to 1.62 MPa.m$^{0.5;}$ group C from 1.31 MPa m$^{0.5}$ to 1.58 MPa.m$^{0.5}$, and group D, from 1.36 MPa. m$^{0.5}$ to 1.47 MPa.m$^{0.5}$.

The mean fracture toughness values for seven-day storage conditions were: control group A, 1.48 MPa.m$^{0.5}$; test group B, 1.47 MPa.m$^{0.5}$ to 1.58 MPa.m$^{0.5}$; group C from 1.39 $MPa.m^{0.5}$ to 1.55 $MPa.m^{0.5}$; and group D, from 1.34 $MPa.m^{0.5}$ to 1.48$MPa.m^{0.5}$ (Figure 15).

Statistically, the storage condition (1 day vs. 7 days) has no effect on fracture toughness of the provisional PMMA resin. There is no significant difference in fracture toughness values within groups in the two storage conditions (Table VI).

Among groups in the one-day storage condition, statistically significant differences were observed between groups A-C3; A-D3; A-D4; B1-C3; B1-D3; B1-D4; B2-C3; B3-C3; B3-D3; B3-D4; C1-C3, and C2-C3 (Table VII). Under the seven-day storage conditions, a statistically significant difference was observed only between groups B2-D3 (Table VIII).

KNOOP MICROHARDNESS

The mean Knoop microhardness values for one-day storage conditions were: control group A, 11.43 kg/mm^2; test group B, from 10.02 kg/mm^2 to 11.23 kg/mm^2; group C from 8.90 kg/mm^2 to 11.03 kg/mm^2, and group D, from 9.83 kg/mm^2 to 11.10 kg/mm^2.

The mean Knoop microhardness values for seven-day storage conditions were: control group A, 12.81 kg/mm^2; test group B range from 10.64 kg/mm^2 to 11.90 kg/mm^2; group C from 10.57 kg/mm^2 to 13.06 kg/mm^2, and group D, from 11.57 kg/mm^2 to 12.10 kg/mm^2 (Figure 16).

Statistically, the storage condition (1 day vs. 7 days) had a statistically significant effect on Knoop microhardness of the provisional PMMA resin except for groups B1, B4, and D4. There was a statistically significant difference in Knoop microhardness values between A, B2, B3, C1, C2, C3, C4, D1, D2, and D3 groups under the two-storage conditions (Table VI).

Among groups under the one-day storage conditions, statistically significant differences were observed between groups A-B1; A-B2; A-B3; A-C1; A-C4; A-D1; A-D2; A-D3; B1-C4; B4-C1; B4-C4; B4-D3; C2-C4; C3-C4; C4-D2; C4-D4, and D3-D4 (Table VII). Under the seven-day storage condition, statistically significant differences were observed between groups A-B1; A-B4; A-C4; B1-B3; B1-C3; B1-D1; B1-D3; B1-D4; B2-C3; B3-C4; B4-C3; C1-C3; C2-C3; C3-C4; C3-D2; C4-D1; C4-D3, and C4-D4 (Table VIII).

ENERGY DISPERSIVE SPECTROMETRY

A total of 36 elemental mapping images (EDS) were taken (3 images/group) using a low-vacuum SEM (JEOL 5310LV) with aluminum being the element of interest. We were constrained by the limitations of the instrument, because EDS maps are generally lower resolution than full SEM micrographs. At the current resolution (128 pixels), every image took 15 minutes to process. We were unable to take images at a higher resolution because it was impractical (more than 1 hour/ image). While the resolution used for mapping was much too low for discerning individual nanofibers, qualitative evaluation of the dispersion of the nanofibers was possible. Dispersion of alumina nanofibers was neither uniform within the same group, nor across different groups. Groups with a higher nanofiber weight percent showed a higher tendency to form more agglomerates (Figures 18-41).

TABLES AND FIGURES

TABLE I

Advantages and disadvantages of methyl methacrylate resin

Advantages	Disadvantages
1) Adequate strength.	1) Insufficient long term mechanical properties.
2) Good esthetics.	2) Poor wear resistance.
3) Obtain high polish.	3) High exothermic reaction
4) Easy to add too.	4) Pulpal irritation from excess monomer.
5) Relatively inexpensive.	5) Strong odor.
	6) High volumetric shrinkage.

TABLE II

Test groups with different weight percentage of nanofibers and dispersant

Dispersant Wt %	Fibers Wt %			
	0.0	0.5	1.0	2.5
0.0	A	B1	C1	D1
1.0	-	B2	C2	D2
2.0	-	B3	C3	D3
5.0	-	B4	C4	D4

TABLE III

Descriptive statistics for all groups

Group	Storage Condition	Flexural Modulus (MPa)		Flexural Strength (MPa)		Fracture Toughness (MPa.m$^{0.5}$)		Microhardness (kg/mm^2)	
		Mean	SD	Mean	SD	Mean	SD	Mean	SD
A	1 day	2203.37	145.32	73.48	5.27	1.66	0.12	11.43	1.39
	7 days	2059.29	77.04	68.83	2.59	1.48	0.10	12.81	0.85
B1	1 day	2226.54	87.97	73.53	4.30	1.62	0.10	10.15	0.43
	7 days	2131.72	108.62	75.96	8.44	1.52	0.12	10.64	0.70
B2	1 day	2136.47	134.3	70.00	4.67	1.55	0.19	10.02	0.48
	7 days	2096.91	71.05	71.11	3.21	1.58	0.13	11.73	0.84
B3	1 day	2263.42	124.96	74.78	4.91	1.61	0.06	10.06	0.56
	7 days	2211.91	103.17	75.38	7.03	1.53	0.09	11.90	0.84
B4	1 day	2186.37	56.66	72.61	3.25	1.49	0.29	11.23	0.74
	7 days	2121.73	90.63	71.45	5.27	1.47	0.11	11.43	0.77
C1	1 day	2190.85	107.41	71.84	3.41	1.58	0.08	9.90	0.56
	7 days	2154.21	110.47	73.04	5.93	1.43	0.11	11.57	0.59
C2	1 day	2187.7	131.66	73.53	4.64	1.57	0.09	10.42	0.70
	7 days	2201.58	78.62	74.28	4.64	1.43	0.16	11.74	0.84
C3	1 day	2178.76	58.93	67.51	3.46	1.31	0.24	11.03	1.17
	7 days	2137.22	102.12	71.83	6.73	1.39	0.25	13.06	1.03
C4	1 day	2200.03	87.28	73.83	7.67	1.51	0.08	8.90	0.72
	7 days	2217.77	159.95	70.06	4.02	1.55	0.10	10.57	0.43
D1	1 day	2330.33	121.32	67.09	4.29	1.47	0.13	10.05	0.43
	7 days	2176.85	50.05	65.46	2.73	1.48	0.08	12.08	1.24
D2	1 day	2309.27	84.73	68.62	3.34	1.47	0.21	10.18	0.55
	7 days	2099.42	112.67	64.00	3.49	1.41	0.18	11.57	1.01
D3	1 day	2255.76	53.2	66.44	6.76	1.36	0.22	9.83	0.90
	7 days	2040.18	113.35	63.53	2.90	1.34	0.13	11.90	0.65
D4	1 day	2263.16	95.05	68.83	2.59	1.36	0.11	11.10	1.08
	7 days	2098.32	102.63	64.58	4.25	1.39	0.06	12.10	0.84

TABLE IV

Summary of mean values of all groups

Test Group	Flexural Strength (MPa)		Flexural Modulus (MPa)		Fracture Toughness (MPa.m$^{0.5}$)		Knoop Microhardness (kg/mm^2)	
	1 Day	7 Days	1 Day	7 Days	1 Day	7 Days	1 Day	7 Days
A	73.48	76.84	2203.37	2059.29	1.66	1.48	11.43	12.81
B1	73.53	75.96	2226.54	2131.72	1.62	1.52	10.15	10.64
B2	70.00	71.11	2136.47	2096.91	1.55	1.58	10.02	11.73
B3	74.78	75.38	2263.42	2211.91	1.61	1.53	10.06	11.90
B4	72.61	71.45	2186.37	2121.73	1.49	1.47	11.23	11.43
C1	71.84	73.04	2190.85	2154.21	1.58	1.43	9.90	11.57
C2	73.53	74.3	2187.70	2202.41	1.57	1.43	10.42	11.74
C3	67.51	71.83	2178.76	2137.22	1.31	1.39	11.03	13.06
C4	73.83	70.06	2200.03	2217.77	1.51	1.55	8.90	10.57
D1	67.09	65.46	2330.33	2176.85	1.47	1.48	10.05	12.08
D2	68.62	64.00	2309.27	2099.42	1.47	1.41	10.18	11.57
D3	66.44	63.53	2255.76	2040.18	1.36	1.34	9.83	11.90
D4	68.83	64.58	2263.16	2098.32	1.36	1.39	11.10	12.10

TABLE V

ANOVA table

Effect	DF	Flexural Modulus		Flexural Strength		Fracture Toughness		Microhardness	
		F-value	P-value	F-value	P-value	F-value	P-value	F-value	P-value
Day	1	61.48	<.0001	3.07	0.0810	6.23	0.0132	241.31	<.0001
Group	12	3.61	<.0001	11.77	<.0001	7.41	<.0001	14.43	<.0001
Group Day	12	3.56	<.0001	2.18	0.0128	1.83	0.0437	3.00	0.0006

TABLE VI

Storage condition comparisons; 1 day vs. 7 days;

Group	Flexural Modulus		Flexural Strength		Fracture Toughness		Microhardness	
	Difference	P-value	Difference	P-value	Difference	P-value	Difference	P-value
Group A	144.08	0.1152	4.66	0.8204	0.17	0.4395	-1.38	0.0129
Group B1	94.83	0.8627	-2.43	1.0000	0.10	0.9951	-0.49	0.9995
Group B2	39.56	1.0000	-1.11	1.0000	-0.03	1.0000	-1.71	0.0002
Group B3	51.50	1.0000	-0.61	1.0000	0.08	0.9999	-1.84	<.0001
Group B4	64.64	1.0000	1.16	1.0000	0.01	1.0000	-0.21	1.0000
Group C1	36.63	1.0000	-1.20	1.0000	0.15	0.7158	-1.68	0.0003
Group C2	-13.88	1.0000	-0.76	1.0000	0.14	0.8388	-1.32	0.0241
Group C3	41.54	1.0000	-4.32	0.9060	-0.09	0.9996	-2.04	<.0001
Group C4	-17.74	1.0000	3.77	0.9779	-0.04	1.0000	-1.67	0.0003
Group D1	153.49	0.0592	1.63	1.0000	-0.02	1.0000	-2.02	<.0001
Group D2	209.85	0.0003	4.62	0.8326	0.06	1.0000	-1.39	0.0112
Group D3	215.58	0.0002	2.91	0.9994	0.02	1.0000	-2.06	<.0001
Group D4	164.84	0.0240	4.25	0.9188	-0.02	1.0000	-1.00	0.3452

TABLE VII

Difference among groups – 1 Day

Group	Flexural Modulus		Flexural Strength		Fracture Toughness		Microhardness	
	Difference	P-value	Difference	P-value	Difference	P-value	Difference	P-value
A-B1	-23.17	1.0000	-0.04	1.0000	0.03	1.0000	1.29	0.0345
A-B2	66.90	0.9978	3.48	0.9918	0.10	0.9940	1.42	0.0083
A-B3	-60.05	0.9996	-1.29	1.0000	0.05	1.0000	1.37	0.0136
A-B4	17.00	1.0000	0.88	1.0000	0.17	0.5033	0.21	1.0000
A-C1	12.52	1.0000	1.64	1.0000	0.07	1.0000	1.54	0.0020
A-C2	15.67	1.0000	-0.04	1.0000	0.09	0.9996	1.01	0.3300
A-C3	24.61	1.0000	5.98	0.3321	0.35	<.0001	0.41	1.0000
A-C4	3.34	1.0000	-0.34	1.0000	0.14	0.8099	2.53	<.0001
A-D1	-126.97	0.3130	6.39	0.2087	0.19	0.2364	1.38	0.0130
A-D2	-105.90	0.6935	4.87	0.7531	0.19	0.2557	1.25	0.0483
A-D3	-52.39	1.0000	7.04	0.0868	0.30	0.0005	1.60	0.0009
A-D4	-59.80	0.9996	4.66	0.8204	0.29	0.0007	0.33	1.0000
B1-B2	90.08	0.9132	3.53	0.9904	0.07	1.0000	0.13	1.0000
B1-B3	-36.88	1.0000	-1.25	1.0000	0.01	1.0000	0.09	1.0000
B1-B4	40.17	1.0000	0.92	1.0000	0.14	0.8797	-1.08	0.2075
B1-C1	35.70	1.0000	1.68	1.0000	0.04	1.0000	0.25	1.0000
B1-C2	38.84	1.0000	0.00	1.0000	0.05	1.0000	-0.28	1.0000
B1-C3	47.79	1.0000	6.02	0.3182	0.32	0.0001	-0.88	0.6246
B1-C4	26.51	1.0000	-0.30	1.0000	0.11	0.9867	1.25	0.0499
B1-D1	-103.79	0.7303	6.43	0.1984	0.16	0.6251	0.09	1.0000
B1-D2	-82.72	0.9639	4.91	0.7385	0.16	0.6518	-0.03	1.0000
B1-D3	-29.21	1.0000	7.08	0.0816	0.26	0.0052	0.31	1.0000
B1-D4	-36.62	1.0000	4.70	0.8078	0.26	0.0068	-0.95	0.4541
B2-B3	-126.95	0.3132	-4.78	0.7840	-0.06	1.0000	-0.04	1.0000
B2-B4	-49.90	1.0000	-2.61	0.9999	0.06	1.0000	-1.21	0.0708

(continued)

TABLE VII (cont.)

Difference among groups – 1 Day

Group	Flexural Modulus		Flexural Strength		Fracture Toughness		Microhardness	
	Difference	P-value	Difference	P-value	Difference	P-value	Difference	P-value
B2-C1	-54.38	0.9999	-1.84	1.0000	-0.03	1.0000	0.12	1.0000
B2-C2	-51.24	1.0000	-3.53	0.9904	-0.02	1.0000	-0.41	1.0000
B2-C3	-42.29	1.0000	2.49	1.0000	0.25	0.0158	-1.01	0.3267
B2-C4	-63.57	0.9990	-3.83	0.9735	0.04	1.0000	1.12	0.1568
B2-D1	-193.87	0.0016	2.91	0.9994	0.09	0.9995	-0.04	1.0000
B2-D2	-172.80	0.0121	1.38	1.0000	0.09	0.9997	-0.17	1.0000
B2-D3	-119.29	0.4434	3.56	0.9891	0.19	0.2290	0.18	1.0000
B2-D4	-126.70	0.3172	1.17	1.0000	0.19	0.2661	-1.08	0.2014
B3-B4	77.05	0.9844	2.17	1.0000	0.12	0.9620	-1.17	0.1036
B3-C1	72.57	0.9928	2.93	0.9994	0.02	1.0000	0.16	1.0000
B3-C2	75.72	0.9875	1.25	1.0000	0.04	1.0000	-0.36	1.0000
B3-C3	84.66	0.9535	7.27	0.0616	0.30	0.0003	-0.97	0.4183
B3-C4	63.39	0.9990	0.95	1.0000	0.10	0.9982	1.16	0.1104
B3-D1	-66.92	0.9977	7.68	0.0311	0.14	0.8014	0.00	1.0000
B3-D2	-45.85	1.0000	6.16	0.2734	0.14	0.8222	-0.12	1.0000
B3-D3	7.66	1.0000	8.33	0.0095	0.25	0.0135	0.22	1.0000
B3-D4	0.25	1.0000	5.95	0.3406	0.24	0.0172	-1.04	0.2717
B4-C1	-4.48	1.0000	0.77	1.0000	-0.10	0.9981	1.33	0.0221
B4-C2	-1.33	1.0000	-0.92	1.0000	-0.08	0.9999	0.80	0.7904
B4-C3	7.61	1.0000	5.10	0.6675	0.18	0.3393	0.20	1.0000
B4-C4	-96.04	0.8475	1.39	1.0000	-0.08	1.0000	0.86	0.6556
B4-D1	-55.12	0.9999	5.99	0.3265	-0.01	1.0000	-0.64	0.9734
B4-D2	22.31	1.0000	7.45	0.0460	0.06	1.0000	-0.14	1.0000
B4-D3	81.55	0.9693	7.92	0.0207	0.13	0.8928	-0.46	0.9998

(continued)

TABLE VII (cont.)

Difference among groups – 1 Day

Group	Flexural Modulus		Flexural Strength		Fracture Toughness		Microhardness	
	Difference	P-value	Difference	P-value	Difference	P-value	Difference	P-value
B4-D4	23.41	1.0000	6.88	0.1105	0.09	0.9995	-0.66	0.9624
C1-C2	-47.36	1.0000	-1.25	1.0000	0.00	1.0000	-0.17	1.0000
C1-C3	16.99	1.0000	1.21	1.0000	0.04	1.0000	-1.49	0.0036
C1-C4	-63.56	0.9990	2.98	0.9992	-0.12	0.9693	1.01	0.3367
C1-D1	-22.63	1.0000	7.58	0.0372	-0.05	1.0000	-0.50	0.9991
C1-D2	54.80	0.9999	9.04	0.0023	0.02	1.0000	0.00	1.0000
C1-D3	114.03	0.5417	9.50	0.0008	0.09	0.9990	-0.32	1.0000
C1-D4	55.89	0.9999	8.46	0.0074	0.05	1.0000	-0.52	0.9984
C2-C3	64.36	0.9988	2.46	1.0000	0.04	1.0000	-1.32	0.0250
C2-C4	-16.19	1.0000	4.23	0.9233	-0.12	0.9565	1.18	0.0965
C2-D1	24.73	1.0000	8.83	0.0036	-0.05	1.0000	-0.33	1.0000
C2-D2	102.16	0.7575	10.28	0.0001	0.02	1.0000	0.17	1.0000
C2-D3	161.40	0.0319	10.75	<.0001	0.09	0.9995	-0.15	1.0000
C2-D4	103.26	0.7393	9.71	0.0005	0.04	1.0000	-0.35	1.0000
C3-C4	-80.55	0.9733	1.77	1.0000	-0.16	0.6507	2.49	<.0001
C3-D1	-39.63	1.0000	6.37	0.2151	-0.09	0.9995	0.98	0.3854
C3-D2	37.81	1.0000	7.82	0.0243	-0.02	1.0000	1.49	0.0036
C3-D3	97.04	0.8343	8.29	0.0103	0.05	1.0000	1.17	0.1051
C3-D4	38.90	1.0000	7.25	0.0632	0.01	1.0000	0.96	0.4295
C4-D1	40.92	1.0000	4.60	0.8373	0.07	1.0000	-1.51	0.0027
C4-D2	118.35	0.4606	6.06	0.3046	0.14	0.8380	-1.00	0.3401
C4-D3	177.59	0.0078	6.53	0.1769	0.21	0.1074	-1.33	0.0225
C4-D4	119.45	0.4405	5.48	0.5157	0.16	0.5512	-1.53	0.0021
D1-D2	77.43	0.9834	1.46	1.0000	0.07	1.0000	0.51	0.9991
D1-D3	136.67	0.1841	1.92	1.0000	0.14	0.8294	0.18	1.0000

(continued)

TABLE VII (cont.)

Difference among groups – 1 Day

Group	Flexural Modulus		Flexural Strength		Fracture Toughness		Microhardness	
	Difference	P-value	Difference	P-value	Difference	P-value	Difference	P-value
D1-D4	78.53	0.9803	0.88	1.0000	0.10	0.9982	-0.02	1.0000
D2-D3	59.24	0.9997	0.47	1.0000	0.07	1.0000	-0.32	1.0000
D2-D4	1.10	1.0000	-0.57	1.0000	0.02	1.0000	-0.53	0.9983
D3-D4	-58.14	0.9998	-1.04	1.0000	-0.05	1.0000	-0.20	1.0000

TABLE VIII

Difference among groups – 7 Day

Group	**Flexural Modulus**		**Flexural Strength**		**Fracture Toughness**		**Microhardness**	
	Difference	P-value	Difference	P-value	Difference	P-value	Difference	P-value
A-B1	-72.43	0.9930	-7.13	0.0757	-0.04	1.0000	2.17	<.0001
A-B2	-37.62	1.0000	-2.28	1.0000	-0.10	0.9977	1.08	0.2014
A-B3	-152.63	0.0631	-6.56	0.1695	-0.05	1.0000	0.91	0.5419
A-B4	-62.45	0.9992	-2.63	0.9999	0.01	1.0000	1.38	0.0131
A-C1	-94.93	0.8615	-4.21	0.9256	0.05	1.0000	1.24	0.0553
A-C2	-142.29	0.1296	-5.46	0.5256	0.05	1.0000	1.07	0.2255
A-C3	-77.94	0.9820	-3.00	0.9991	0.09	0.9994	-0.25	1.0000
A-C4	-158.49	0.0533	-1.23	1.0000	-0.07	1.0000	2.24	<.0001
A-D1	-117.56	0.4752	3.37	0.9949	0.00	1.0000	0.73	0.9012
A-D2	-40.13	1.0000	4.82	0.7674	0.07	1.0000	1.24	0.0544
A-D3	19.11	1.0000	5.29	0.5922	0.14	0.8132	0.91	0.5379
A-D4	-39.03	1.0000	4.25	0.9188	0.10	0.9977	0.71	0.9239
B1-B2	34.81	1.0000	4.85	0.7589	-0.06	1.0000	-1.09	0.1883
B1-B3	-80.20	0.9747	0.58	1.0000	-0.01	1.0000	-1.26	0.0438
B1-B4	9.99	1.0000	4.51	0.8620	0.05	1.0000	-0.80	0.7983
B1-C1	-22.50	1.0000	2.92	0.9994	0.09	0.9994	-0.94	0.4850
B1-C2	-69.86	0.9958	1.68	1.0000	0.09	0.9989	-1.11	0.1671
B1-C3	-5.51	1.0000	4.13	0.9383	0.13	0.9314	-2.42	<.0001
B1-C4	-86.06	0.9448	5.90	0.3579	-0.03	1.0000	0.07	1.0000
B1-D1	-45.13	1.0000	10.50	0.0001	0.04	1.0000	-1.44	0.0062
B1-D2	32.30	1.0000	11.96	<.0001	0.11	0.9859	-0.94	0.4889
B1-D3	91.54	0.8993	12.43	<.0001	0.18	0.3478	-1.26	0.0445
B1-D4	33.40	1.0000	11.38	<.0001	0.14	0.8816	-1.46	0.0049
B2-B3	-115.00	0.5233	-4.27	0.9141	0.05	1.0000	-0.17	1.0000

(continued)

TABLE VIII (cont.)

Difference among groups – 7 Day

Group	Flexural Modulus		Flexural Strength		Fracture Toughness		Microhardness	
	Difference	P-value	Difference	P-value	Difference	P-value	Difference	P-value
B2-C1	-57.30	0.9998	-1.93	1.0000	0.15	0.7612	0.15	1.0000
B2-C2	-104.67	0.7152	-3.17	0.9978	0.15	0.7178	-0.02	1.0000
B2-C3	-40.31	1.0000	-0.72	1.0000	0.19	0.2876	-1.33	0.0211
B2-C4	-120.86	0.4151	1.05	1.0000	0.03	1.0000	1.16	0.1104
B2-D1	-79.94	0.9756	5.65	0.4503	0.10	0.9971	-0.35	1.0000
B2-D2	-2.51	1.0000	7.11	0.0786	0.17	0.4829	0.16	1.0000
B2-D3	56.73	0.9998	7.58	0.0374	0.24	0.0227	-0.17	1.0000
B2-D4	-1.41	1.0000	6.53	0.1750	0.19	0.2160	-0.37	1.0000
B3-B4	90.18	0.9123	3.93	0.9636	0.05	1.0000	0.46	0.9998
B3-C1	57.70	0.9998	2.35	1.0000	0.10	0.9982	0.32	1.0000
B3-C2	10.34	1.0000	1.10	1.0000	0.10	0.9967	0.15	1.0000
B3-C3	74.69	0.9895	3.56	0.9891	0.13	0.8867	-1.16	0.1066
B3-C4	-5.86	1.0000	5.32	0.5789	-0.02	1.0000	1.33	0.0221
B3-D1	35.07	1.0000	9.93	0.0003	0.05	1.0000	-0.18	1.0000
B3-D2	112.50	0.5709	11.38	<.0001	0.12	0.9705	0.32	1.0000
B3-D3	171.73	0.0133	11.85	<.0001	0.19	0.2734	0.00	1.0000
B3-D4	113.59	0.5500	10.81	<.0001	0.14	0.8206	-0.20	1.0000
B4-C1	-32.48	1.0000	-1.59	1.0000	0.04	1.0000	-0.14	1.0000
B4-C2	-79.85	0.9759	-2.83	0.9996	0.05	1.0000	-0.31	1.0000
B4-C3	-15.49	1.0000	-0.37	1.0000	0.08	0.9999	-1.63	0.0006
B4-C4	-96.04	0.8475	1.39	1.0000	-0.08	1.0000	0.86	0.6556
B4-D1	-55.12	0.9999	5.99	0.3265	-0.01	1.0000	-0.64	0.9734
B4-D2	22.31	1.0000	7.45	0.0460	0.06	1.0000	-0.14	1.0000
B4-D3	81.55	0.9693	7.92	0.0207	0.13	0.8928	-0.46	0.9998
B4-D4	23.41	1.0000	6.88	0.1105	0.09	0.9995	-0.66	0.9624

(continued)

TABLE VIII (cont.)

Difference among groups – 7 Day

Group	Flexural Modulus		Flexural Strength		Fracture Toughness		Microhardness	
	Difference	P-value	Difference	P-value	Difference	P-value	Difference	P-value
C1-C3	16.99	1.0000	1.21	1.0000	0.04	1.0000	-1.49	0.0036
C1-C4	-63.56	0.9990	2.98	0.9992	-0.12	0.9693	1.01	0.3367
C1-D1	-22.63	1.0000	7.58	0.0372	-0.05	1.0000	-0.50	0.9991
C1-D2	54.80	0.9999	9.04	0.0023	0.02	1.0000	0.00	1.0000
C1-D3	114.03	0.5417	9.50	0.0008	0.09	0.9990	-0.32	1.0000
C1-D4	55.89	0.9999	8.46	0.0074	0.05	1.0000	-0.52	0.9984
C2-C3	64.36	0.9988	2.46	1.0000	0.04	1.0000	-1.32	0.0250
C2-C4	-16.19	1.0000	4.23	0.9233	-0.12	0.9565	1.18	0.0965
C2-D1	24.73	1.0000	8.83	0.0036	-0.05	1.0000	-0.33	1.0000
C2-D2	102.16	0.7575	10.28	0.0001	0.02	1.0000	0.17	1.0000
C2-D3	161.40	0.0319	10.75	<.0001	0.09	0.9995	-0.15	1.0000
C2-D4	103.26	0.7393	9.71	0.0005	0.04	1.0000	-0.35	1.0000
C3-C4	-80.55	0.9733	1.77	1.0000	-0.16	0.6507	2.49	<.0001
C3-D1	-39.63	1.0000	6.37	0.2151	-0.09	0.9995	0.98	0.3854
C3-D2	37.81	1.0000	7.82	0.0243	-0.02	1.0000	1.49	0.0036
C3-D3	97.04	0.8343	8.29	0.0103	0.05	1.0000	1.17	0.1051
C3-D4	38.90	1.0000	7.25	0.0632	0.01	1.0000	0.96	0.4295
C4-D1	40.92	1.0000	4.60	0.8373	0.07	1.0000	-1.51	0.0027
C4-D2	118.35	0.4606	6.06	0.3046	0.14	0.8380	-1.00	0.3401
C4-D3	177.59	0.0078	6.53	0.1769	0.21	0.1074	-1.33	0.0225
C4-D4	119.45	0.4405	5.48	0.5157	0.16	0.5512	-1.53	0.0021
D1-D2	77.43	0.9834	1.46	1.0000	0.07	1.0000	0.51	0.9991
D1-D3	136.67	0.1841	1.92	1.0000	0.14	0.8294	0.18	1.0000
D1-D4	78.53	0.9803	0.88	1.0000	0.10	0.9982	-0.02	1.0000
D2-D3	59.24	0.9997	0.47	1.0000	0.07	1.0000	-0.32	1.0000
D2-D4	1.10	1.0000	-0.57	1.0000	0.02	1.0000	-0.53	0.9983
D3-D4	-58.14	0.9998	-1.04	1.0000	-0.05	1.0000	-0.20	1.0000

FIGURE 1. Jet Tooth Shade, powder and liquid.

FIGURE 2. Quaternary ammonium
acetate dispersant, CC-59.

FIGURE 3. Magnetic stirrer.

FIGURE 4. Stainless steel mold used to fabricate bar specimens for flexural strength test.

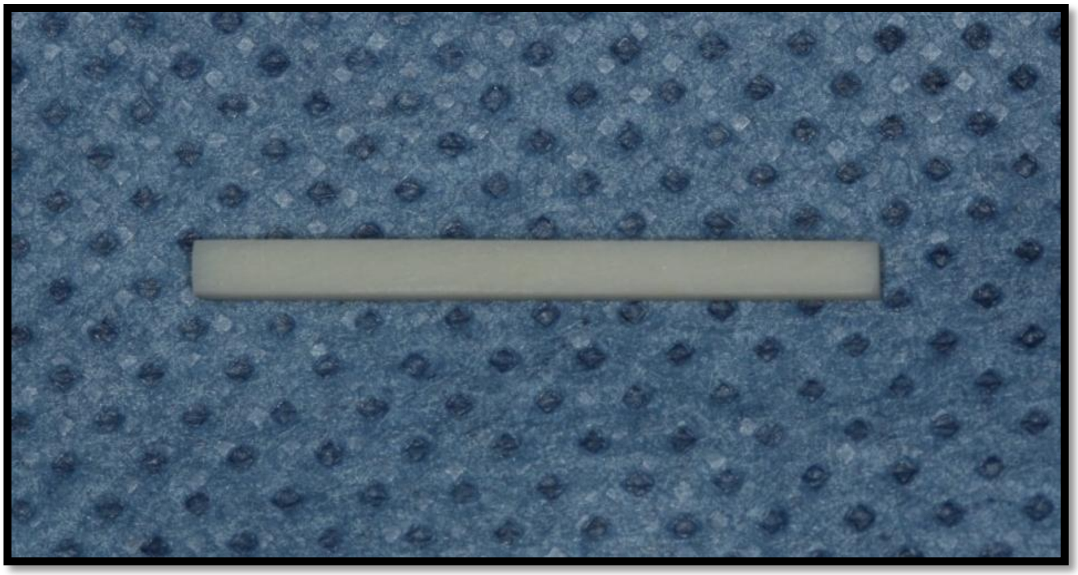

FIGURE 5. Flexural strength specimen.

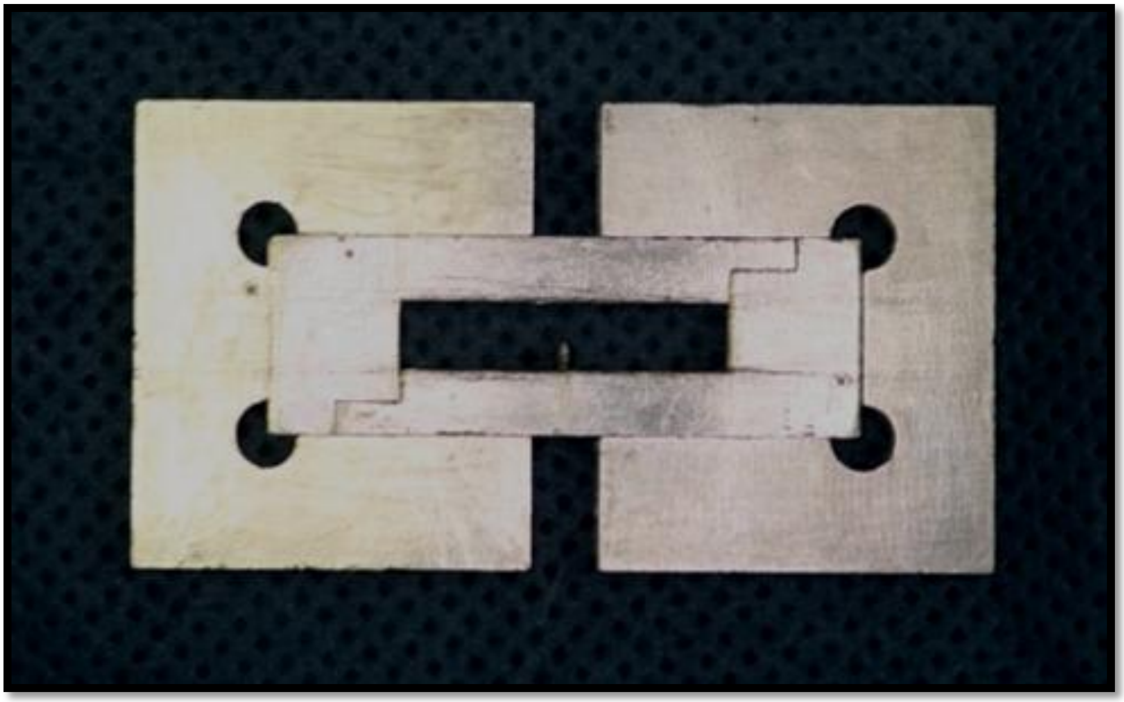

FIGURE 6. Stainless steel mold used to fabricate bar specimens for fracture toughness test.

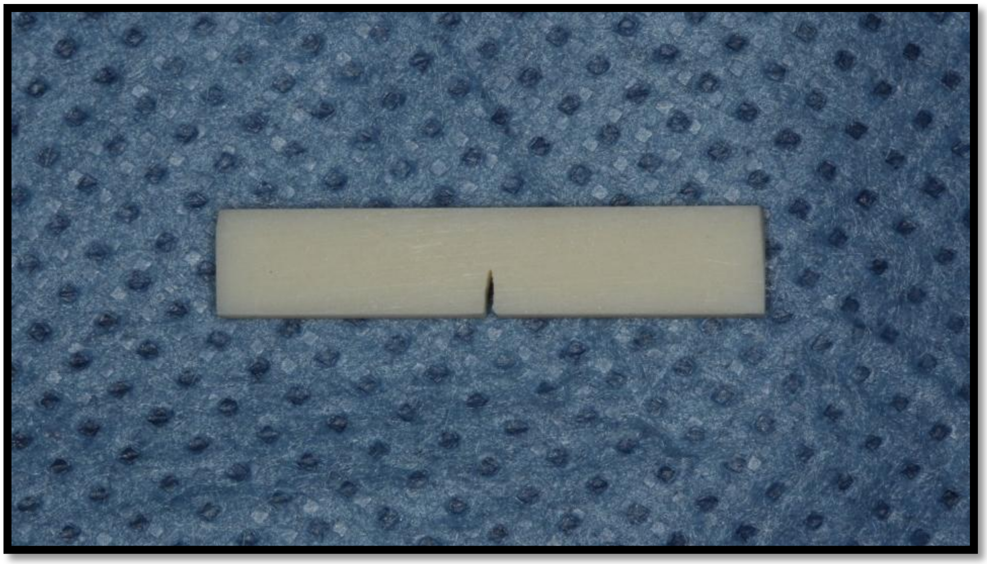

FIGURE 7. Fracture toughness specimen.

FIGURE 8. Metal ring used to fabricate specimens for Knoop microhardness test.

FIGURE 9. Microhardness specimen.

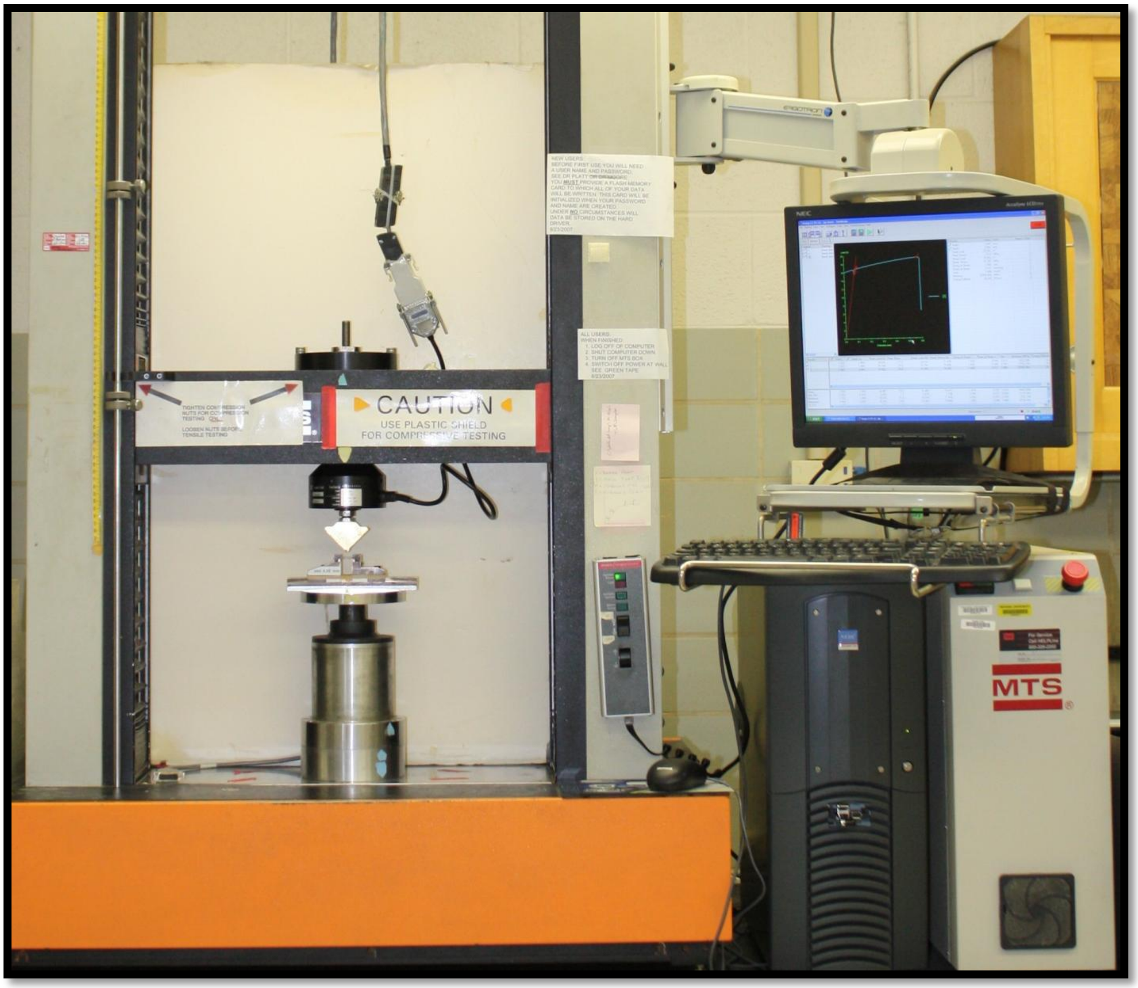

FIGURE 10. Universal testing machine.

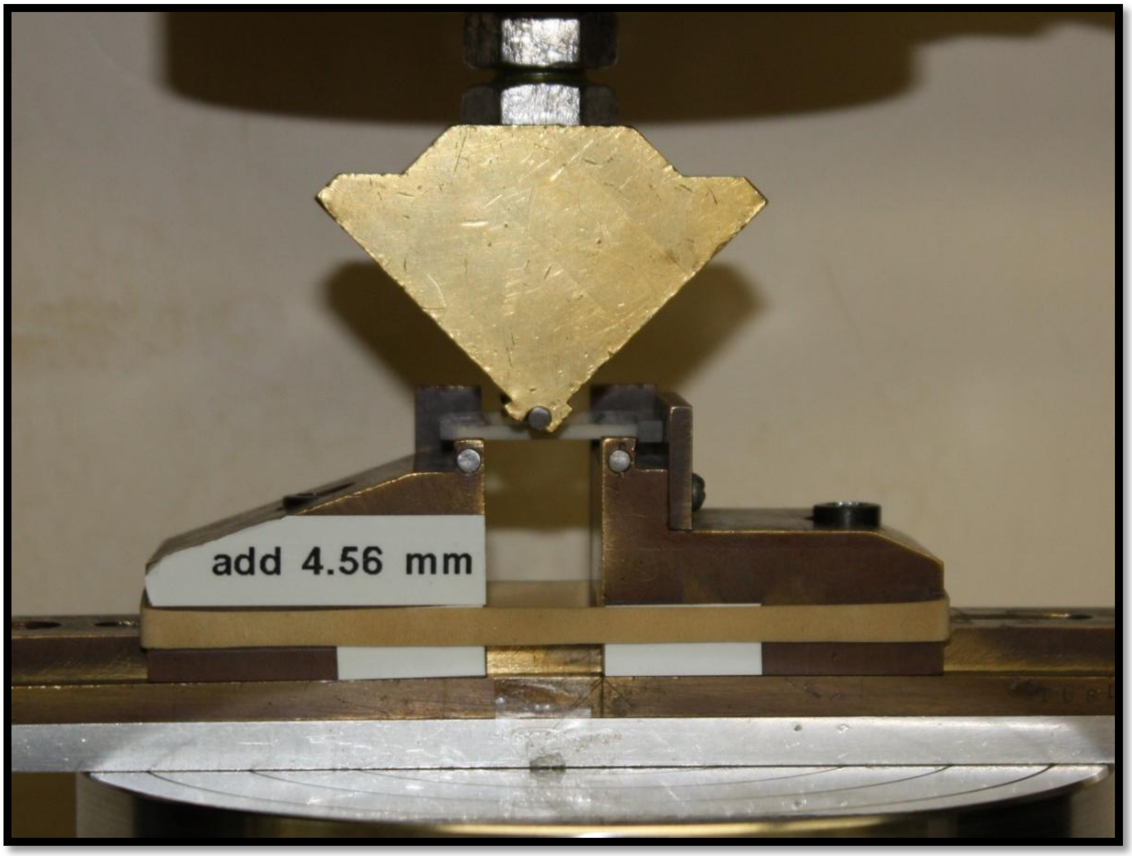

FIGURE 11. Flexural strength test.

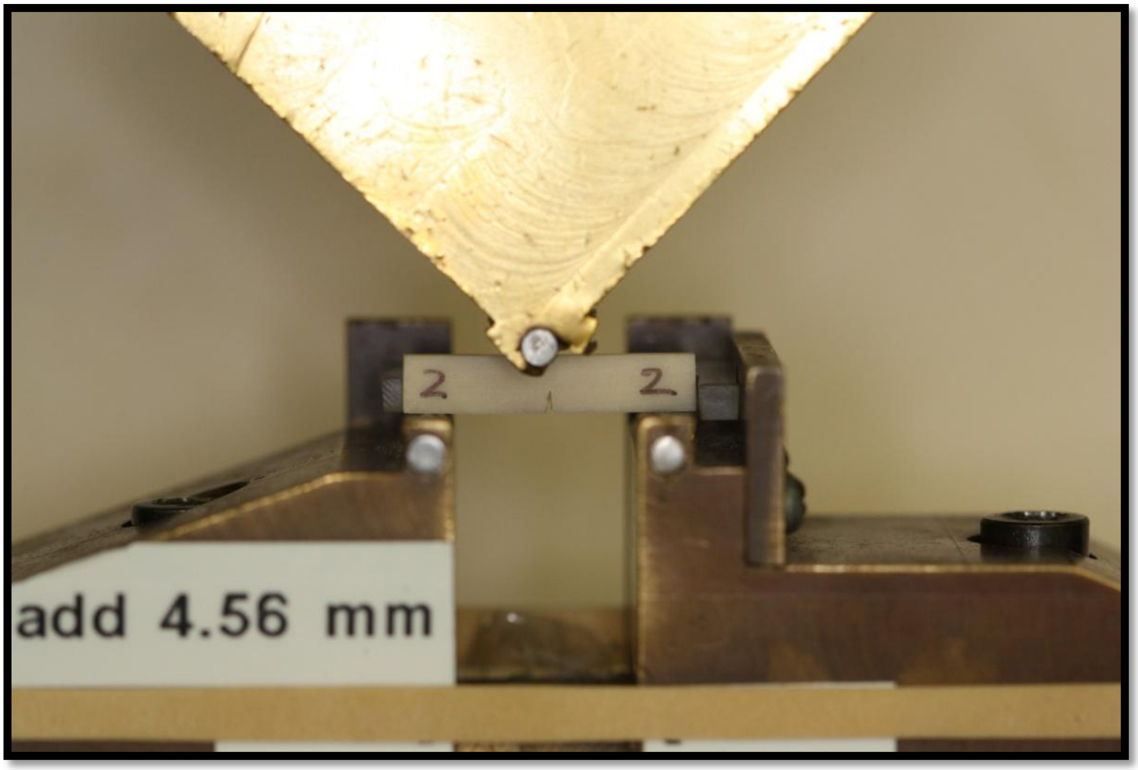

FIGURE 12. Fracture toughness test.

FIGURE 13. Knoop microhardness test.

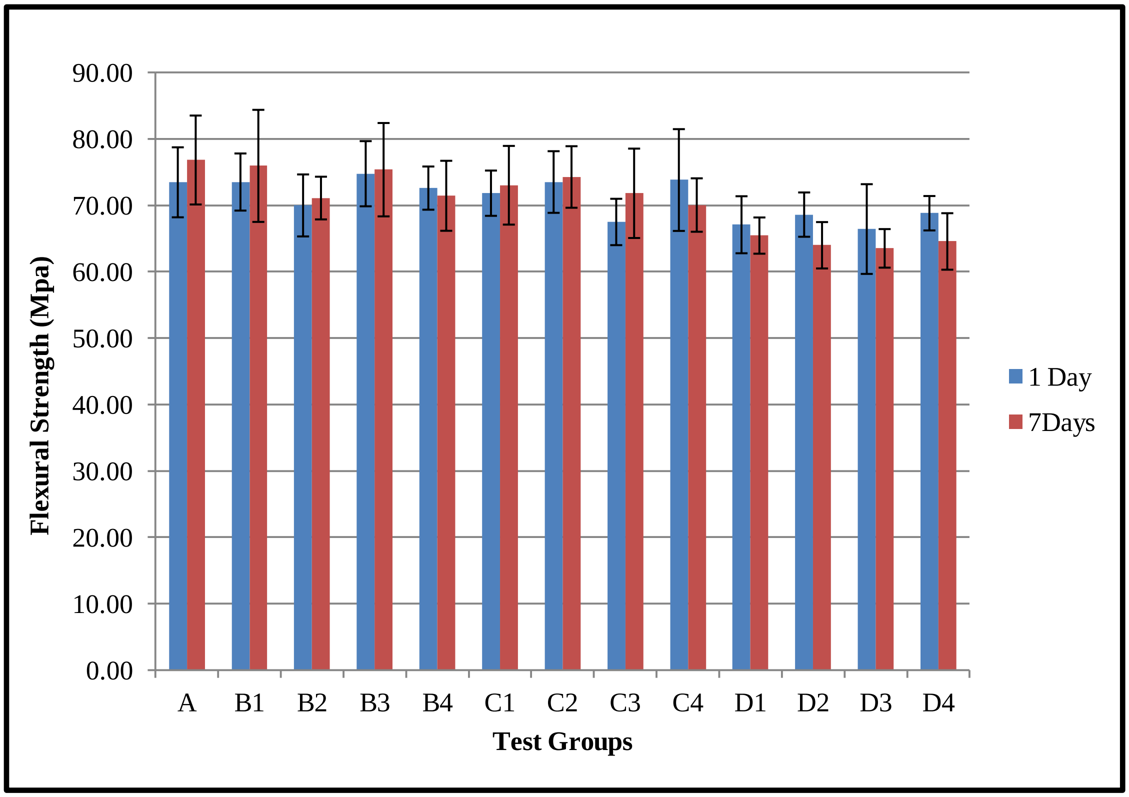

FIGURE 14. Mean values of flexural strength, comparison among groups.

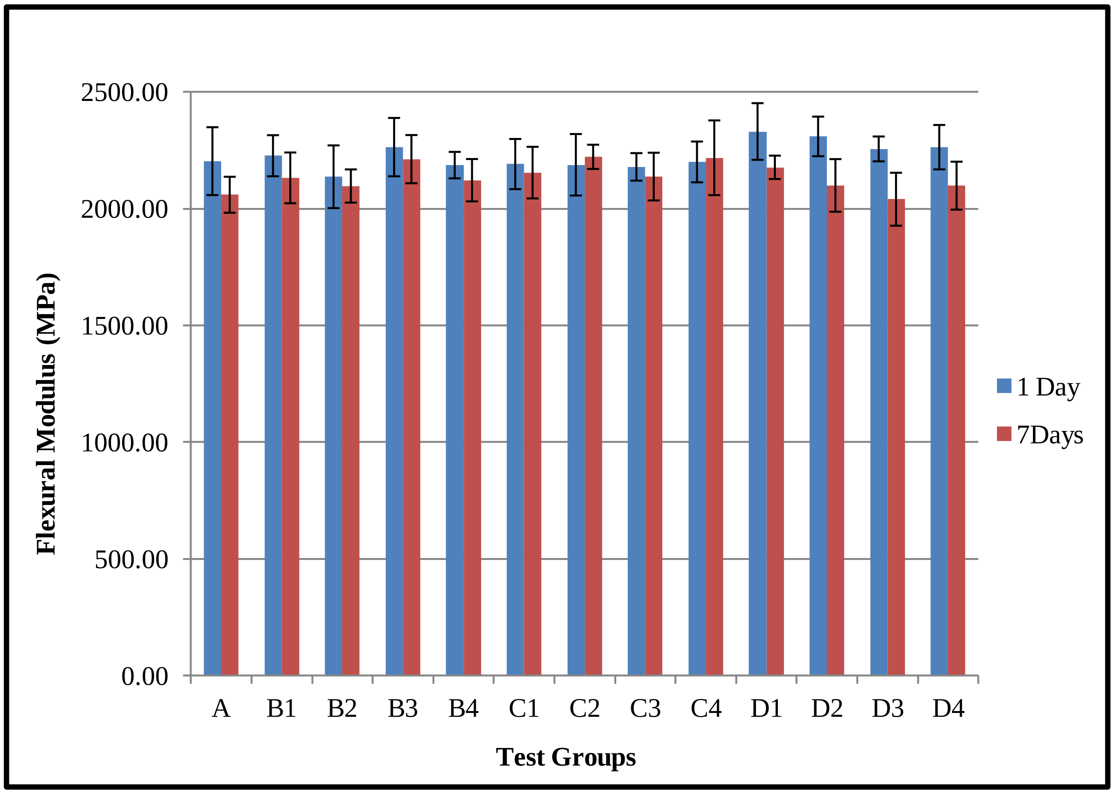

FIGURE 15. Mean values of flexural modulus, comparison among groups.

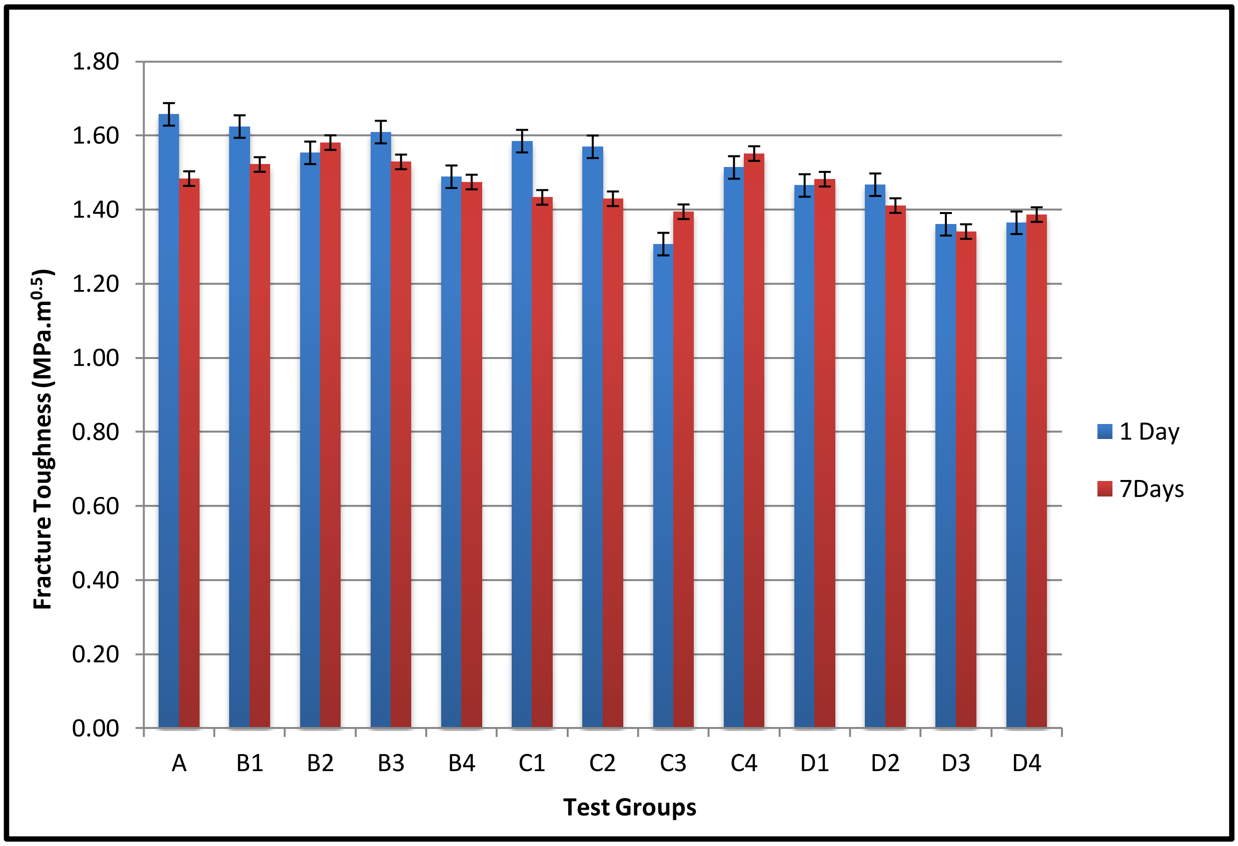

FIGURE 16. Mean values of fracture toughness; comparison among groups.

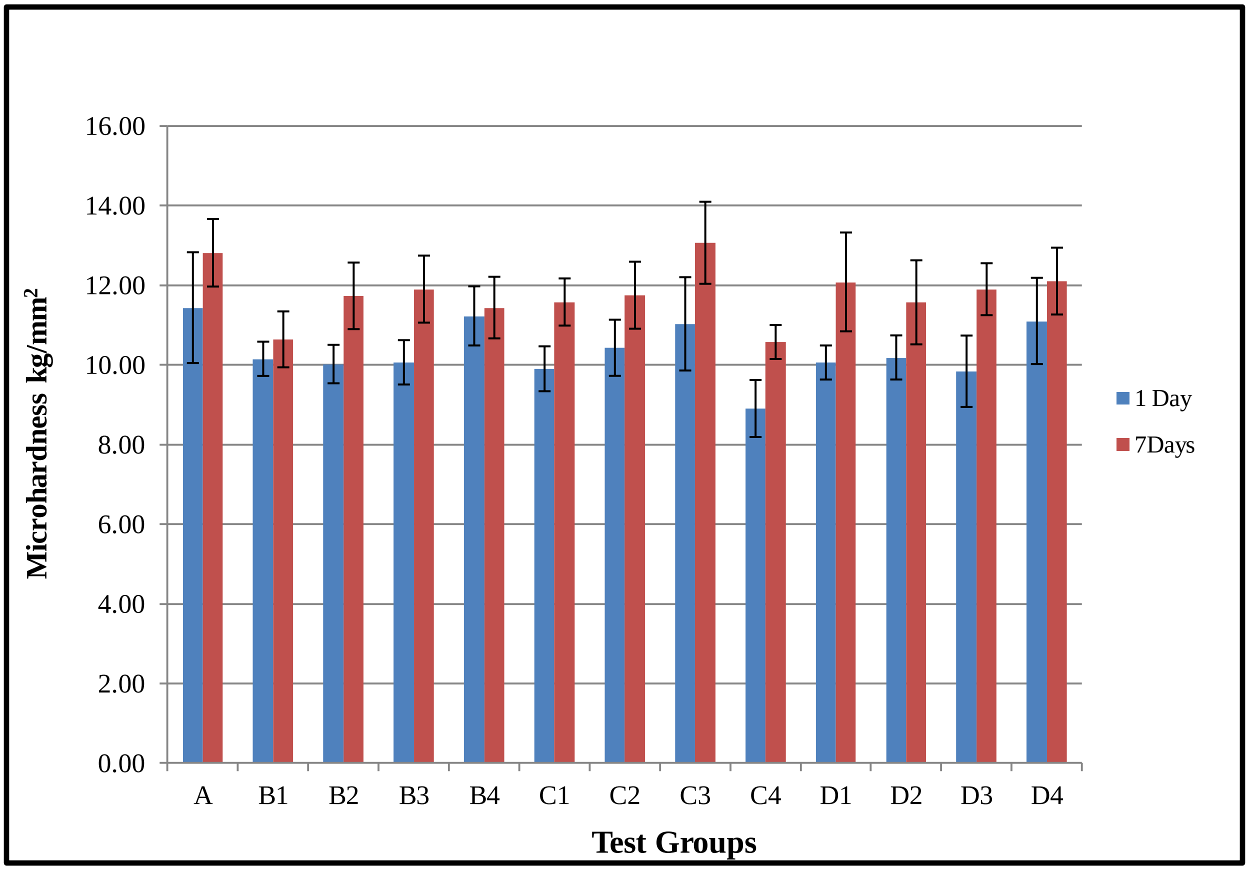

FIGURE 17. Mean values of Knoop microhardness; comparison among groups.

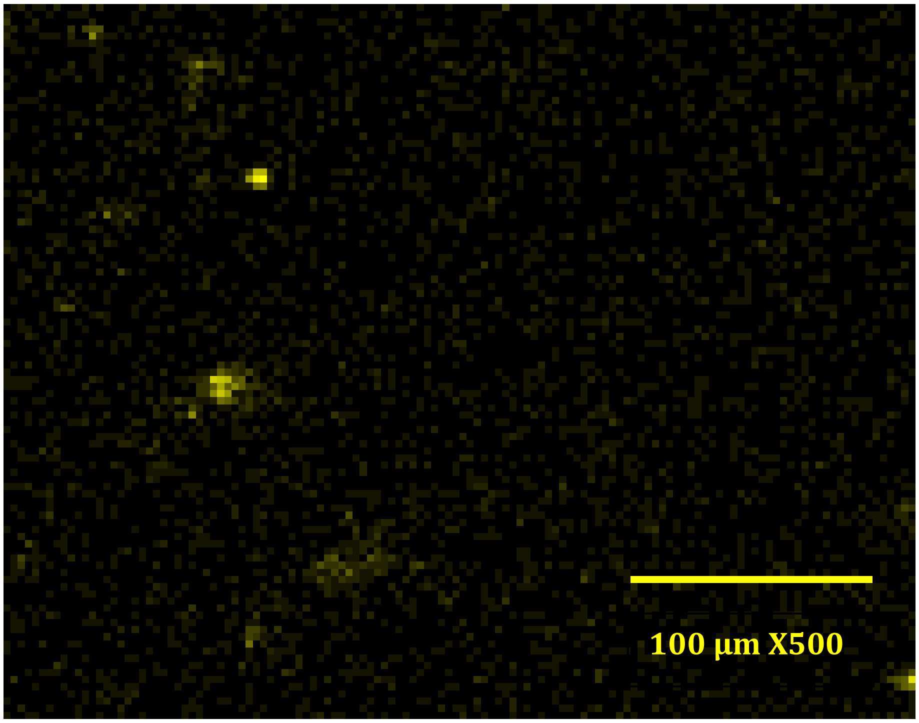

FIGURE 18. EDS image, group B1, Specimen 1.

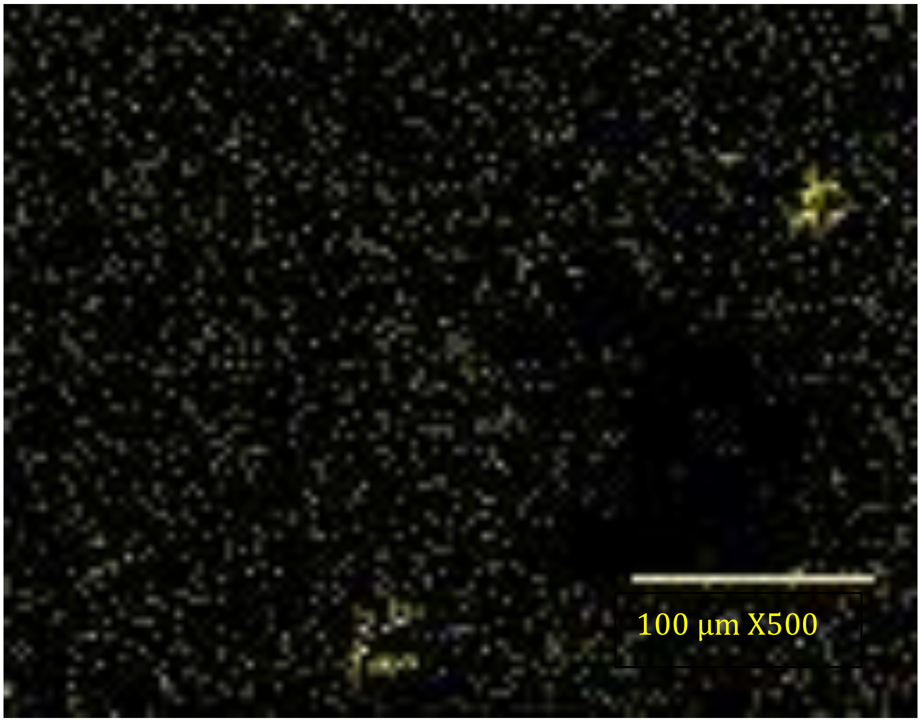

FIGURE 19. EDS image, group B1, Specimen 2.

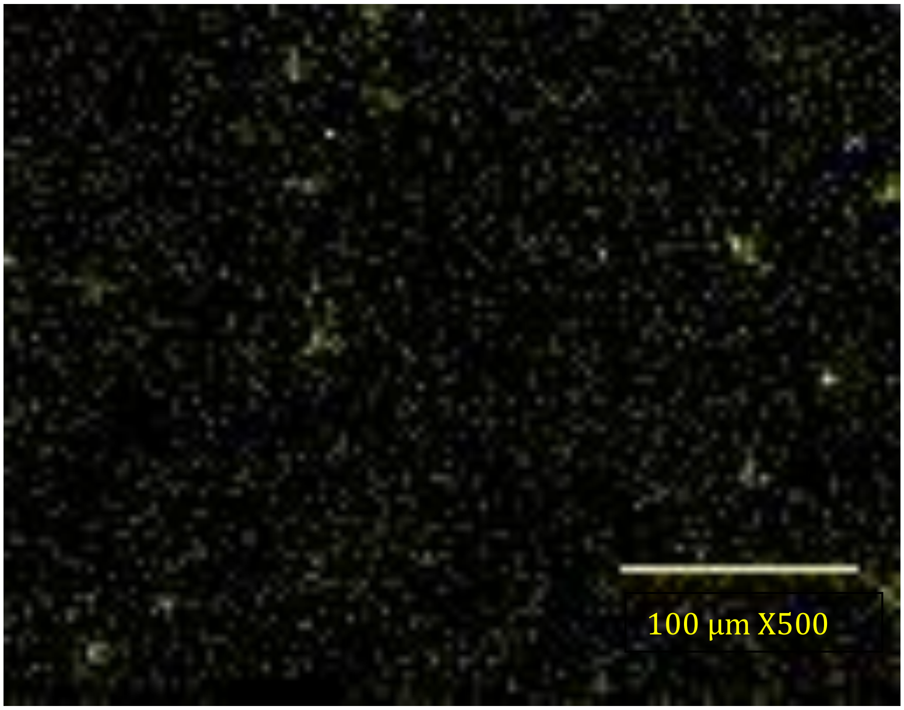

FIGURE 20. EDS image, group B2, Specimen 1.

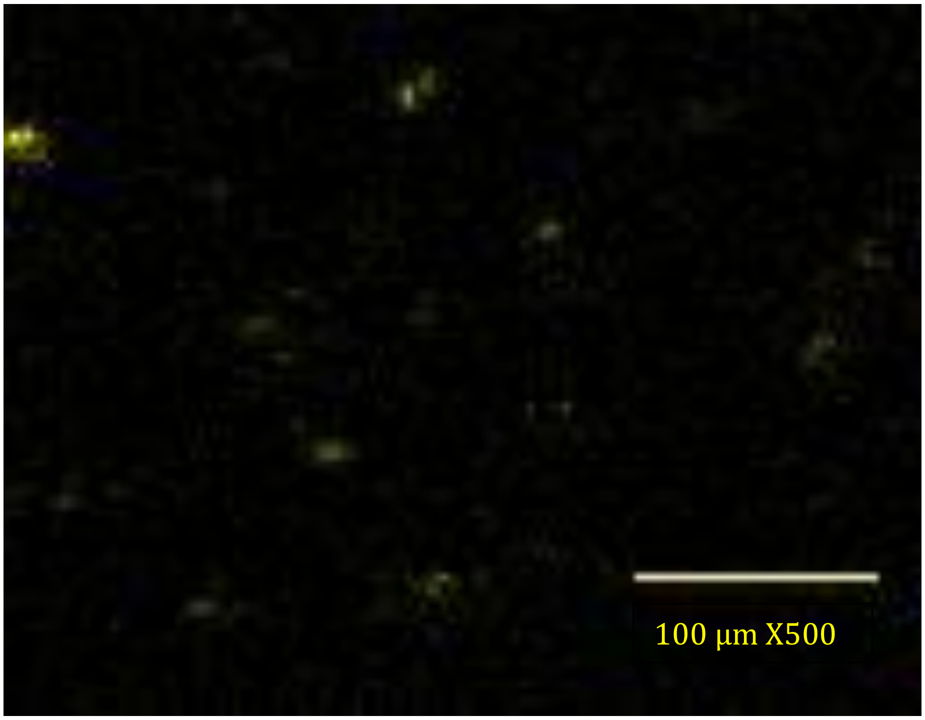

FIGURE 21. EDS image, group B2, Specimen 2.

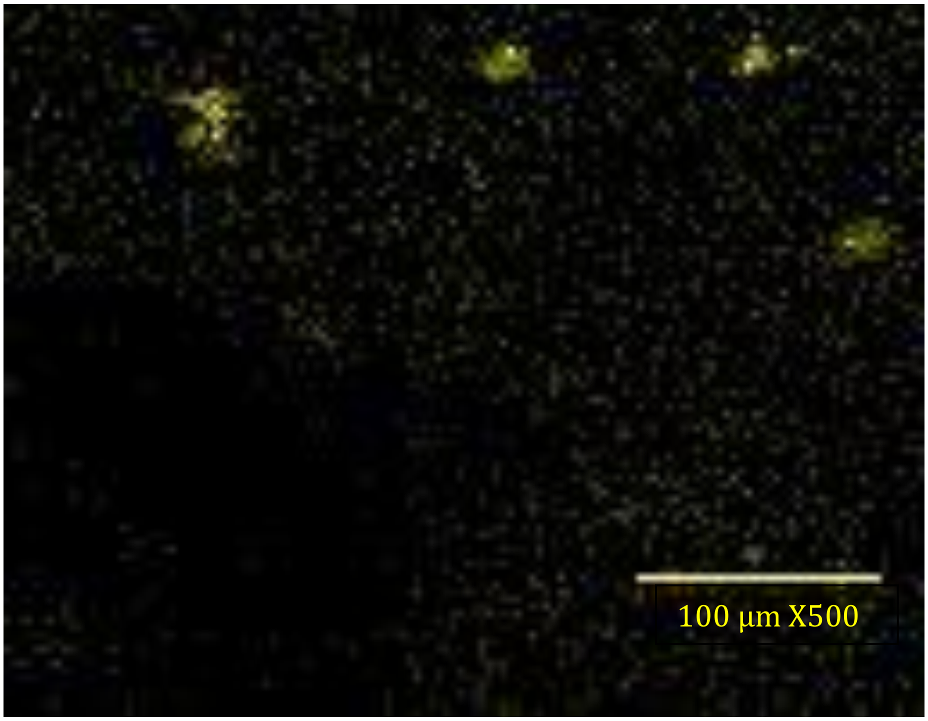

FIGURE 22. EDS image, group B3,Specimen 1.

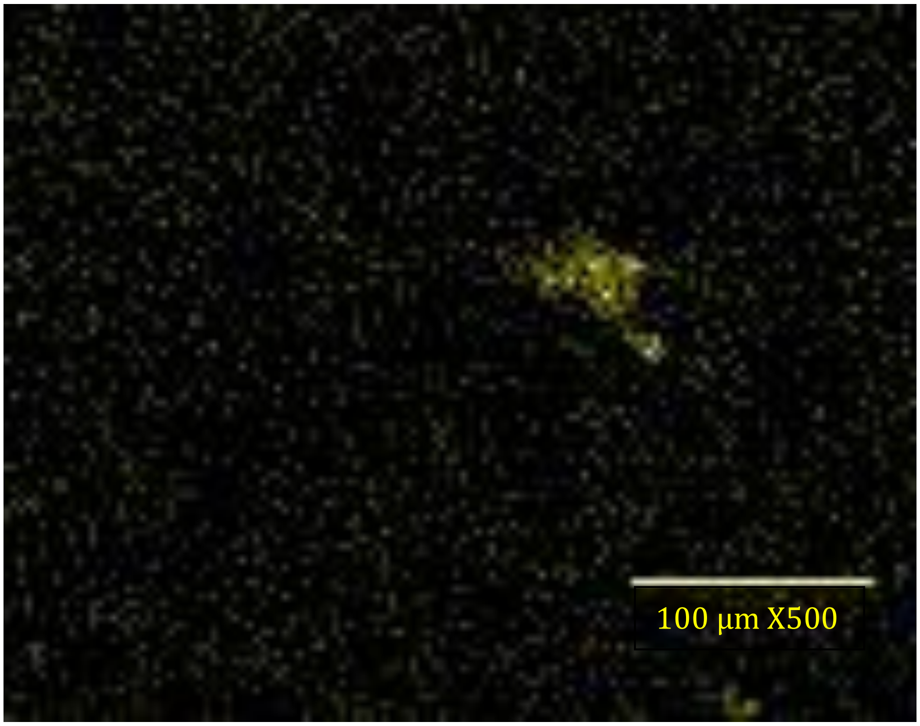

FIGURE 23. EDS image, group B3, Specimen 2.

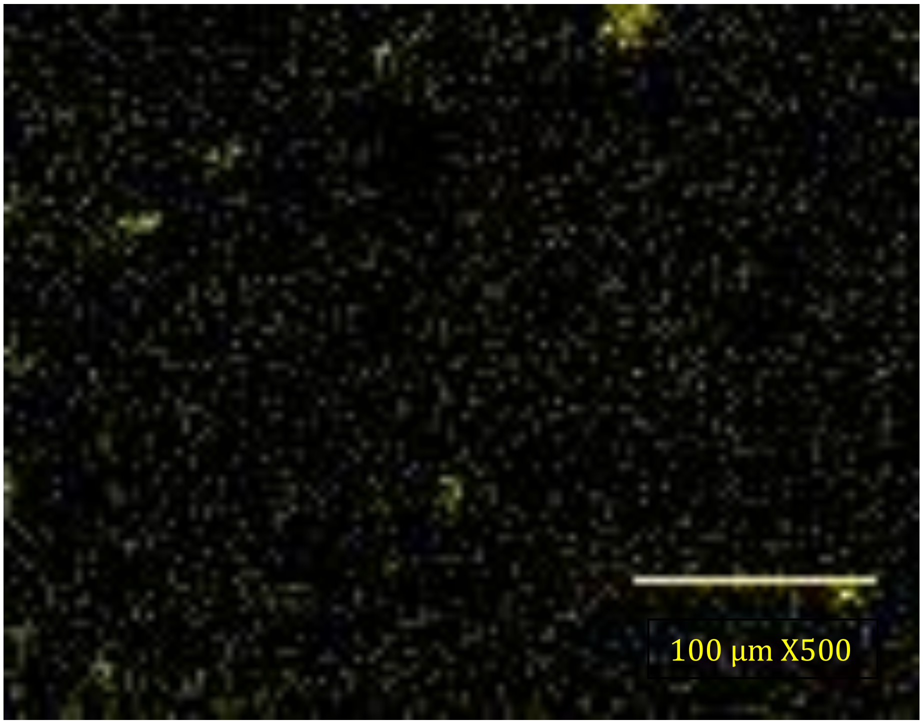

FIGURE 24. EDS image, group B4, Specimen 1.

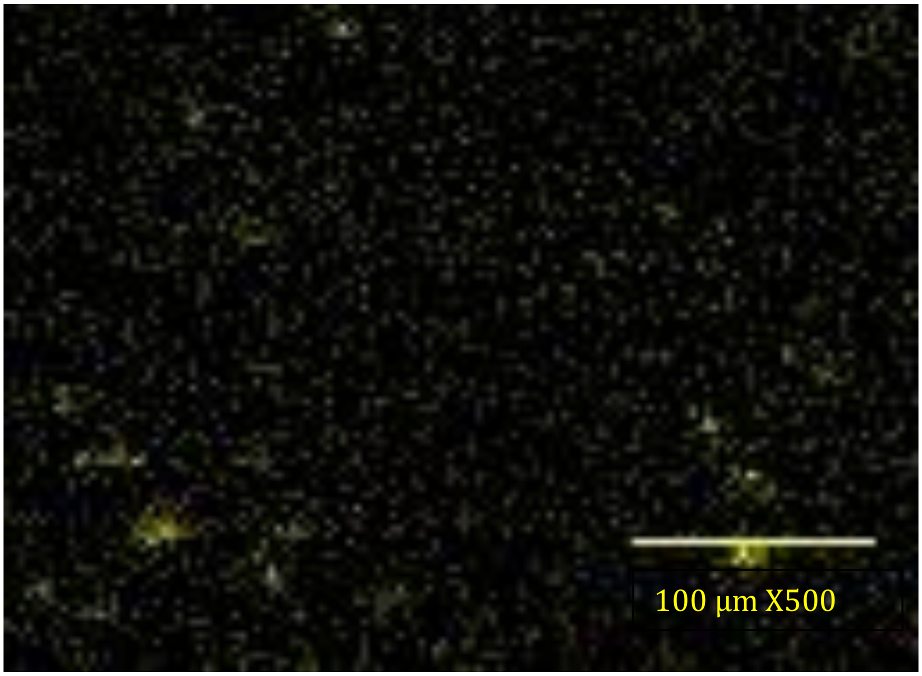

FIGURE 25. EDS image, group B4, Specimen 2.

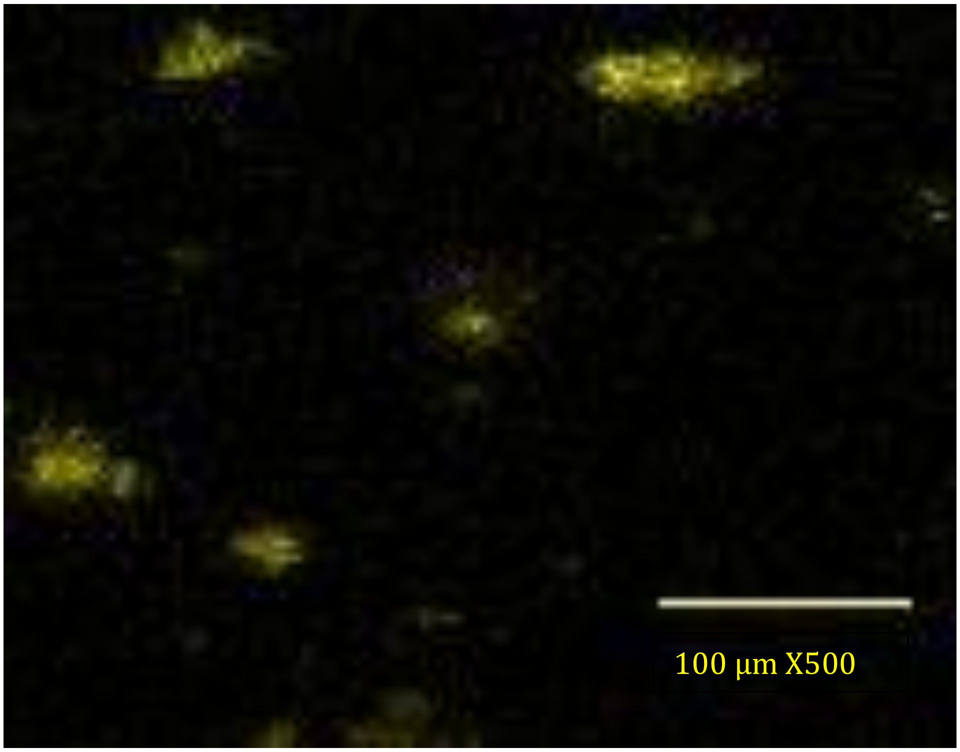

FIGURE 26. EDS image, group C1, Specimen 1.

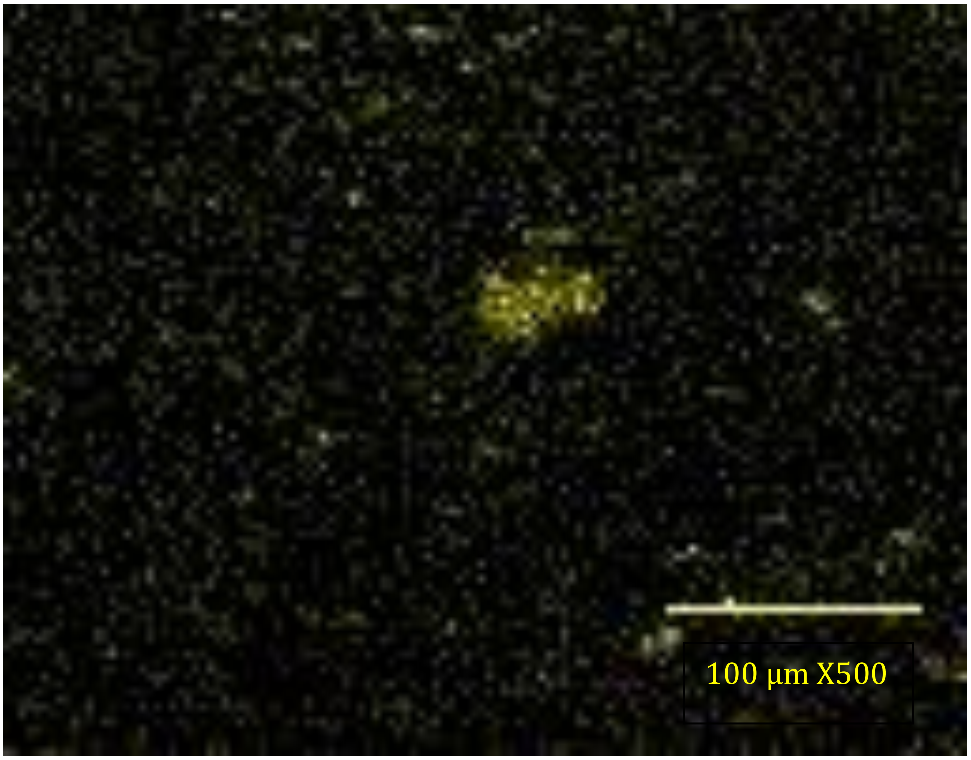

FIGURE 27. EDS image, group C1, Specimen 2.

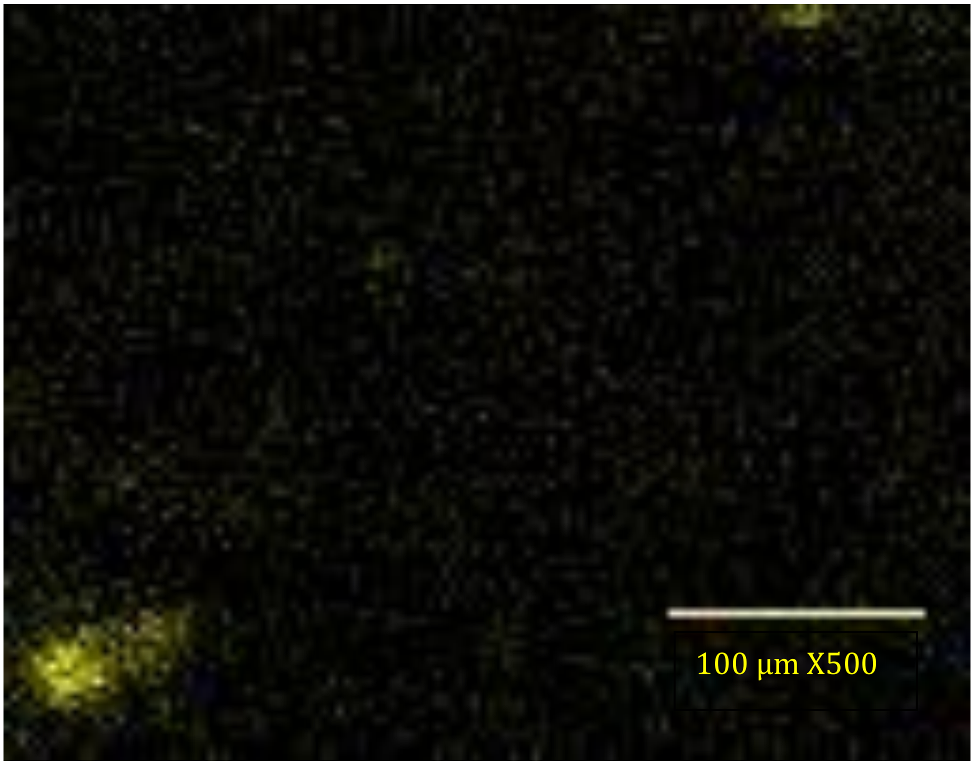

FIGURE 28. EDS image, group C2, Specimen 1.

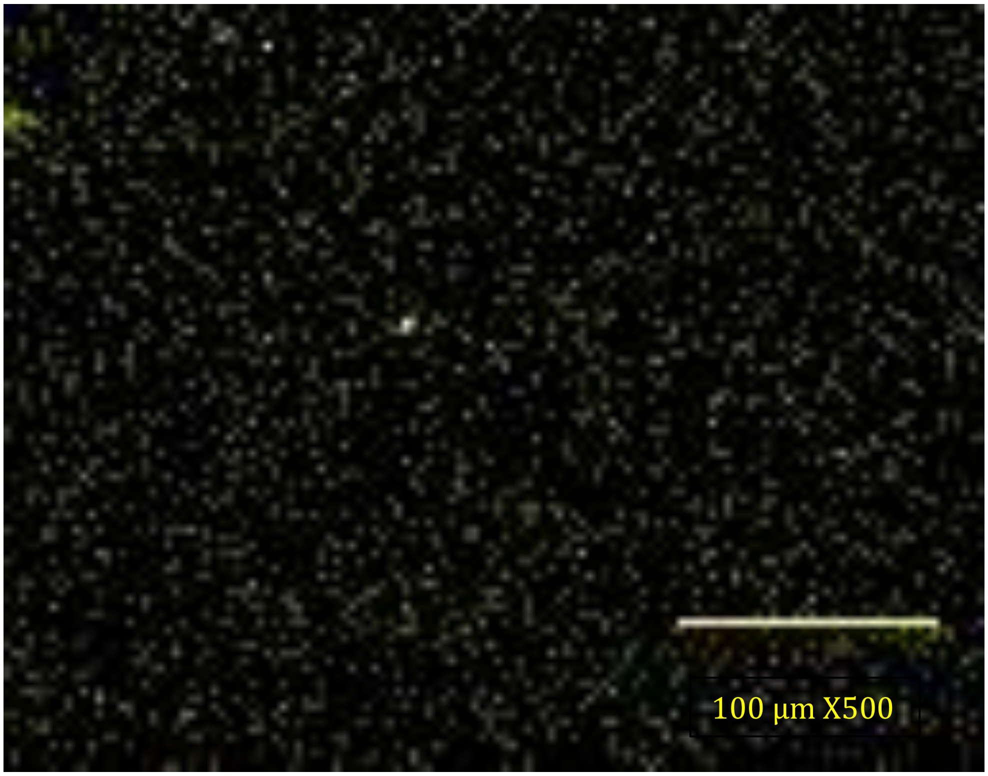

FIGURE 29. EDS image, group C2, Specimen 2.

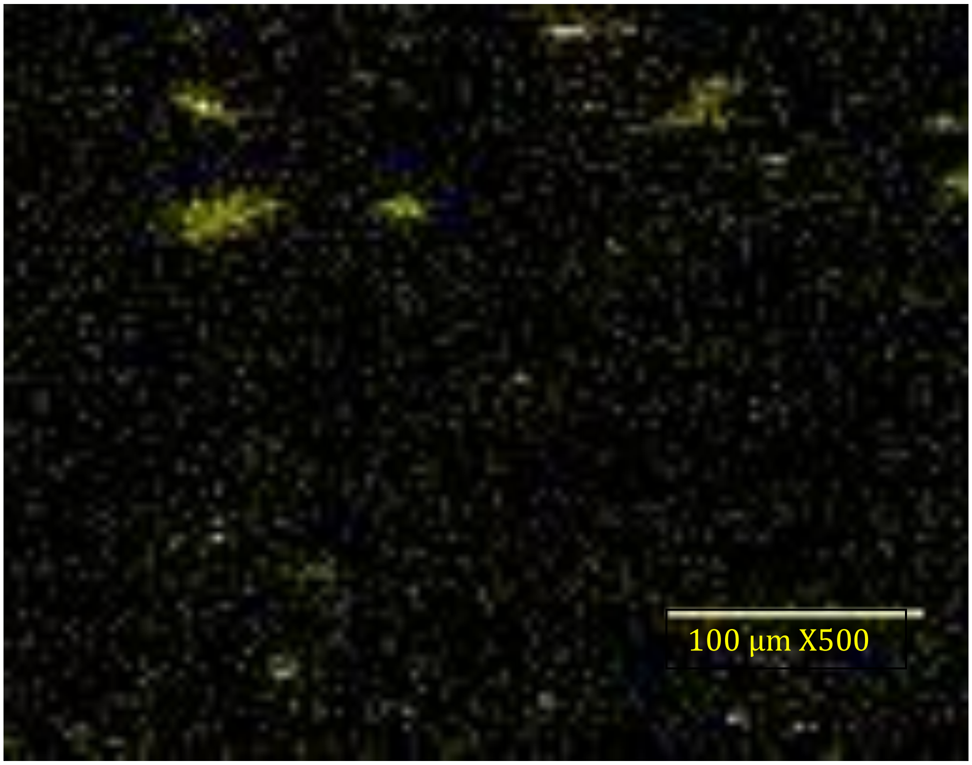

FIGURE 30. EDS image, group C3, Specimen 1.

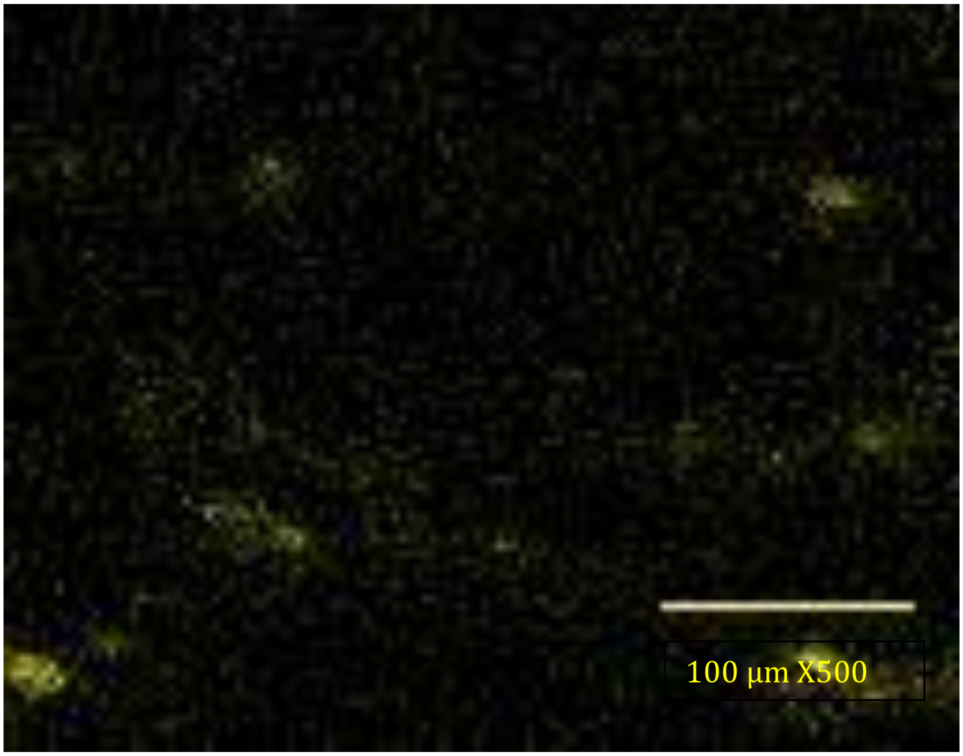

FIGURE 31. EDS image, group C3, Specimen 2.

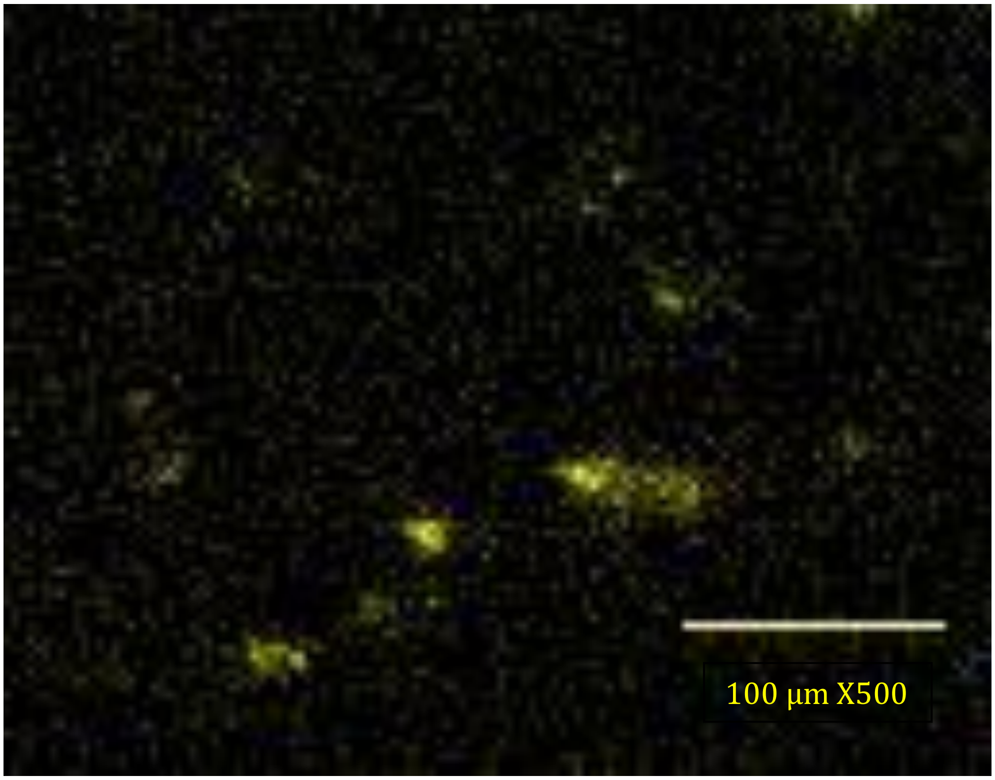

FIGURE 32. EDS image, group C4, Specimen 1.

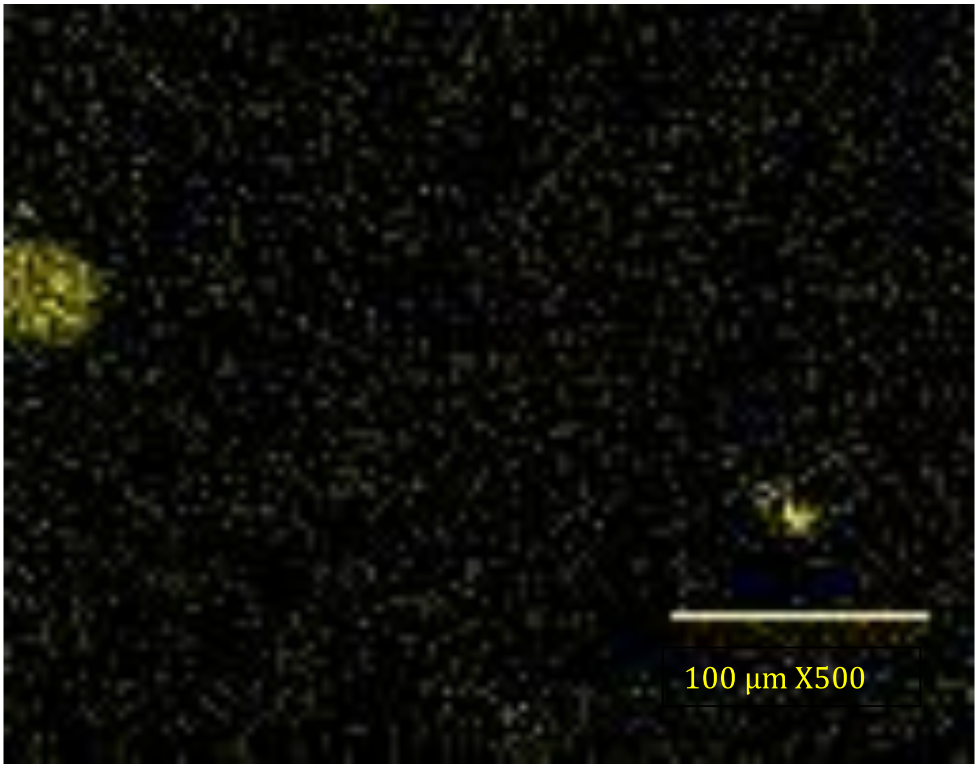

FIGURE 33. EDS image, group C4, Specimen 2.

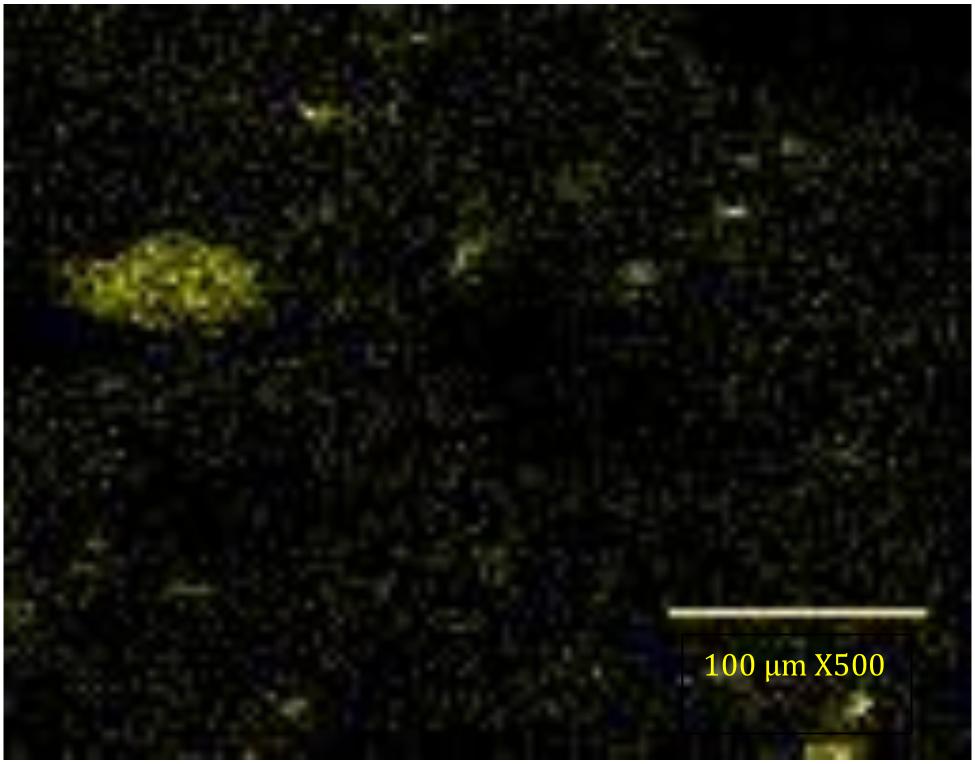

FIGURE 34. EDS image, group D1, Specimen 1.

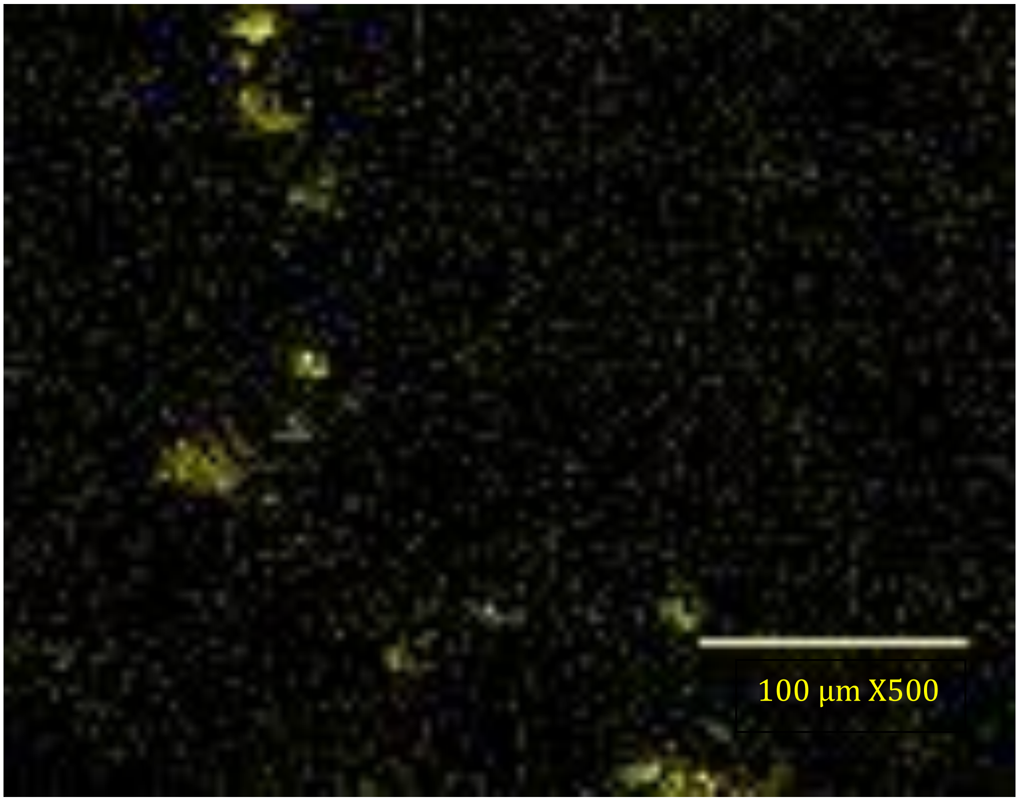

FIGURE 35. EDS image, group D1, Specimen 2.

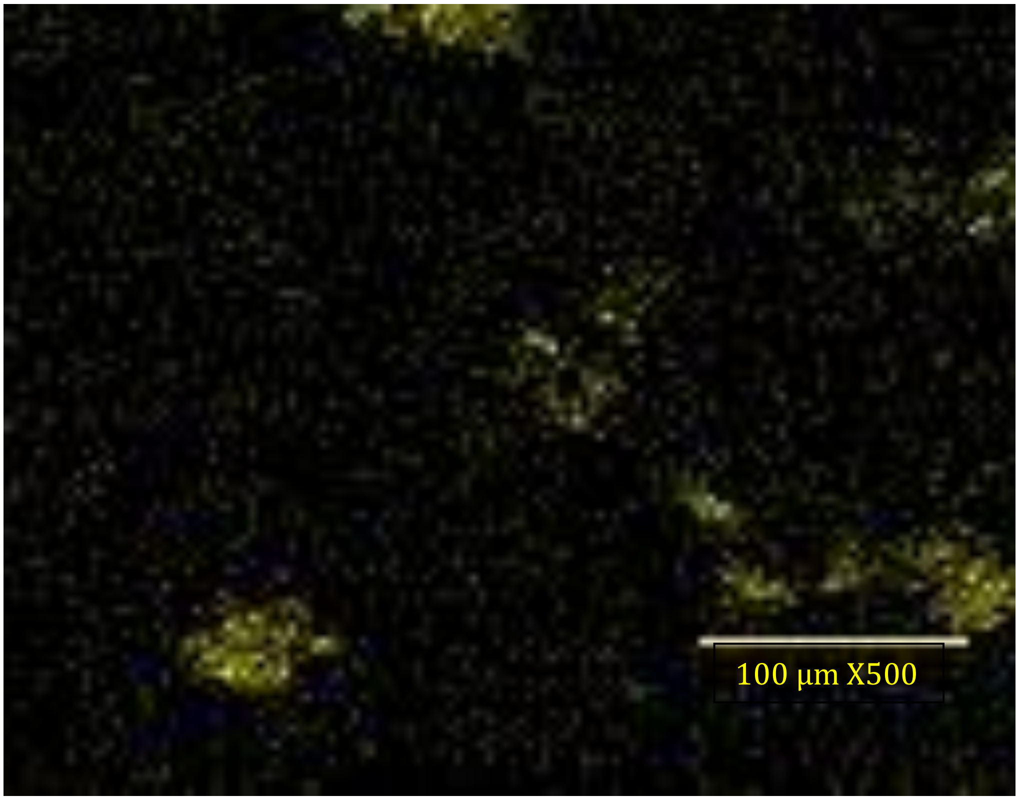

FIGURE 36. EDS image, group D2, Specimen 1.

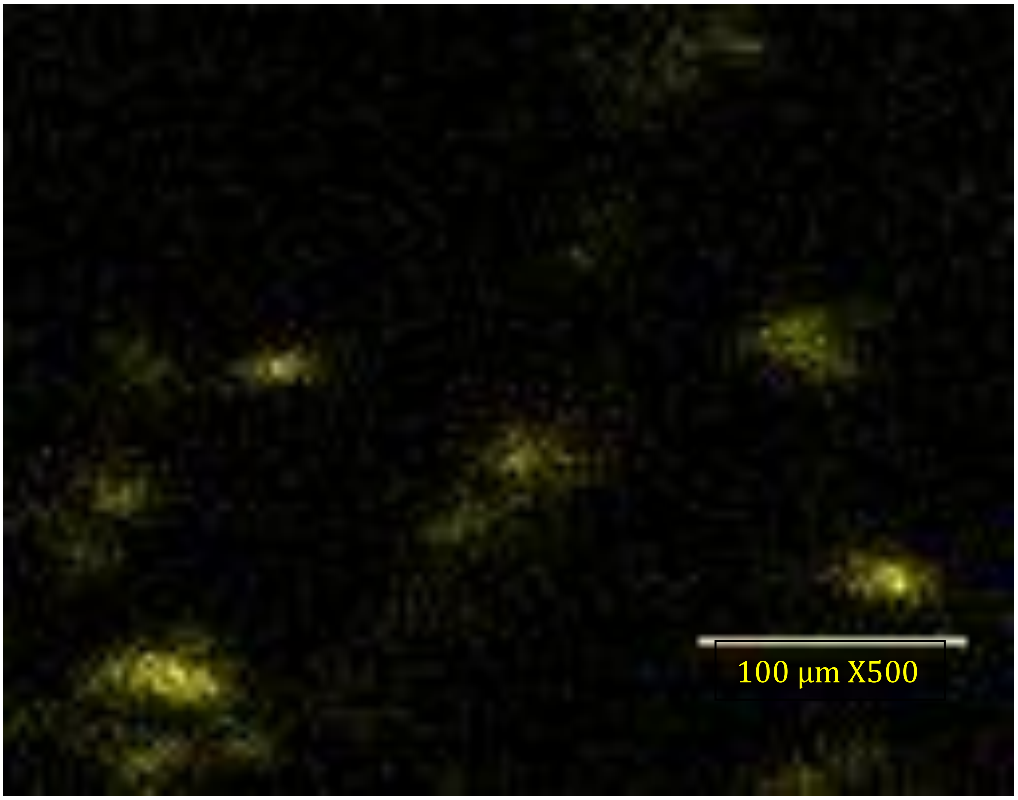

FIGURE 37. EDS image, group D2, Specimen 2.

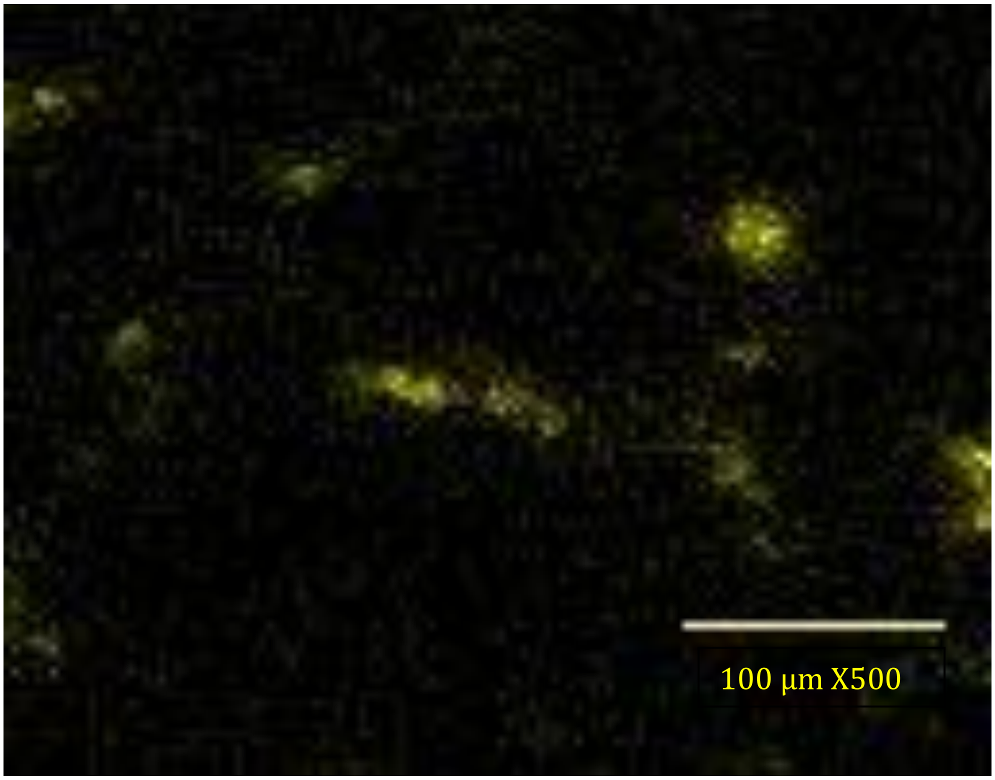

FIGURE 38. EDS image, group D3, Specimen 1.

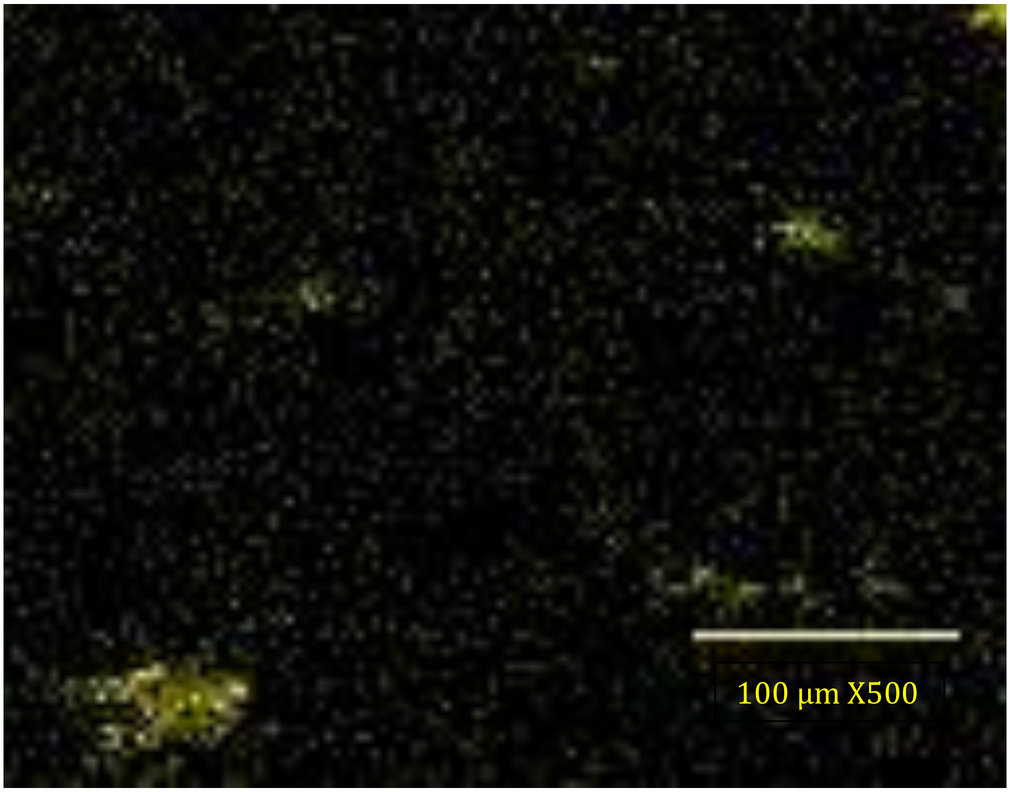

FIGURE 39. EDS image, group D3, Specimen 2.

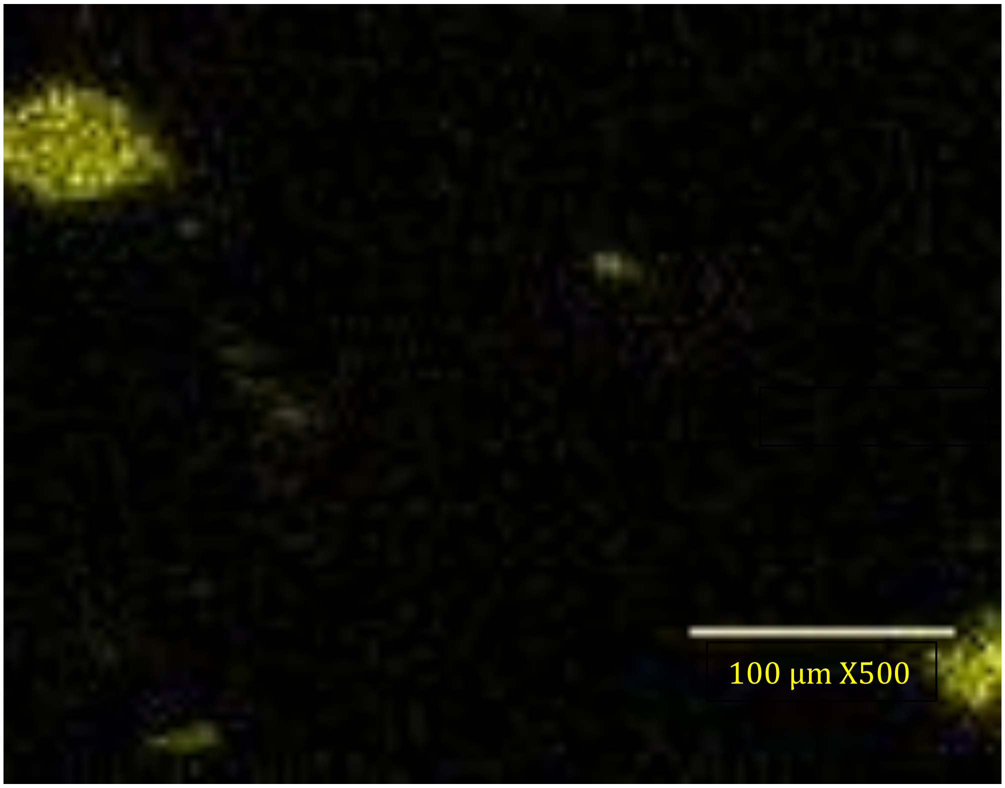

FIGURE 40. EDS image, group D4, Specimen 1.

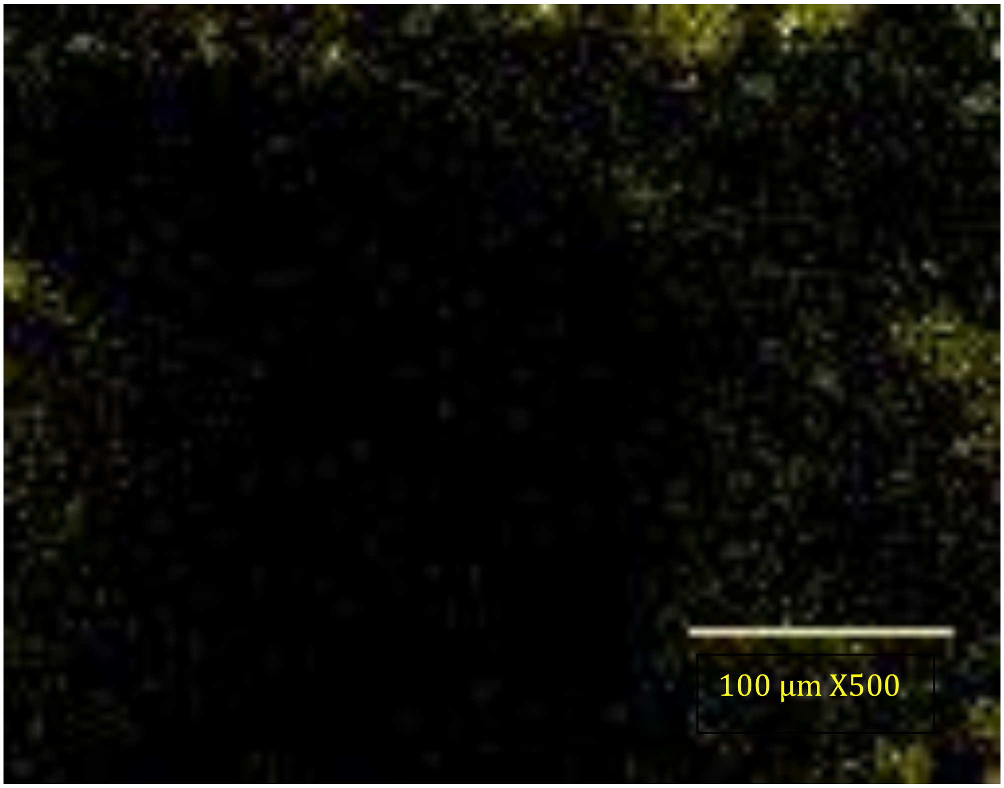

FIGURE 41. EDS image, group D4, Specimen 2.

DISCUSSION

Throughout the literature, many attempts have been made to evaluate the mechanical properties of different PMMA acrylic resins. Results of these studies have shown a wide range of variation depending on the type of the material, testing settings, specimens' geometry, or reinforcing materials. Although laboratory testing under static loading may not reflect intraoral conditions; these values are nevertheless helpful in comparing materials under controlled situations and may be a useful predictor of clinical performance.

Wang et al.[26] reported control flexural strength values for Jet acrylic resin in the range of 56 MPa and Knoop microhardness values at 11 kg/mm^2. Gegauff et al.[30,31] reported the mean value of fracture toughness for Jet acrylic resin to be 1.2 MPa.m$^{0.5}$. Hamza et al.[33] reported lower flexural strength values of 52.88 MPa and fracture toughness values of 1.25 MPa.m$^{0.5}$. Larson et al.[36] reported the mean modulus of elasticity of unreinforced Jet acrylic at 1950 MPa. Solnit [44] reported flexural strength values of 67.69 MPa for unreinforced controls. Shimizu [55] reported mean control values for Jet acrylic of flexural strength at 68.3 MPa, flexural modulus at 1698.32 MPa, fracture toughness at 1.37 MPa.m$^{0.5}$, and microhardness of 10.17 kg/mm^2. Haselton et al.[56] and Hernandez et al.[21] reported higher flexural strength values up to 89.9 MPa and 100 MPa, respectively. In our current project, we obtained flexural strength of 73.48 MPa, flexural modulus of 2203.37 MPa, fracture toughness of 1.66 MPa.m$^{0.5}$, and Knoop hardness of 11.43 kg/mm^2 for the control group. The data obtained from this study showed that control sample values were in the acceptance range compared with previous research.

Provisional fixed partial denture acrylic resins have been evaluated in several studies with different methods of reinforcements. Hamza et al.[33] found that the use of glass and polyethylene fibers may be an effective way to reinforce resins used to fabricate fixed provisional restorations. Vallittu showed that unidirectional and woven glass fibers significantly improved fracture resistance and prevented catastrophic failures.[34] Samadzadah et al.[35] found the incidence of catastrophic failures of different provisional restorative materials has been reduced with the incorporation of polyethylene fibers. Larson et al.[36] compared elastic moduli of three different provisional resins after addition of carbon graphite fibers, and he concluded the modulus of elasticity increased significantly with fiber incorporation of the three resins tested. Yazdanie and Mahood found that carbon fiber-acrylic resin composites were stronger and stiffer than unfilled controls.[37]

In the current study, there was no significant difference in flexural strength values within groups under the two storage conditions (1 day vs. 7 days) (Table V). The experimental groups did not significantly enhance or decrease the values of this particular test compared with the control group (Table VI, VII). Flexural strength results of our study suggest that adding alumina nanofibers (0.5 wt %, 1.0 wt %, or 2.5 wt %) with quaternary ammonium acetate dispersant (1.0 wt %, 2.0 wt %, or 5.0 wt %) to Jet Tooth Shade PMMA acrylic resin material did not improve the flexural strength of our experimental resin.

Multiple comparisons between test groups showed group D (group with the highest percentage of alumina nanofibers 2.5 percent) was significantly weaker than most of the other test groups. This decrease in flexural strength with increased alumina nanofiber loading was contrary to expectations. With conventional resin composite systems, increased filler content is usually responsible for improved mechanical properties. The reduction in flexural strength of alumina nanofiber-PMMA composites can be attributed to the poor bonding at the filler/matrix interface. In fact, the presence of poorly bonded fibers, to which little load is transferred, can be almost equivalent to voids.[57] Poor adhesion between the acrylic matrix and reinforcement leads to lower mechanical properties.[58] Solnit tested the flexural strength of PMMA resin after the addition of silane-treated and untreated glass fibers. Samples with untreated fibers tested weaker than unreinforced controls. The samples with treated fibers tested stronger, but the difference was not statistically significant.[44] Kotha et al. demonstrated the effect of using a silane coupling agent to improve the mechanical properties of steel fiber-reinforced acrylic bone cements. He found that the elastic modulus and fracture toughness of the silane-coated, steel fiber-reinforced bone cements are significantly higher than the bone cements reinforced with uncoated steel fibers. The use of coupling agents improve bonding between the acrylic matrix and reinforcements by the formation of strong covalent bonds instead of weak secondary bonds (van der Waals).[58] In addition, as seen with EDS images, alumina nanofibers had a tendency to agglomerate. Lack of proper dispersion of the nanofibers detrimentally affects the mechanical properties.[50] The loose-form fibers, without a chemical bond between the fibers and the matrix, caused microfractures and significantly weakened the resulting resin.[52] Wang et al. evaluated the effect of carbon fibers reinforcement on cement-based composite with the use of dispersant and ultrasonic vibration.[51] He found that when the fibers are properly dispersed, there were significant improvements in compressive and tensile strength.

When they used more than 0.6-percent carbon fibers by weight, it was difficult to achieve proper dispersion, which resulted in significant reduction in the mechanical properties.[51]

In the current study, the storage condition (1 day vs. 7 days) has no effect on flexural modulus of the provisional PMMA resin except groups D2, D3, and D4. Flexural modulus of the aforementioned groups in one-day storage conditions is significantly higher than their counterparts in the seven-day group (Table V). This could be attributed to the higher percentage of alumina nanofibers. Water is known to penetrate the polymer network and thereby cause dissolution of unreacted monomers and polymer chains. The loose-form alumina nanofibers acting as inclusion bodies, and without a chemical bond between the fibers and the matrix, each fiber actually caused a microfracture and significantly weakened the resulting PMMA resin.[44]

The experimental groups did not significantly enhance or decrease the values of this particular test compared with the control group (Tables VI, VII). Flexural modulus results of our study suggest that adding alumina nanofibers (0.5 wt %, 1.0 wt %, or 2.5 wt %) with quaternary ammonium acetate dispersant (1.0 wt %, 2.0 wt %, or 5.0 wt %) to Jet Tooth Shade PMMA acrylic resin material did not improve the flexural modulus of our experimental resin.

In the current study, the storage condition (1 day vs. 7 days) has no effect on fracture toughness of the provisional PMMA resin. Fracture toughness results of our study suggest that adding alumina nanofibers (0.5 wt %, 1.0 wt %, or 2.5 wt %) with quaternary ammonium acetate dispersant (1.0 wt %, 2.0 wt %, or 5.0 wt %) to Jet tooth shade PMMA acrylic resin material did not improve the fracture toughness of our experimental resin. This could be attributed to the poor bonding at the filler/matrix interface and the lack of proper dispersion of the nanofibers.

In the current study, the storage condition (1 day vs. 7 days) has a significant effect on Knoop microhardness of the provisional PMMA resin except in groups B1, B4, and D4. Microhardness numbers of seven-day storage condition groups A, B2, B3, C1, C2, C3, C4, D1, D2, and D3 were significantly higher than their one-day storage condition counterparts (Table V). This increase in microhardness with increased storage time was contrary to expectations. Water acts as external plasticizer, and with increased storage time, there is a supposed decrease in microhardness.[47] It is possible that the polymer was undergoing further polymerization reaction. The increase in the mechanical properties may have exceeded the effects of plasticizing and lead to a higher microhardness at 7 days. The plasticizing effect is expected to affect the surface layer of the specimen where water can penetrate by diffusion. In microhardness testing, the specimen is under compressive stress. When the diffusion layer is thin, the softened surface layer can be supported by the stronger and more-crosslinked layer underneath and show a higher hardness value. On the contrary, in bending, the softened layer can serve as a crack-initiation site. Though the layer underneath is stronger at 7 days, the crack-initiation site becomes an area with stress concentration and leads to a lower flexural strength and toughness at 7 days.

Some test groups under one-day storage conditions (B1, B2, B3, C1, C4, D1, D2 and D3) and under seven-day storage conditions (B1, B4 and C4) had significantly lower Knoop microhardness numbers than the control group.

ENERGY DISPERSIVE SPECTROMETRY

A total of 36 elemental mapping images (EDS) were taken (3 images/group) using low-vacuum SEM (JEOL 5310LV) with aluminum being the element of interest. EDS maps showed poor dispersion of the alumina nanofibers with groups of higher fiber contents tends to form more agglomerates. This suggested that direct dispersion of alumina nanofibers in methyl methacrylate monomer and quaternary ammonium acetate dispersant was not effective to separate the nanofibers into nano-scaled single crystals. Also, it suggested that the use of a magnetic stirrer was not effective in physically breaking nanofibers agglomerates.

SUMMARY AND CONCLUSIONS

This study investigated the effects of alumina nanofiber reinforcement on the mechanical properties of commercially available provisional fixed partial denture polymethyl methacrylate resin (Jet Tooth Shade). Alumina nanofibers were added at 0.0 wt %, 0.5 wt %, 1.0 wt %, and 2.5 wt %, of total material weight. A quaternary ammonium acetate dispersant was added to the acrylic monomer at 0.0 wt %, 1.0 wt %, 2.0 wt % and 5.0 wt % of the nanofiber weight. Samples from each group were evaluated for flexural strength, flexural modulus, fracture toughness, and microhardness. The samples were tested after storing in distilled water for 24 hours and 7 days at 37ºC. Energy dispersive spectrometry was used to qualitatively evaluate the dispersion of the fibers. Two-way analysis of variance (ANOVA) was used to test the effects of storage time and combinations of alumina nanofiber level and quaternary ammonium acetate dispersant level on the flexural strength, fracture toughness, and microhardness of the provisional PMMA resin. Pair-wise comparisons between groups were performed using Tukey’s multiple comparisons procedure to control the overall significance level at 5 percent.

The data obtained from this study showed that control sample values were in the acceptance range compared with previous research. The experimental samples did not reinforce the provisional resin in the flexural strength, modulus, fracture toughness, or microhardness.

Several factors may attribute to these results, such as poor bonding at the filler/matrix interface. The more homogeneous the mixture of PMMA and fiber, the stronger the acrylic resin.[44] In fact, the presence of poorly bonded fibers, to which little load is transferred, can be almost equivalent to voids.[57] The use of a magnetic stirrer was not effective in physically separating the nanofibers agglomerates. In addition, as seen with EDS images, alumina nanofibers had a tendency to agglomerate. Direct dispersion of alumina nanofibers in methyl methacrylate monomer and quaternary ammonium acetate dispersant was not effective in separating the nanofibers into nano-scaled single crystals. The presence of fiber agglomerates acts as a structural defect that detrimentally affects the mechanical properties.[50]

With the advancement of the technology and with new procedures, it will be possible to produce stronger and more cost-effective composites. Further studies are needed to evaluate the effectiveness of fibers, dispersion techniques, and coupling agents to enhance the mechanical properties of the provisional PMMA resin.

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ABSTRACT

EVALUATION OF MECHANICAL PROPERTIES OF PROVISIONAL FIXED PARTIAL DENTURE PMMA MATERIAL CONTAINING ALUMINA NANOFIBERS

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Provisional restorative treatment is an essential part of fixed prosthodontics. Incorporation of adequately constructed provisional restorations will enhance the success rate of definitive restorations. Repairing or replacing failed provisional restorations is a concern for both clinicians and patients.

The objective of this investigation was to study the effects of alumina nanofibers reinforcement on the mechanical properties of commercially available provisional fixed partial denture PMMA material. The hypothesis was that the addition of alumina nanofibers to commercially available PMMA resin will significantly increase its flexural strength, fracture toughness, and microhardness. Alumina nanofibers at 0.0 wt %, 0.5 wt %, 1.0 wt %, and 2.5 wt % were added to commercially available provisional fixed partial material (Jet Tooth Shade). A quaternary ammonium acetate dispersant (CC-59,

Goldschmidt, Janesville, WI) was added to the acrylic monomer at 0.0 wt %, 1.0 wt %, 2.0 wt % and 5.0 wt % of the nanofiber weight (12 test groups, 1 control). Samples from each group were evaluated for flexural strength, flexural modulus, fracture toughness, and microhardness. The samples were tested after storing in distilled water for 24 hours and 7 days at 37ºC. Two-way analysis of variance (ANOVA) was used to test the effects of storage time and combinations of alumina nanofiber level and quaternary ammonium acetate dispersant level on the flexural strength, fracture toughness, and microhardness of the provisional PMMA resin. Pair-wise comparisons between groups were performed using Tukey's multiple comparisons procedure to control the overall significance level at 5 percent. Three fracture toughness samples/group were randomly selected for Energy Dispersive Spectrometry (EDS) to qualitatively evaluate the dispersion of the fibers.

The data obtained from this study showed that control sample values were in the acceptance range compared with previous research. The experimental samples did not reinforce the provisional resin in the flexural strength, modulus, fracture toughness, or microhardness. There are several factors may attribute to these results, such as poor bonding at the filler/matrix interface. The more homogeneous the mixture of PMMA and fiber, the stronger the acrylic resin. In fact, the presence of poorly bonded fibers, to which little load is transferred, can be almost equivalent to voids. In addition, as seen with EDS images, alumina nanofibers had a tendency to agglomerate. The use of a magnetic stirrer was not effective in physically separating nanofibers agglomerates. Direct dispersion of alumina nanofibers in methyl methacrylate monomer and quaternary ammonium acetate dispersant was not effective in separating the nanofibers into nano-scaled single crystals. The presence of fiber agglomerates acts as a structural defect that detrimentally affects the mechanical properties. Further studies are needed to evaluate the effectiveness of fibers, dispersion techniques, and coupling agents to enhance the mechanical properties of the provisional PMMA resin.

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