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Effect of Fucoidan Incorporation on the Physicochemical Properties of Dental Restorative Materials

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Research Article

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ABSTRACT

Objectives: The aim of this study was to evaluate the wettability, surface and molecular characterization of three different restorative materials (Mineral Trioxide Aggregate [MTA], glass ionomer cement [GIC] and alkasite [AL]) added with fucoidan.

Materials and Methods: Nine experimental groups were established by incorporating 0%, 2.5%, and 5% fucoidan by weight into the powder phase of MTA, AL, and GIC, followed by mixing according to manufacturers' instructions. Cylindrical samples (3 mm diameter × 2 mm height) were molded and stored at 37°C under 100% humidity. Contact Angle Analysis**,** SEM/EDS, and FTIR spectroscopy evaluations were performed. One-way ANOVA and Tukey post-hoc tests ($p < 0.05$) were applied for statistical analysis.

Results: Fucoidan incorporation induced material-specific changes in wettability, chemical composition, and surface morphology. While contact angle values increased in MTA and AL, GIC showed improved wettability at 2.5%. FT-IR analysis confirmed chemical interactions, especially through glycosidic and sulfate bands. SEM-EDS revealed disrupted or reorganized microstructures and altered elemental distributions, including increased fluorine and silicate presence.

Conclusions: Fucoidan addition improved wettability without compromising the matrix, supporting its potential for advanced restorative materials.

Keywords: Alkasite, fucoidan, glass ionomer cement, MTA, wettability

Fukoidan İlavesinin Dental Restoratif Malzemelerinin Fizikokimyasal Özelliklerine Etkisi

Araştırma Makalesi

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ÖZ

Amaç: Bu çalışmanın amacı, fukoidan eklenmiş üç farklı restoratif materyalin (Mineral Trioksit Agregat [MTA], cam iyonomer siman [CİS] ve alkasit [AL]) ıslanabilirlik, yüzey özellikleri ve moleküler karakterizasyonunu değerlendirmektir.

Gereç ve Yöntemler: MTA, AL ve GIC'in toz fazına ağırlıkça %0, %2,5 ve %5 oranlarında fukoidan ilavesiyle dokuz deney grubu oluşturulmuştur. Karışımlar üretici talimatlarına göre hazırlanmış, silindirik örnekler (3 mm çap × 2 mm yükseklik) kalıplanarak 37°C'de ve %100 nem koşullarında bekletilmiştir. Temas Açısı Analizi, SEM/EDS ve FTIR spektroskopi değerlendirmeleri gerçekleştirilmiştir. İstatistiksel analizde tek yönlü ANOVA ve Tukey çoklu karşılaştırma testleri uygulanmıştır ($p < 0,05$).

Bulgular: Fukoidan ilavesi, materyale özgü değişiklikler oluşturmuştur. MTA ve AL gruplarında temas açısı değerleri artarken, CİS grubunda %2,5 fukoidan ilavesiyle ıslanabilirlik artışı gözlenmiştir. FTIR analizleri, özellikle glikosidik ve sülfat bantları üzerinden kimyasal etkileşimlerin varlığını doğrulamıştır. SEM-EDS incelemeleri, bozulmuş veya yeniden düzenlenmiş mikroyapılar ile flor ve silikat varlığında artış dahil olmak üzere değişen elementel dağılımlar göstermiştir.

Sonuçlar: Fukoidan ilavesi, matriks bütünlüğünü koruyarak ıslanabilirliği artırmış ve ileri düzey restoratif materyallerin geliştirilmesinde potansiyel bir katkı maddesi olarak değerlendirilebileceğini ortaya koymuştur.

Anahtar Kelimeler: Alkazit, fukoidan, cam iyonomer siman, MTA, ıslanabilirlik

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Introduction

In the field of restorative dentistry, bioactive materials have gained significant attention due to their ability to support tissue regeneration, antimicrobial resistance, and long-term sealing effectiveness.[1] Conventional materials such as Mineral Trioxide Aggregate (MTA), Glass Ionomer Cement (GIC), and the more recent Alkasite (AL) materials are frequently used in vital pulp therapies and as base, liner or filling materials. Despite their clinical success, these materials have limitations,

including suboptimal antibacterial properties, insufficient early mechanical strength, and poor handling characteristics.[2] To overcome these drawbacks, researchers have explored the incorporation of biopolymers and natural bioactive compounds into dental materials.

Fucoidan is a high molecular weight sulfated polysaccharide extracted from various species of brown seaweed, such as *Fucus vesiculosus*, *Laminaria japonica*, and *Undaria pinnatifida*. Structural derivatives of fucose-containing sulfated polysaccharides, its composition varies depending on algal source and extraction method.[3] Fucoidan exhibits a wide range of bioactivities, including anticoagulant, antioxidant, antibacterial, antiviral, anti-inflammatory, and even osteoinductive properties.[4-6] These features make fucoidan an attractive candidate for biomedical applications, including drug delivery, tissue engineering, and regenerative medicine.[3,7,8]

Within the dental field, there has been growing interest in polysaccharide-based materials, such as chitosan and alginate, for their adhesive and regenerative potential.[9] However, the use of fucoidan in dental biomaterials remains relatively underexplored. Fucoidan supports tissue regeneration by enhancing growth factor activity, promoting collagen deposition, angiogenesis, and osteoblast function. Fucoidan may also influence hydroxyapatite (HA) formation due to its structural similarity to sodium alginate.[10] Its sulfate groups may also interact with calcium ions, influencing material hydration and setting processes in calcium silicate-based cements such as MTA.[11]

GICs, known for their chemical bonding to tooth structure and fluoride release, may benefit from fucoidan's biological properties by improving tissue response and surface bioactivity. Likewise, alkasite-based materials-which are resin-modified ion-releasing composites-may gain enhanced biofunctional surfaces upon fucoidan incorporation, particularly in terms of wettability and cell adhesion.

The surface wettability of a material is determined by measuring the contact angle (the angle formed by the solid sample surface and the tangent to the surface of the liquid droplet) and provides an idea of the material's hydrophobicity. As the angle increases, wettability decreases and is an indicator of the surface free energy, which plays an important role in bacterial adhesion.[12] Wettability plays a decisive role in adhesive bonding, curing kinetics and biocompatibility.

These three materials represent three main restorative systems with different setting mechanisms and matrix structures: calcium silicate-based pulp capping agents (MTA), acid-base-cured glass-ionomer cements (GICs), and resin-based ion-releasing hybrid systems (ALs). Despite its promising properties, no study has yet comprehensively evaluated the effects of fucoidan incorporation on the wettability, surface and molecular characterization of these three major classes of dental materials. To address this gap, the present study investigates the structural, surface, and wetting behavior of fucoidan-modified dental materials using contact angle analysis, Fourier-Transform Infrared Spectroscopy (FTIR), Scanning Electron Microscopy with Energy Dispersive X-ray Spectroscopy (SEM-EDS) analysis.

The null hypothesis of this study was that the incorporation of fucoidan into MTA, AL, and GIC does not significantly alter their surface chemistry and wettability.

Materials and Methods

This article does not contain any studies with human participants or animals performed by any of the authors; therefore, ethical approval was not required.

Materials

Three commercially available dental restorative materials were used in this study:

- Mineral Trioxide Aggregate (MTA Plus, Prevest Detpro Limited, Jammu, India)
- Glass Ionomer Cement (Fuji IX, GC, Tokyo, Japan)
- Alkasite (Cention N, Ivoclar Vivadent, Liechtenstein)
- Fuciodan (Henan Qianbaiguan Biotechnology Co., Ltd, China)

The properties of the materials used in the study are summarized in Table 1.

Methods

Experimental Groups

In this study, a total of nine experimental groups were established using three different dental materials. For each material, three subgroups were created based on the fucoidan incorporation ratios: 0% (control group), 2.5%, and 5% by weight. In the control groups, samples were prepared according to the manufacturers' instructions without any additive. (MTA0, GIC0, AL0) In the experimental groups, fucoidan was incorporated into the powder phase of each material at 2.5% and 5% (w/w) concentrations (MTA2.5, MTA5, GIC2.5, GIC5, AL2.5, AL5) replacing an equivalent amount of the original powder. The study design is shown in Figure 1.

Sample Preparation

For all three materials one scoop of each material's powder was placed into a glass dappen dish and weighed using a high-precision analytical balance (Precisa XB 220A, Precisa Instruments Ltd., Dietikon, Switzerland; sensitivity: 0.0001 g). The corresponding amount of fucoidan was then weighed separately and used to replace an equivalent amount of the base material. The powders were mixed thoroughly to achieve a homogeneous blend. This modified mixture was transferred to a mixing pad or glass slab, depending on the material, and one drop of the manufacturer-supplied liquid or gel was added. For all three materials (MTA, GIC, AL), fucoidan was added to the material powder manually using 2.5% or 5% of the manufacturer's recommended powder amount. Mixing was performed with a spatula for at least 60 seconds to ensure homogeneous distribution. Mixing was performed in accordance with each material's manufacturer's instructions until a uniform, paste-like consistency was obtained. Visual inspection and observation of clumping were performed after mixing to ensure no aggregates formed.

Molding and Storage

After mixing, all groups were placed into cylindrical molds (3 mm diameter × 2 mm height). Because this study focused on surface characterization, the sample dimensions do not directly represent clinical restoration dimensions. The 3 mm ×

2 mm cylindrical form was chosen to ensure adequate sample surface area for contact angle analyse, FTIR, SEM/EDS analyses and to facilitate device adaptation. The samples were allowed to set for 24 hours at 37°C in a humidified chamber (100% relative humidity). FTIR, SEM/EDS and contact angle measurements were made after this 24-hour curing period.

Contact Angle Measurement

For this analysis, three samples were prepared in each group. The measurements were performed using the fixed drop method with an optical goniometer (OCA 15 Plus, Dataphysics Instruments GmbH, Filderstadt, Germany). At room temperature, a 2.0 µL droplet of distilled water with a defined surface tension was placed on the solid surface using a micropipette. In this study, deionized water was chosen as the standard reference liquid to ensure direct comparability between different materials. Due to the constant surface tension of deionized water, the results directly reflect the differences in the surface energies of the materials.

The resulting contact angle was recorded with an optical camera and the droplet profile was captured. The software (SCA 20; Dataphysics Instruments GmbH, Filderstadt, Germany) automatically calculated the contact angle based on the measurement of the height and width of the droplet from the captured image.

Scanning Electron Microscopy and Energy Dispersive X-ray Spectroscopy (SEM-EDS)

The filler morphology, particle size and elemental composition of fucoidan added materials obtained by SEM-EDS analysis were investigated. Two sample from each group were coated with gold (Leica EM ACE200, Leica Microsystems, Washington DC, USA) and evaluated with scanning electron microscope (Hitachi SU5000 FE-SEM). The entire surface of the samples was scanned and photographs were obtained at 1000×, 5000×, and 10,000× magnification from areas showing the surface structure with high vacuum SEM operating at 1 Torr pressure and 10 kV.

Two sample from each group were subjected to energy dispersive X-ray spectroscopy (EDS) analysis to determine the surface chemistry and composition. An EDS detector with an acceleration voltage of 30 kV and a spot size of 5.5 (Oxford X-MaxN 80 model, EDS detector with 80 mm^2 detector area) was used. EDS analyses were performed on three separate points from samples, and the elemental distribution is reported as an average.

Fourier Transform Infrared Spectroscopy (FTIR)

For this analysis, two samples were prepared in each group. Fourier transform infrared spectroscopy (Jasco FT/IR 6X, JASCO Corporation, Tokyo, Japan) was used to study and analyze the functional groups in the samples before and after immersion in the wavenumber range from 400 cm^{-1} to 4000 cm^{-1}. An average of 32 scans was performed using an air background reference at a spectral resolution of 4 cm^{-1}. All spectra were analyzed using JASCO Spectra Manager software after background correction and normalization.

Statistical Analysis

IBM SPSS 27.0 (Armonk, NY, USA) package program was used for statistical evaluations. Kolmogorov-Smirnov test, Shapiro-Wilk test and graphical technique (Normal Q-Q Plot) were used to determine whether the data were normally distributed. One-way analysis of variance (ANOVA) followed by Tukey post-hoc test was used to compare differences between groups in contact angle values. A significance level of $p < 0.05$ was considered statistically significant.

Table 1. Properties of the materials used in the study

Material Type	Manufacturer	Polymerization	Bonding Agent	Content
MTA Plus	Prevest Detpro Limited, Jammu, India	Self cure	-	Powder: Calcium Oxide, Silicon Oxide, Bismuth Oxide Liquid: Hydrated Polymer Gel
Fuji IX LC	Fuji IX, GC, Tokyo, Japan			Powder: Alumino-silicate glass Liquid: Polyacrylic acid, water, polybasic carboxylic acid
Cention N	Ivoclar Vivadent, Liechtenstein	Self or Light Cure (20 sn)	Available/ Optional	Powder: calcium fluoro-silicate glass, barium glass, calcium-barium-aluminum fluoro-silicate glass, iso-fillers, ytterbium trifluoride, initiators and pigments. Liquid: dimethacrylates, initiators, stabilizers, additives and mint flavor.”
Material		**Purity**	**Water Solubility**	
Fucoidan powder	Henan Qianbaiguan Biotechnology Co., Ltd, China	%85	+	

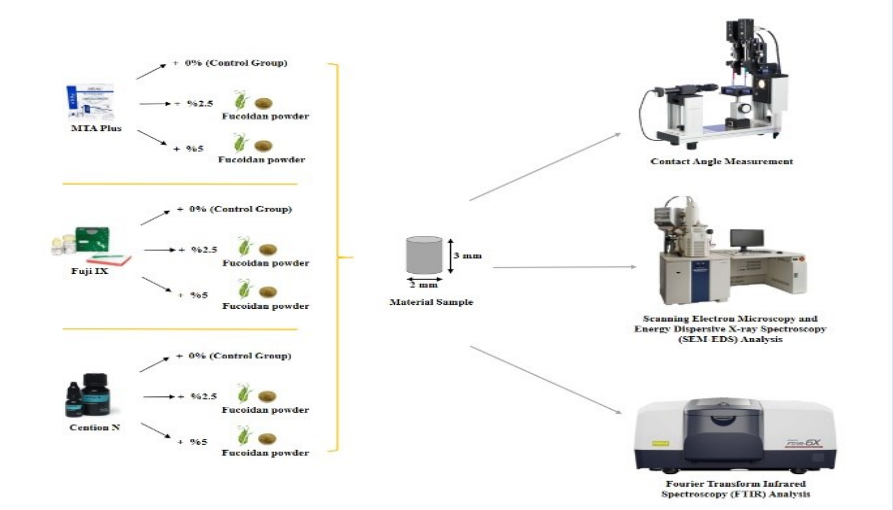


Figure 1. Study design.

Results

Contact Angle Analysis

The contact angle test was performed to evaluate the surface wettability of the fucoidan-modified restorative materials. In general, lower contact angles indicate higher surface wettability.

The results showed that the addition of fucoidan significantly influenced the contact angle values across all material types. In the MTA group, the control group exhibited a relatively low average contact angle of 17.13° ± 7.68, which increased to 29.13° ± 4.62 and 43.07°± 7.52 for the 2.5% and 5% fucoidan groups, respectively. This suggests a reduction in surface hydrophilicity with increased fucoidan content.

In contrast, the GIC group showed a decrease in contact angle with the addition of fucoidan up to 2.5%, followed by an increase at 5%. The control group had a mean contact angle of 47.73° ± 8.47**,** which dropped to 36.40° ± 15.22 at 2.5% fucoidan. However, the 5% fucoidan group exhibited a higher mean value of 41.83° ± 11.34**,** indicating a non-linear effect of fucoidan concentration on surface wettability.

AL groups demonstrated the most variable wettability behavior. The control group had a mean contact angle of 53.00° ± 8.24**,** which increased substantially to 65.63° ± 10.07 with 2.5% fucoidan. The 5% group maintained a similarly high contact angle of 64.50° ± 4.61, suggesting that fucoidan incorporation may lead to reduced wettability in resin-based systems, potentially due to incomplete dispersion or surface segregation of hydrophobic components.

As a result of post-hoc analyses, the MTA0 group showed statistically significant differences compared to several other groups. In particular, the contact angle values were found to be markedly higher when compared to the groups containing AL. Moreover, the AL2.5 group also demonstrated significant differences from both MTA- and GIC-based groups ($p < 0.05$). The groups exhibiting statistically significant differences are summarized in Table 2.

Fourier Transform Infrared Spectroscopy (FTIR)

FTIR analysis was performed to evaluate the chemical interactions and structural modifications resulting from the incorporation of fucoidan into the tested restorative materials. The spectra of all groups were recorded in the range of 400–4000 cm^{-1},[13,14] and the major absorption bands were compared among the control (0%) and fucoidan-modified (2.5% and 5%) groups for each base material. FTIR spectra of fucidan-modified dental materials are presented in Figure 2.

In the FTIR spectrum, a prominent band around 1020^{-1} was observed, indicating the C-O-C stretching vibrations of glycosidic linkages, consistent with carbohydrate backbone structures.[15,16] Additionally, the broader absorption in the 1190^{-1} region is attributed to overlapping C-O stretching modes from saccharide units and possible sulfation patterns, corroborating the polysaccharide nature of fucoidan.[17] In the FTIR spectrum, a broad absorption band observed around ~3300 cm^{-1} is attributed to O-H stretching vibrations, including contributions from bound water.[18]

The FTIR spectra of MTA samples containing 0%, 2.5%, and 5% fucoidan demonstrated distinct spectral variations indicating structural integration of the polysaccharide. Notably, characteristic absorption bands appeared around ~1160 cm^{-1} and ~1010 cm^{-1} in the fucoidan-containing groups, corresponding to sulfate ester (S=O) and glycosidic (C-O-C) vibrations, respectively, which were absent or weaker in the control group. Additionally, the broadening of the ~3400 cm^{-1} band suggests increased hydroxyl group content and hydrogen bonding with higher fucoidan concentrations.[11] These spectral differences confirm that fucoidan was successfully incorporated into the MTA matrix and induced chemical interactions at a molecular level.

GIC samples demonstrated strong peaks at ~1420 cm^{-1} (COO- stretching from polyacrylic acid salts), ~1040 cm^{-1} (Si-O stretching), and ~3400 cm^{-1} (O-H groups).[19] Upon fucoidan addition, new bands emerged at ~1230 cm^{-1} and ~1025 cm^{-1}, with gradually increasing intensity in the 2.5% and 5% groups. These shifts indicated potential chemical interactions between fucoidan and the polyacid component of GIC.

AL samples revealed typical polymeric and inorganic filler bands, including C=O stretching near 1720 cm^{-1} and Si-O bands around 1000-1100 cm^{-1}. After fucoidan incorporation, subtle changes were observed in the 1200–1250 cm^{-1} and 1000-1050

cm^{-1} regions, indicative of functional group overlap and possible weak hydrogen bonding or ionic interactions between the polymer matrix and fucoidan components. Overall, the FTIR spectra confirmed the successful incorporation of fucoidan into all three material types. The increase in characteristic carbohydrate and sulfate-related bands with rising fucoidan concentration provides molecular-level evidence of structural modification.

Table 2. Groups with statistically significant differences

Group 1	Group 2	p
0% Alkasite	0% MTA	p = 0.0040 < **0.05**
0% MTA	2.5% Alkasite	p = 0.0001 < **0.05**
0% MTA	5% Alkasite	p = 0.0002 < **0.05**
0% GIC	0% MTA	p = 0.0170 < **0.05**
2.5% Alkasite	2.5% GIC	p = 0.0246 < **0.05**
2.5% Alkasite	2.5% MTA	p = 0.0034 < **0.05**
2.5% GIC	5% Alkasite	p = 0.0332 < **0.05**

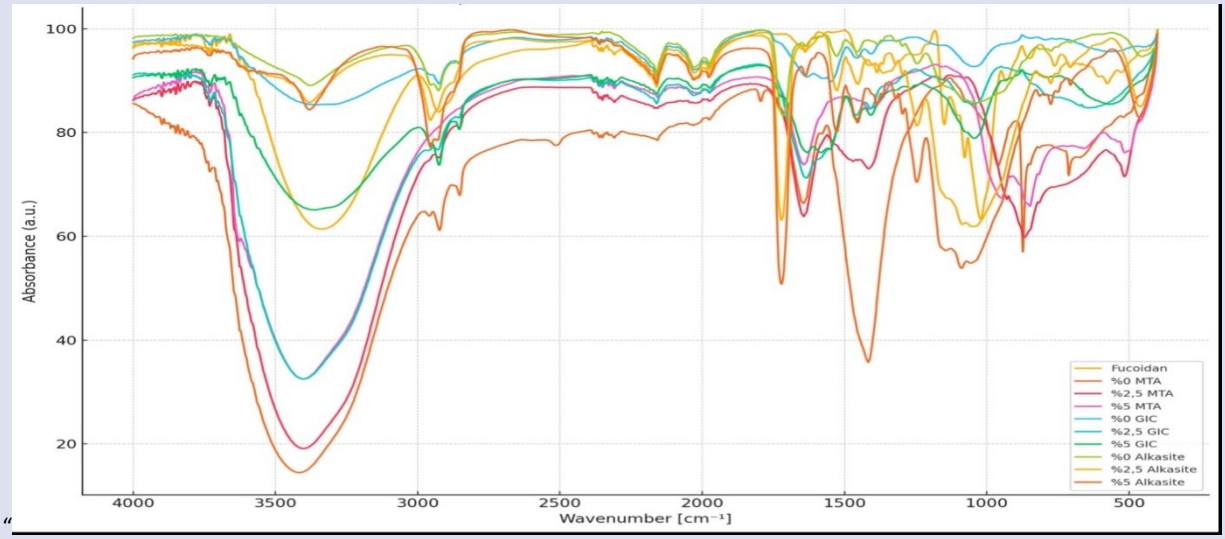


Figure 2. FT-IR spectra of fucoidan-incorporated dental materials

SEM-EDS Analysis

SEM images were taken at 1000×, 5000×, and 10000× magnifications, allowing for the assessment of both overall morphology and microstructural details. However, for clarity and understandability, images at 5000× magnification, which best facilitates interpretation of surface results, have been included in the article.

In the SEM images of the MTA0 group, typical MTA characteristics were observed as crystalline, needle-like structures, while in the MTA2.5 group, a fragmented, fibrous and irregular structure was observed in which the crystal structure was disrupted. In the MTA5 group, a granular, dense, lumpy structure, an amorphous and compact surface was observed. In terms of surface roughness, in the 0% MTA group, the roughness was high due to crystal protrusions, but in the MTA2.5 group, very high roughness was observed due to irregular and pointed structures, and in the MTA5 group, very high roughness was observed due to the granular, lumpy structure. In the MTA2.5 group, it was observed that fucoidan disrupted the crystal growth and the structure became more fibrous. In the MTA5 group, it was observed that the crystalline structure was completely disrupted and a compact amorphous structure emerged (Figure 3).

In the SEM images of the GIC0 group, irregular, amorphous porous areas were observed, while in the GIC2.5 group, a crystalline, scattered structure with dense particle protrusions was observed. In the GIC5 group, a more organized surface was observed with oval, capsule-like structures. In terms of surface roughness, in the GIC0 group, the roughness was high due to an irregular and wavy surface, but in the GIC2.5 group, very high roughness was observed due to sharp-edged structures, and in the GIC5 group, low surface roughness was observed due to more regular, oval structures. In the GIC2.5 group, it was observed that fucoidan disrupted the surface organization and formed crystalline structures. In the GIC5 group, it was observed that fucoidan reorganized the surface and provided a structural order with capsule-like structures (Figure 3).

In the SEM images of the AL0 group, an irregular, heterogeneous structure was observed, while a more regular, partially compact structure was observed in the AL2.5. A compact, dense and homogeneous structure was observed in the AL5. In terms of surface roughness, moderate roughness was observed in the 0% alkaloid group, while a decrease in roughness was observed in the AL2.5. A significantly reduced, smooth surface was observed in the AL5. It was observed that fucoidan showed partial integration with the matrix in the

AL2.5. In the AL5, it was observed that fucoidan provided stabilization of the structure with its high integration into the structure (Figure 3).

In MTA material, it was determined that Ca content was high in all groups and tended to increase with the addition of fucoidan and fucoidan did not disrupt the basic calcium silicate structure of MTA. It can be said that the Si ratio was the highest in the MTA2.5 group, a significant decrease was observed in this ratio in the MTA5 group and therefore fucoidan may reduce the visibility of silicates on the surface. Sulfur was not detected in any group (Figure 4).

In GIC material, 2.5% fucoidan addition increased the glass phase ratio in the glass ionomer structure, and especially Al, Si, Sr ions became dominant on the surface. Sulfur was not detected. The preservation or increase in fluorine content shows that the material can maintain its anticariogenic potential (Figure 4).

In AL material, the carbon ratio decreased significantly as the fucoidan addition increased. This situation suggests that the polymer matrix is structurally different with the fucoidan addition. As fucoidan addition increases, the increase in silicon ratio shows that the glass content becomes more pronounced. It was observed that the fluorine ratio of the material almost doubled with fucoidan addition (Figure 4).

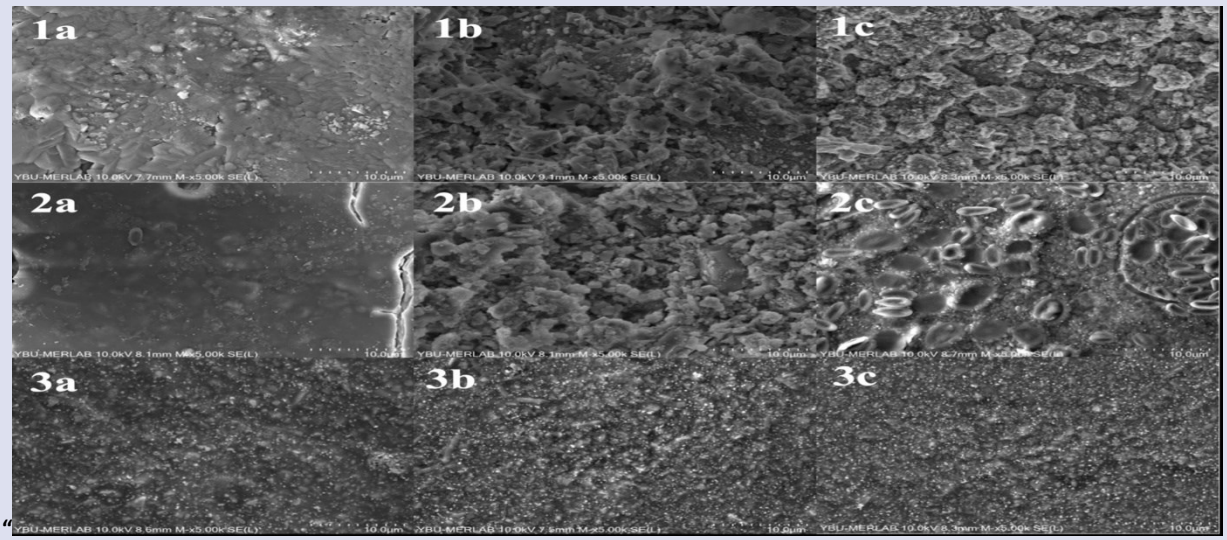


Figure 3. SEM images of samples at 5000x magnification

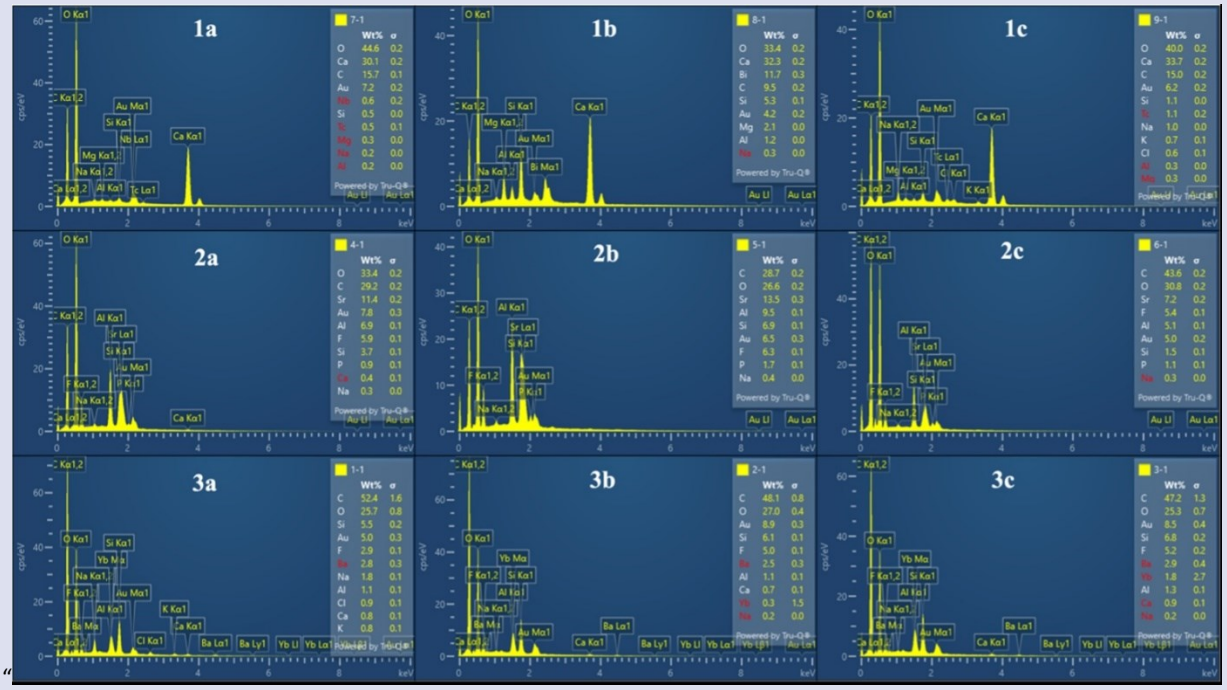


Figure 4. EDS analysis graphs of samples

Discussion

This study aimed to investigate the structural and surface modifications that occur when fucoidan, a high molecular weight sulfated polysaccharide, is incorporated into three commonly used dental materials. Comprehensive analytical techniques, including FTIR spectroscopy, SEM-EDS analysis, and contact angle measurements, were employed to assess the chemical interactions, morphological alterations, and wettability behavior of the modified materials. In this study, the null hypothesis that the incorporation of fucoidan-a high molecular weight sulfated polysaccharide derived from brown macroalgae-into MTA, AL, and GIC does not significantly alter surface chemistry and wettability was rejected.

Contact angle measurement in dentistry is typically performed using the sessile drop technique, where a small volume (e.g., ~3 µL) of liquid-commonly water-is deposited on the material surface and imaged to determine the angle between the liquid–solid and liquid–air interfaces using a goniometer or drop-profile image analysis system. Clinically, contact angles below 90° indicate hydrophilicity and favorable wetting-promoting adhesive bonding or irrigant penetration-while angles above 90° suggest hydrophobicity, which may hinder material interaction or increase microbial adhesion.[20,21]

Contact angle measurements revealed a dose-dependent increase in hydrophilicity for MTA: from 17.1° (±7.3) in the control to 43.1° (±7.8) at 5 wt%, indicating enhanced wettability critical for improved adhesion and hydration kinetics. Similar trends were observed for AL and GIC, consistent with known surface-energy theory stating that lower contact angles promote better wetting and material integration.[11,22,23]
The contact angle values indicate that the effect of fucoidan on wettability is material-dependent and likely influenced by matrix composition, surface energy, and fucoidan distribution. The increased contact angle observed in MTA and AL may reflect hydrophobic surface presentation or phase separation of fucoidan chains, while GIC's initial improvement in wettability at 2.5% may stem from improved dispersion or hydrogen bonding at moderate concentrations.
Fucoidan's role in enhancing wettability aligns with prior findings in polymeric and cementitious systems. For instance, in chitosan-fucoidan hydrogels, increased wettability due to sulfated groups promoted cell interaction and material absorption.[24,25] Likewise, studies on MTA-fucoidan composites demonstrated accelerated hydration and setting behavior with improved handling, correlating with increased surface hydrophilicity measured by contact angle assessment.[11]

FTIR spectroscopy confirmed the successful integration of fucoidan into all cement matrices. Characteristic sulfate stretching bands (~820–850 cm^{-1}) were present, matching prior observations in alginate-fucoidan scaffolds.[24,26] These bands indicate preserved sulfate functionalization, which likely interacts with calcium or aluminum ions in the cement matrix, contributing to ionic cross-linking and matrix cohesion without disrupting key cement phases.

As SEM analysis revealed, increasing fucoidan concentration disrupted the crystalline morphology across all materials, shifting from well-defined needle-like or porous structures to more irregular, fibrous, granular, or amorphous surfaces. This structural transformation is consistent with findings by Kim *et al.,*[11] who reported that fucoidan addition to MTA disrupted crystal growth and modified surface roughness while maintaining calcium silicate integrity. Similarly, studies on composite scaffolds have shown that fucoidan integration results in altered micro-morphologies-such as fibrous or dense amorphous structures-while enhancing compositional stability, particularly through increased calcium and silicate content.[27] 2.5% fucoidan induced fibrous disruptions in MTA and crystalline formation in GIC, while 5% drove a transition toward compact amorphous or capsule-like structures, correlating with observed high roughness in MTA/GIC and smooth, homogenous surfaces in AL.

According to the EDS analysis results, the elements found in the highest amounts in MTA groups are oxygen and calcium, but carbon is also found in high amounts. In other materials, the elements found in the highest amounts are oxygen and carbon. However, in the alkaside material; as the addition of fucoidan increased, the carbon ratio decreased significantly. This situation shows that the polymer matrix is structurally different with the addition of fucoidan. The fact that the fluoride ratio of the material increased significantly with the addition of fucoidan is a very important finding in terms of preventive dentistry applications. In addition, preserving or increasing the fluoride content with the addition of fucoidan in glass ionomer material is very important in terms of showing that the material can maintain its anticariogenic potential.

Combining these analytical results, we propose that fucoidan enhances material wettability via two mechanisms: 1) chemical-sulfate-mediated ionic interactions improving surface energy and hydrophilicity; 2) physical-micro-roughness from polysaccharide inclusion creating a capillary network that further supports spreading. The net effect is a more hydrophilic surface favourable for clinical applications, especially in dental scenarios where intimate material-tooth or material-tissue interfaces are essential.

Despite these favorable surface characteristics and previously reported enhanced setting times and bioactivity in MTA-fucoidan systems, mechanical integrity-particularly compressive strength-has been shown to decrease at higher fucoidan concentrations (e.g., 5 wt%) in similar studies.[11] Therefore, while our data indicate improved surface wettability, future work should evaluate the trade-off between hydrophilicity and mechanical performance, exploring an optimal fucoidan range.

This study focused on evaluating the surface effects of fucoidan addition on MTA, glass ionomer cement (GIC), and Alkasite (AL) materials. However, some limitations should be considered: Only contact angle, FTIR, and

SEM/EDS analyses were performed in the study; mechanical properties (compressive, flexural strength, microhardness), solubility, setting time, and biological evaluations were excluded. Therefore, the results represent changes at the surface and chemical levels only. Contact angle measurements were performed with deionized water. This approach is suitable for comparison, but does not fully reflect the clinical environment. More realistic conditions should be created using artificial saliva or biological fluids in the future.

Conclusions

- Surface wettability improved significantly across MTA, AL, and GIC after fucoidan addition, as shown by lower contact angles.
- FTIR sulfate peaks and SEM-EDS mapping confirm fucoidan presence without matrix disruption.

Increased micro-roughness and preserved sulfate groups appear to act synergistically to enhance wettability.

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None.

Conflicts of Interest Statement

The authors declare no conflict of interest.

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