Multifunctional Characterization of Fucoidan: Structural Insights and Efficient Removal of Toxic Metal Ions

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Abstract: In this study, commercial fucoidan was subjected to comprehensive physicochemical and structural characterization to evaluate its potential for removing heavy metal ions from aqueous media. Fourier transform infrared spectroscopy (FTIR) and proton nuclear magnetic resonance (1H NMR) confirmed the presence of sulfated heterofucans, with distinct signals corresponding to carboxyl, sulfate, and sugar ring functionalities. Thermogravimetric analysis (TGA) revealed notable thermal stability, with major degradation occurring above 240 °C. Interaction mechanisms have been proposed on the basis of functional groups, particularly sulfates and carboxylates. Finally, the efficacy of fucoidan for Ni(II), Pb(II), and Cu(II) removal was validated via inductively coupled plasma (ICP) spectroscopy, which revealed removal rates of up to 99.89% for Cu and 99.84% for Pb. These findings highlight the dual role of fucoidan as both a bioactive polymer and a promising biopolymer for wastewater remediation.

Keywords: Fucoidan, Brown algae polysaccharide, FTIR, 1H NMR, Thermogravimetric analysis, Heavy metal removal, Biosorption, Sulfated heterofucans.

1. INTRODUCTION

Marine-derived polysaccharides, particularly fucoidans extracted from brown algae, have garnered significant attention because of their complex structures and versatile bioactivities. Fucoidans are typically composed of α-L-fucose units with varying degrees of sulfation and branching, contributing to their biological effects, such as anticoagulant, antioxidant, and metal-chelating properties [1-3]. These sulfated polysaccharides are known for their structural diversity, which is influenced by the algal species, geographic origin, seasonal factors, and extraction procedures. Such variability underscores the need for thorough characterization when assessing their functional potential.

The structural complexity of fucoidans depends on their algal source, extraction method, and postprocessing. Techniques such as FTIR and NMR spectroscopy have proven essential in elucidating the sulfation pattern and sugar backbone [4,5]. For example, sulfate groups identified by characteristic vibrational bands (S=O) and NMR shifts serve as critical biomarkers of bioactivity [6,7]. These structural features are directly associated with the biological performance of a polymer [8,9], including its anti-inflammatory, antiviral, and cytoprotective properties [10-12]. In addition to biomedical applications, the ability of fucoidans to interact with metallic ions has opened avenues in environmental science, particularly in the removal of hazardous pollutants from aqueous media.

Recent studies have also shown that the affinity of fucoidans for divalent cations such as Pb(II), Cu(II), and Ni(II) can be leveraged for environmental remediation [8,9]. The high density of functional groups, especially sulfates and carboxyls, enables these polymers to bind and remove heavy metals from contaminated aqueous systems. This chelation capability is not only valuable for detoxification but also suggests potential use in water treatment technologies that are biocompatible, renewable, and environmentally friendly.

Despite the widespread availability of commercial fucoidan products, little is known about their detailed structure–function relationships, particularly in relation to their physicochemical behavior and sorptive capacity. Moreover, there remains a critical gap in the application of these insights to regionally abundant algal biomass, which could serve as a raw material for cost-effective bioremediation solutions. In particular, brown seaweeds such as *Sargassum* spp., which proliferate along the coasts of Mexico and other tropical regions, present a unique opportunity for sustainable development.

The novelty of this study lies in evaluating the potential of reference fucoidan as a model for future applications based on *Sargassum* spp., a genus of

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*Address correspondence to this author at the Faculty of Chemical Sciences and Engineering, Autonomous University of Baja California, Baja California, Tijuana, 22424, México; E-mail: elopez92@uabc.edu.mx brown algae that has become a significant environmental concern owing to its excessive accumulation along Mexican shorelines. These massive blooms, driven by climate change and nutrient pollution, lead to serious ecological and socioeconomic impacts, including beach degradation, marine habitat disruption, and tourism losses. However, *Sargassum* is also a rich source of sulfated polysaccharides similar in structure to commercial fucoidans, suggesting a promising opportunity for valorization. Its high content of functional groups, particularly sulfates and carboxyls, could be harnessed for the removal of heavy metals from polluted water, offering the dual benefit of mitigating environmental nuisance while contributing to water sanitation.

The present study addresses this gap by providing an integrated spectroscopic and thermal analysis of a commercial fucoidan sample. Moreover, this work evaluated its capacity to remove hazardous metal ions under simple experimental conditions. This dual characterization approach contributes to a better understanding of how structural attributes influence practical performance, offering insights relevant for both biomedical and environmental applications of marine biopolymers. Ultimately, this research lays the groundwork for translating these findings to native macroalgal biomass such as *Sargassum*, promoting innovative, low-cost solutions for coastal pollution management and resource recovery.

2. EXPERIMENTAL

2.1. Materials

Commercial fucoidan was used as received. Solutions of Ni(II), Cu(II), and Pb(II) at 100 ppm were prepared using analytical grade salts. All the experiments were conducted with deionized water under ambient laboratory conditions.

2.2. Fourier Transform Infrared Spectroscopy (FTIR)

The FTIR spectrum was recorded to identify key functional groups in the polymer. The samples were analyzed in KBr pellets, and spectra were obtained in the range 4000-400 cm⁻¹.

2.3. Proton Nuclear Magnetic Resonance (^{1}H-NMR)

^{1}H-NMR was performed using a 400 MHz instrument with the sample dissolved in D_2O. The spectral range spanned 0-6 ppm to capture sugar-ring and methyl signals indicative of sulfation patterns.

2.4. Thermogravimetric Analysis (TGA)

Thermal degradation was assessed under a nitrogen atmosphere. The samples were heated from room temperature to 750 °C at a rate of 20 °C/min to identify the degradation stages and thermal stability.

2.5. Cation Removal Assay

First, 10 mg of unmodified, untreated commercial fucoidan was weighed and added to a vial containing 10 mL of M^{2+} solution in water (Cu, Ni, or Pb), with the pH adjusted to 5 and 100 ppm for each metal ion. The mixture was stirred with a magnetic stir bar for 10 min, after which it was stirred and allowed to rest for 24 h. The mixture was filtered, and the residual metal ion concentrations in the supernatant were determined via ICP spectroscopy.

3. RESULTS AND DISCUSSION

3.1. Structural Analysis by FTIR

In the FTIR spectrum of fucoidan, the intense bands at approximately 1700-1600 cm⁻¹ are attributed to the stretching vibration of the carboxyl or acetyl group bonds (C=O) due to the presence of uronic acids, mainly from guluronic and mannuronic acid residues [1-3]. The bands observed at approximately 1400 cm⁻¹ are caused by the asymmetric overlap between the symmetric CH_2 deformation of galactose and the asymmetric CH_3 deformation of fucose [4]. The absorption bands at approximately 1270-1220 cm⁻¹ are related to the asymmetric stretching vibration of the sulfate group (S=O), a characteristic component of fucoidans [5,6]. This is further supported by the presence of an additional symmetric stretching vibration band of the sulfate group at approximately 600-570 cm⁻¹. The bands at approximately 1080 and 1020 cm⁻¹ correspond to symmetric stretching

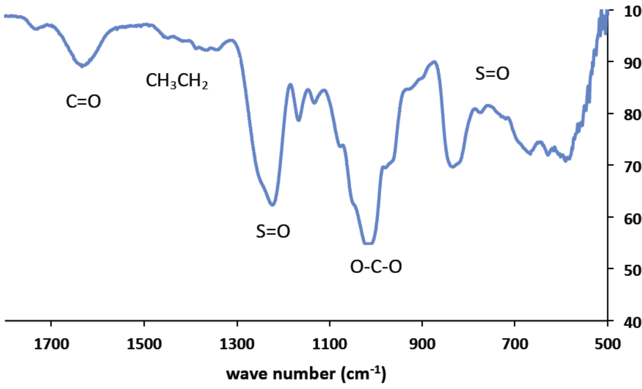

Figure 1: FTIR spectrum of commercial fucoidan.

vibrations characteristic of the hemiacetal linkage in the sugar ring (C-O-C); thus, more than one absorption peak is observed, confirming the presence of more than one type of sugar. These findings indicate that the spectrum corresponds to sulfated heterofucans. The position of the sulfate groups plays an important role in the biological activity of fucoidans.

Algal samples are generally subjected to separation processes via chromatography. The commercial fucoidan sample analyzed presented infrared signals similar to those obtained in the work of Garcia-Zamora *et al*. [7]. in the fraction obtained at room temperature (F1). The observed signals are attributed to sulfated heterofucans with anticoagulant activity, as reported for fucalean species such as Fucus vesiculosus, Sargassum tenerrimum, S. wightii, Turbinaria conoides, *T. ornata*, and Padina tetrastromatica [8-12].

3.2. Proton NMR Spectroscopy

In the ¹H NMR spectrum, the multiplicity of the signals is determined by the diversity in the positioning of glycosidic linkages and the substitution pattern in the polymer, as previously reported for fucoidans from brown algae [5, 8, 13-15]. The obtained spectrum presents some differences; however, it was possible to identify representative signals that confirm the presence of sulfated α-L-fucose residues, with characteristic α-proton signals observed between 5.1-5.5 ppm and signals between 1.25-1.5 ppm assigned to the methyl group of fucose [5], complementing the information obtained from the FT-IR spectra. Anomeric signals are observed at 4.9 ppm, and methylene signals appear at 3.6 ppm, corresponding to β-D-galactose residues [8,9,16,17]. The signals between 3.6-4.4 ppm correspond to the H2 to H5 protons of the pyranoid ring, where changes in the signal multiplicity suggest the presence of different families of disulfated and monosulfated monomer units [5, 14]. Here, the signals shifted to 4.4 ppm indicate the presence of sulfate residues at C-3; this is consistent with what has been reported for anticoagulant fucoidans sulfated at C-2 and C-3 of the sugar ring, as predicted from the FT-IR spectra in this study [18,19]. The signals at 4.6 ppm correspond to uronate residues, and the presence of protein residues is confirmed by the signals at 1.5 and 2.0 ppm [5,8].

Using the structural information obtained, the commercial fucoidan molecule is expected to contain units similar to those proposed in Figure **3**, with galactose-derived groups, a ring derived from Cladosiphon okamuranus (with an acidic group), a ring

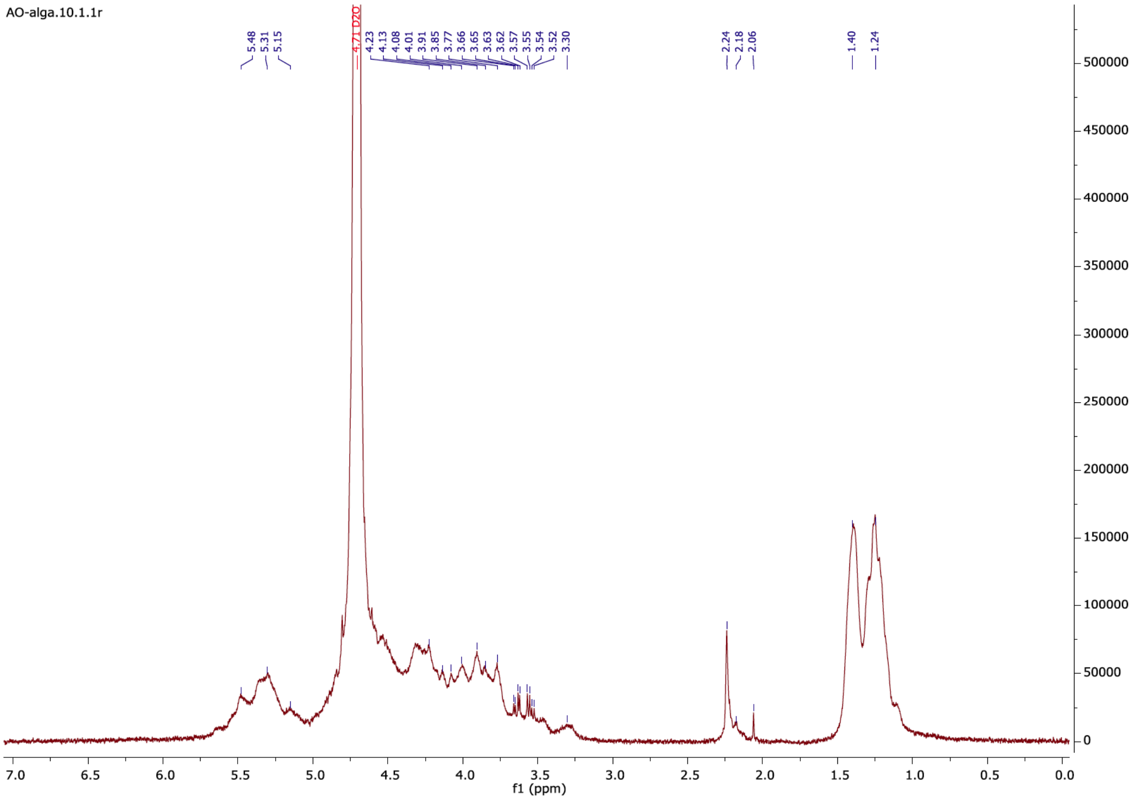

Figure 2: 400 MHz RMN ^{1}H of fucoidan in D_2O.

Figure 3: Fucoidan structure proposed from the spectroscopic characterization.

present in the heterofucan from Fucus vesiculosus (with sulfate groups), and a ring found in most heterofucans containing methyl and hydroxyl groups.

3.3. Thermogravimetric Analysis

Thermogravimetric analysis was performed from room temperature to 750°C with an increase in temperature of 20°C/min in a N_2 atmosphere. In the thermogravimetric analysis, initial weight loss was observed at 73 °C, corresponding to the desorption of water present in the molecule. The main decomposition of the compound subsequently occurs at 241 °C, with a weight loss of approximately 30% of the molecular mass. Decomposition continues above 700 °C, where a carbon residue of approximately 24% is observed.

3.5. Metal Ion Removal Performance

The predominant interaction at different pH values is expected to occur with the carboxylate and sulfate groups present in the C. closiphon and F. vesiculosus fractions, as these groups remain deprotonated over a wide pH range. To a lesser extent, supramolecular interactions may also occur with the hydroxyl groups of various rings, especially under alkaline conditions.

3.5.1. Cation Removal Analysis by ICP (Ionized Coupled Plasma)

The experiments were performed at room temperature (25°C) with an initial pH of 5. For the removal assays, initial fucoidan concentrations of 1 mg/mL were used in 10 mL vials containing 100 ppm contaminant cations (M^{2+}): Ni, Pb, and Cu. The solutions were stirred for 10 minutes and then left to stand for 24 hours to promote flocculation. The solid was filtered, and the supernatant was analyzed via ICP, which revealed high removal percentages of Pb and Cu (Table **1**), reaching 99.84% and 99.89%, respectively. The pH of the solution was not significantly affected at the end of the measurement period (±0.2 pH units). The removal rates of Ni ranged from 32.34% to 17.2%. Measurements were performed in triplicate, and owing to the nature of the material—which may contain different fractions with varying structures—the results show variations in removal percentages of up to 12.6% for Pb (99.84-87.24%),

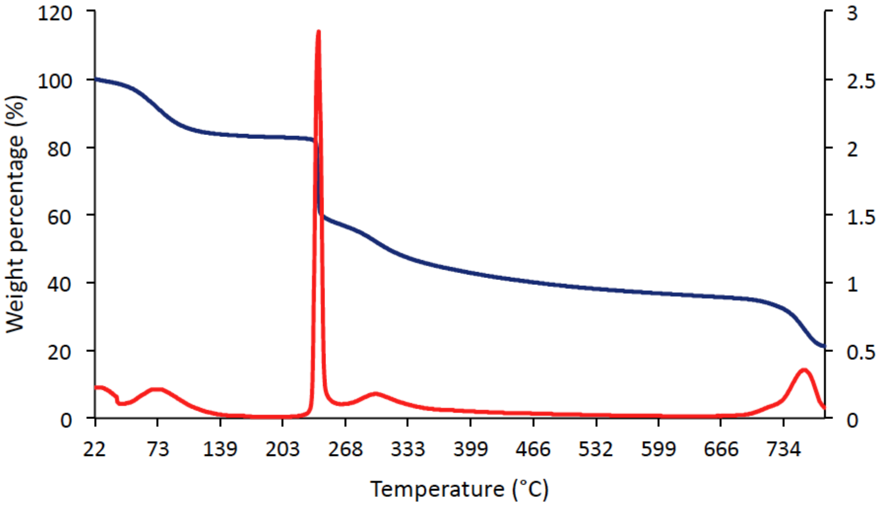

Figure 4: Thermogravimetric analysis of fucoidan at 20°C/min in a N_2 atmosphere.

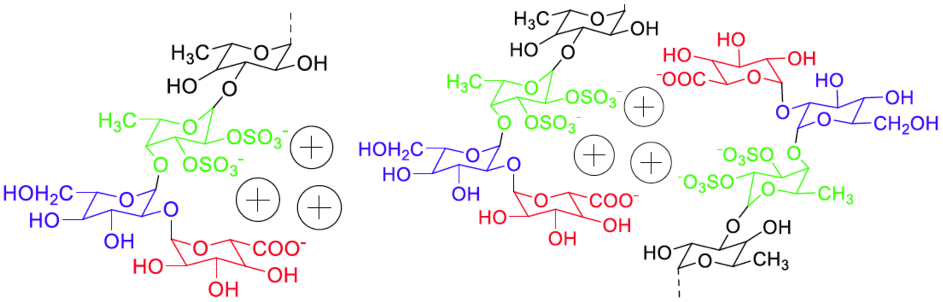

Figure 5: Schematic representation of fucoidan and its possible interactions with cations as a function of pH.

Table 1: Percentage of Removal Measured with ICP

% of removal	Measurement 1	Measurement 2	Measurement 3	Mean +SD
Ni	17.20	25.40	32.34	7.6
Pb	99.84	91.76	87.24	6.4
Cu	99.89	96.54	95.91	2.1

3.98% for Cu (99.89-95.91%), and 15.14% for Ni (32.34-17.2%). Despite these variations, the removal values obtained for Pb and Cu are high and offer a natural alternative for metal removal in aqueous effluents. The results obtained are consistent with previous reports that Cu^{2+} and Pb^{2+} have a larger ionic radius and are heavier metals than Ni^{2+}, which favors the formation of more stable, less soluble precipitates and a faster rate of aggregate formation, allowing for the removal of contaminants. [20] Furthermore, interactions with sulfates are more favorable for Pb^{2+} than for Cu^{2+} and Ni^{2+}; all of these interactions have the ability to interact with carboxylates, so interactions with sulfates should be the most predominant. Mangal *et al*. [21] reported that Cu^{2+} is present mainly in the particulate and colloidal fractions. Coagulation significantly reduces its bioavailability, although some Cu remains bound to dissolved organic matter, limiting its complete removal. Pb^{2+} is found mainly in solid particles (>70% in the particulate phase before treatment), which explains its high removal efficiency with coagulants. After treatment, its bioavailability was nearly zero. Moreover, Ni^{2+} is present mainly in the dissolved fraction, which reduces the effectiveness of the flocculation treatment. Only a fraction was removed, forming precipitates or being entrained with large flocs. The results are very similar to those obtained in this work.

Most reusable materials require acid treatment to release the metal from the matrix, allowing it to be reused. However, owing to the nature of fucoidan, it

Table 2: Comparison of Nickel Removal with Algae at Different pH Values

Material	pH	qmax ($mmolg^{-1}$)	reference
Sargassum. Sp	5.0	0.913	[22]
Ca-alginate	5.0	1.221	[22]
Sargassum. Sp.	5.5	0.61	[23]
S. filipendula	4.0	0.857	[24]
S.wightii	4.0	1.383	[25]
Ca-alginate	4.5	0.71-1.21	[26]
Ulva sp.	5.5	0.29	[27]
Codium vermilara	6.0	0.22	[28]
Spirogyra insignis	6.0	0.29	[28]
Ulva lactuca	4.5	1.14	[29]
This work	5.0	0.293-0.55	-

Table 3: Comparison of Copper Removal with Algae Removal at Different pH Values

Material	pH	qmax (mmolg^{-1})	reference
Sargassum. Sp	5.0	1.483	[22]
Ca-alginate	5.0	1.863	[22]
Sargassum. Sp.	5.0	1.21	[23]
S.Filipendula	4.5	0.99	[26]
Ca-alginate	4.5	1.13-1.25	[26]
Ulva sp.	5.0	0.75	[27]
Codium vermilara	5.0	0.26	[27]
Spirogyra insignis	4.0	0.22	[28]
Spirogyra neglecta	4.5	1.80	[28]
Ulva fasciata	5.5	1.14	[30]
This work	5.0	1.46-1.52	-

Table 4: Comparison of Lead Removal with Algae at Different pH Values

Material	pH	qmax (mmolg^{-1})	reference
Ascophyllum nodosum	3.5	1.31	[31]
Fucus veciculosus	3.5	1.11	[31]
Sargassum vulgare	3.5	1.10	[31]
Sargassum hystrix	4.5	1.37	[32]
Sargassum natans	4.5	1.14	[32]
Padina pavonia	4.5	1.04	[32]
Fucus spiralis	3.0	0.98	[33]
Ulva Lactuca	4.5	0.61	[32]
Caldophora glomerata	4.5	0.35	[32]
Ulva sp.	5.0	1.45	[28]
This work	5.0	1.26-1.44	-

can degrade or change its structure under acidic conditions, making reuse of the materials impossible. Algae are materials with high natural availability that have shown promising results in removing heavy metals from water for treatment, influenced mainly by the functional groups present and the pH of the medium.

4. CONCLUSIONS

The commercial fucoidan evaluated herein has structural characteristics consistent with those of sulfated heterofucans, as confirmed by FTIR and ^{1}H-NMR spectroscopy. Thermogravimetric analysis revealed moderate thermal stability, whereas the functional groups of the polymer facilitate strong binding with divalent metal cations. The high removal efficiencies of Pb(II) and Cu(II) affirm their potential as biopolymers for water treatment applications. Future work should explore its scalability, recyclability, and selectivity in complex effluent matrices.

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Received on 17-04-2025 Accepted on 14-05-2025 Published on 14-06-2025

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