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Biocomposite Films Based on Carrageenan and Graphite: Elaboration and Characterization

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Abstract: This work focuses on the development and characterization of novel biodegradable composite materials combining carrageenan matrices with graphite reinforcement. Graphite, a material of significant contemporary interest due to its exceptional electrical conductivity, thermal stability, and mechanical strength, was exfoliated using an optimized mechanical ultrasound method in aqueous medium to produce few-layer graphene sheets with minimal defects. The exfoliation parameters, including sonication time (2 h), amplitude (80 %), and temperature control (maintained below 40 °C), were carefully calibrated to ensure reproducible results. Carrageenans, sustainable biopolymers, were extracted from two red seaweed species: *Kappaphycus alvarezii* (Cottonii) and *Eucheuma denticulatum* (Spinosum), collected from coastal regions of Madagascar. The extraction protocol involved sequential steps of washing, alkaline treatment, filtration, precipitation with isopropanol, and final drying, yielding high-purity κ- and ι-carrageenan types respectively. Composite films were fabricated using solution mixing and casting-evaporation techniques, involving dissolution of carrageenan in distilled water at 80 °C and homogeneous dispersion of exfoliated graphite via probe sonication. The mixtures were then cast onto glass plates and dried under controlled conditions (25 °C, 50 % RH) for 48 h, producing uniform films with thicknesses ranging from 80 µm to 120 µm. The resulting bio(nano)composite films were systematically characterized using Thermogravimetric Analysis (TGA), X-ray Diffraction (XRD), and Fourier Transform Infrared Spectroscopy (FT-IR). TGA revealed a significant increase in degradation temperature from 242 °C for pure Carr-Co to 278 °C for composites with 2 % filler, representing a 36 °C improvement in thermal stability. XRD analysis showed characteristic graphite peaks at $2\theta = 26.53°$ with d-spacing values of approximately 0.34 nm, confirming the preservation of crystalline structure after processing. FT-IR spectra confirmed successful integration of graphite within the carrageenan matrix through observed band shifts and reduced hydroxyl stretching intensities. These promising results highlight the potential of graphite-reinforced carrageenan composites for advanced applications including sustainable packaging materials with enhanced barrier properties, biomedical scaffolds for tissue engineering with improved structural integrity, and biodegradable electronics with tunable conductivity. Future work will focus on comprehensive mechanical property evaluation using dynamic mechanical analysis, detailed biodegradability studies in simulated environmental conditions, systematic investigation of water vapor permeability, and preliminary scale-up feasibility assessment for industrial production. Additionally, the antimicrobial properties and cytotoxicity profiles of these composites will be explored to broaden their applicability in medical and food packaging sectors.

Keywords: Red Seaweed, Carrageenan, Graphite, Graphene, Casting-evaporation, Bio(nano)composites

1. Introduction

In the current context, we are witnessing a period of rapid and profound change affecting all aspects of society. This transition, characterised by technological advances and increased energy consumption, has given rise to a number of challenges that threaten the integrity of our environment. In response to these concerns, researchers and industry leaders are working to identify more efficient materials that can meet both market demands and environmental commitments [1,2]. This strategy consists of substituting more destructive materials with other, less degrading alternatives, without harming the environment [3]. In Toliara, there are many sources of materials that can be used as reinforcements, including graphite, to expand the areas where seaweed can be used [4]. The bonds within the graphite planes are covalent, while the inter-planar bonds are of the Van der Waals type (weak bonds), which makes graphite easy to exfoliate [5, 6]. An isolated sheet of graphite is called Graphene, which was first isolated(mechanical exfoliation) in 2004 by Andre Geim and Konstantin Novoselov [7]. Over the past twenty years, with its exceptional properties in a single material, graphene has a strong, hard quality that can be used in bionanocomposites [8-10].

Since 1989, Madagascar has been among the countries cultivating and producing red seaweed such as Cottonii and Spinosum, cultivating mainly the territorial zone and the South-West region, and continues to this day with the support of the University of Toliara and international partners join forces to make village aquaculture a real lever for sustainable development [11]. Global production of marine macro-seaweed has more than tripled since the turn of the millennium, rising from 10.6 million tonnes in 2000 to 32.4 million tonnes, with Madagascar producing 9.665 tonnes in 2019 [12] . These red seaweed are among the algae with high levels of polymers after extraction also known as carrageenans [13,14]. Their main structural differences lie in the position and number of sulfate groups, and the presence or absence of the 3,6 anhydrogalactose(DA) unit [15]. A distinction is made between κ-carrageenan (22% sulfate), ι-carrageenan (32% sulfate) and λ-carrageenans (38% sulfate) [16]. κ-carrageenan is soluble in water, but insoluble in organic solvents and oils. The ι-carrageenanhas is partially soluble when cold and totally soluble when hot [17]. The aim of this work is to exploit carrageenans extracted from seaweed by developing new composite materials. It aims to develop high performance bio(nano)composite films using the casting-evaporation method, to study their morphological and structural properties, and to help evaluate the potential benefits of these materials.

2. Materials and Methods

The raw materials used in this work were red seaweed: Kappaphycus alvarezii or Cottonii and Eucheuma denticulatum or Spinosum, donated by Ocean Farmers. The graphite powder used in this work was received from Fotadrevo deposit, Ampanihy District, Atsimo Andrefana Region (South coasts of Madagascar), Toliara.

As illustrated in Figure 1, a specific strategy was employed to produce the bionanocomposite film under investigation.

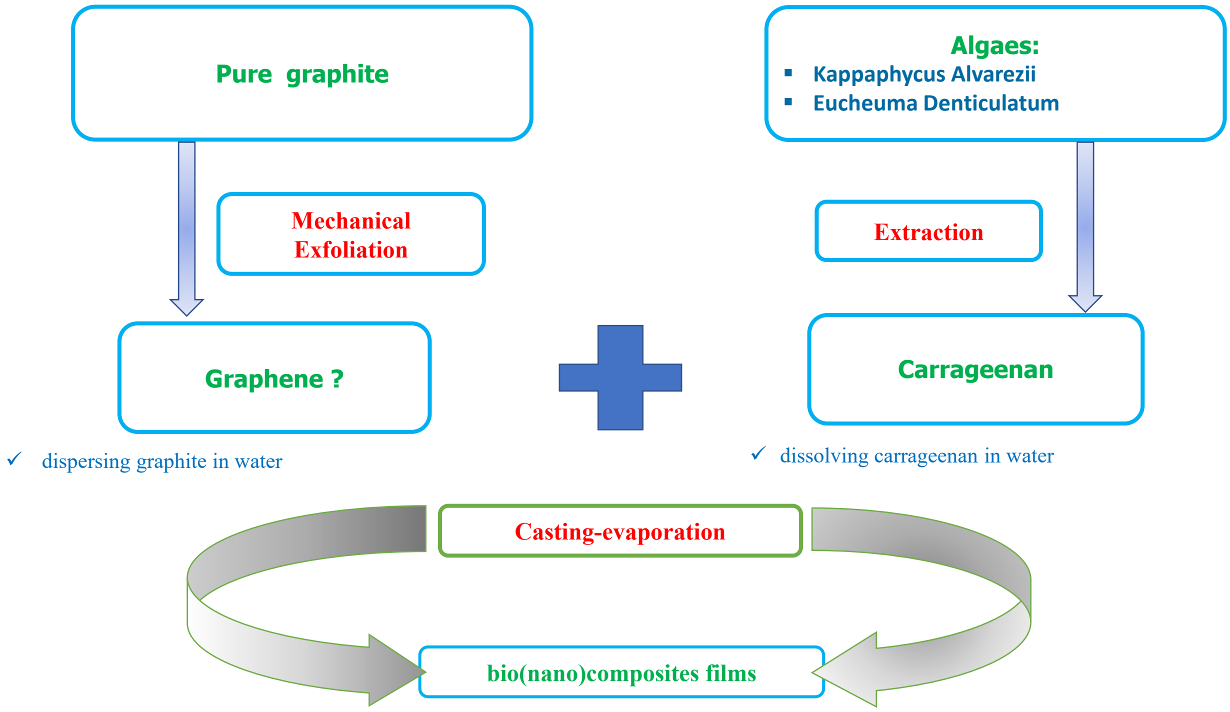


Figure 1. *Strategy for preparing bio(nano)composite films.*

2.1. Extraction of Carrageenan

The 5 grams of dry seaweed cut into small pieces were tremerized for 12 hours in a beaker containing 200 ml (milliliter) of distilled water. After 12 hours of rehydration, the distilled water used was now replaced by 40 ml of Sodium Hydroxide solution (N$_a$OH) and 160 mL of distilled water. At the same time, the Memmert-type thermostatic bath was heated to 90°C to accommodate the prepared samples. When the required temperature (90°C) was reached, the beakers were placed in the bath for 4 hours. After 4 hours of heating, the samples are removed from the Marie bath and sieved through a 125 μm fine mesh filter. After sieving, 500 ml of 90° ethanol was added to the filtrate to precipitate the carrageenans. This step separated the desired carrageenans from the rest of the samples. Alcohol-insoluble carrageenans were manually collected in Petri dishes using a spatula. The precipitates were then recovered using a sieve and stored in petri dishes. Finally, leave the carrageenans to dry in the sun for at least 72 hours [18].

2.2. Film Production Using the Casting-evaporation Technique

Firstly, the quantity of graphite powder (0.05 and 0.1 grams, i.e. 1 and 2%, respectively) was dissolved in 200 ml of distilled water. Concurrently, the ultrasonic bath was heated to 80°C in order to receive the prepared graphite solution.Subsequent to the graphite being in solution, exfoliation could begin. This was achieved by subjecting the graphite solution to 60 minutes of sonication in an ultrasonic bath, followed by shearing every 10 minutes using an immersion blender.The application of shear (extrusion) ensures exfoliation by progressively breaking or detaching the graphite sheets. The intensity of the shear then encourages the polymer chains to intercalate in the interfoliar space, exerting local stresses that promote the exfoliation mechanism. The solution mixing of graphite and carrageenan used in this work is made possible by the ability to disperse graphite and dissolve carrageenan in water. Dispersing fine powders in water with agitation and ultrasound can transform graphite into graphene. The mixture of polymer and graphite was prepared at different percentages (by mass) using the solution mixing method followed by the casting-evaporation approach. All blends are prepared with 75% carrageenan and 25% glycerol, with or without filler (by mass). While the graphite is being ultrasonically exfoliated, the carrageenan (3.75 grams, 75%) is dissolved in a beaker containing 100 ml of distilled water under magnetic stirring at a temperature of 40-60°C with the glycerol (1.25 grams, 25%). Following sonication, the carrageenan and glycerol were mixed and poured into the graphite suspension. The resulting mixture was then heated to 80°C and stirred for 45 minutes to homogenise the solution and ensure good dispersion of the graphite in the matrix (carrageenan). Finally, the resulting mixture was spread onto plastic supports (Petri dishes), which were placed in air at room temperature to evaporate the water. During evaporation and drying, the Petri dishes were covered with parchment paper to avoid any effect of evaporation on the mixture and then left at room temperature for 4 to 5 days to ensure complete evaporation of the water.

2.3. Characterizations

Three characterisation methods are employed in this study: thermogravimetric analysis (TGA) on an STD Q600 V8.1 Build 99 (Instrument TA), X-ray diffraction(XRD) measurements using a Panalytical Empyrean XRD and Fourier transform infrared analysis(FT-IR) on a Vertex 70V.

3. Results and Discussion

3.1. Thermogravimetric Analysis (TGA)

Figure 2 shows the mass loss curves for carrageenan from spinosum (Carr-Sp) and cottonii (Carr-Co). Quantitative analysis reveals that Carr-Co exhibits 5.2% weight loss at 62°C (water evaporation), 62.3% at 242°C (polymer decomposition), and leaves 32.5% residue at 600°C. For Carr-Sp, water evaporation occurs at 82°C with 4.8% weight loss, followed by 58.7% decomposition at 229°C, leaving 36.5% residue [19, 20].

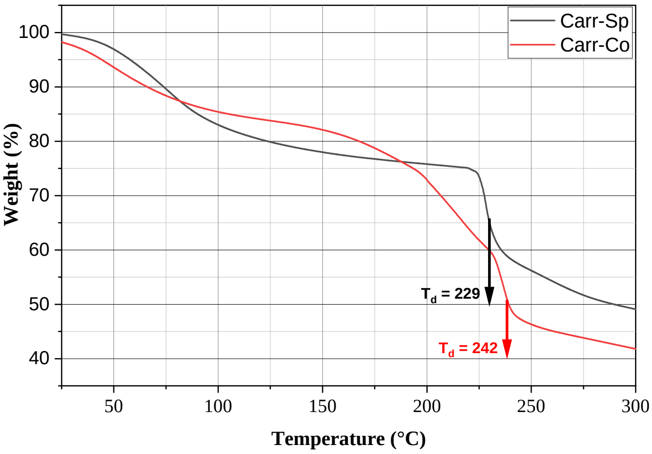
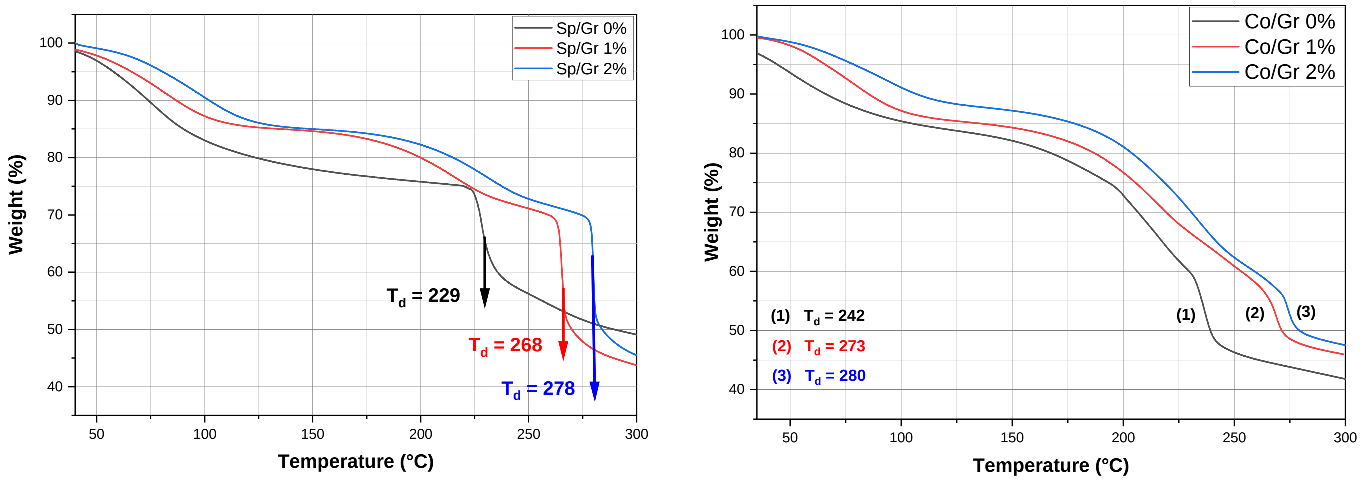


Figure 2. TGA curves of Carr-Co and Carr-Sp.

For the Sp/Gr (carrageenan from spinosum) films(Figure 4a), the trends in the curves are still similar but they differ in the carrageenan decomposition temperatures, which are 268°C for $Sp/Gr1\%$ and 278°C for $Sp/Gr2\%$. This implies that the addition of fillers increased the temperature of carrageenan degradation. This finding suggests that graphenes are present, which was the objective in the composites, as graphites alone would not give such an improvement. Jdidi Haythem et al. [21] showed that the addition of graphite to the polymer brought an improvement of only 5°C, whereas in most cases the addition of graphenes can greatly improve the decomposition temperature of polymers. Xiaoxin LU et al. [22] showed that the degradation temperature of nanocomposites with a graphene filler is plus 40°C compared to pure polymer. Other studies also show that a nanocomposite containing 1% graphene degraded at a temperature of around 40-50°C compared with pure polymer [23]. We can therefore say that graphene exists in composites. In the case of the composite with 2% filler, the difference in decomposition temperature is around 10°C. According to TGA analysis, carrageenan with 1% filler decomposes at 268°C, while carrageenan with 2% filler decomposes at 278°C. This can be attributed to the decrease in the exfoliated portion of graphites in the structure. That is, the graphene content of the composite decreases as the percentage increases. Alternatively, it is also conceivable that the fillers exceed the percolation threshold.

According to the Figure 4b, the trends of Co/Gr (carrageenan from cottonii) are the same when compared with those of Sp/Gr. That is, at 1% loading the improvement is very significant, whereas at 2% the increase is relatively small. This can be explained in the same way as for Sp/Gr. And the small difference between the temperatures can also be explained by the small temperature difference of the matrices. Graphene show a higher thermal property than natural graphite due to the size effect. This study demonstrates the effectiveness of graphene nanosheets in enhancing the physicochemical properties of carrageenan matrices. The Figure 2 and Figure 4 shows the decomposition temperatures of the matrices(Carr-Co and Carr-Sp) and composites(Co/Gr and Sp/Gr). Carr-Co can be said to have a higher T_d (°C) than Carr-Sp, whether loaded or not.



(a) *TGA curves of Sp/Gr*

(b) *TGA curves of Co/Gr*

Figure 3. *TGA curves of Sp/Gr and Co/Gr.*

3.2. X-ray Diffraction (XRD)

Figure 3 compares the diffractograms of graphite, the Co/Gr and the Sp/Gr films with different filler quantities. The spectra of Carr-Co and Carr-Sp show an amorphous structure, hence the absence of peaks in the Co/Gr 0% and Sp/Gr 0% films. The Co/Gr 1% film shows a basal reflection peak at 2θ = 26.53° with a lower intensity than that of pure graphite, with a value shifted by 0.13°. The value of 2θ, can vary on the order of 0.2° [24]. The presence of this peak shows that graphite is incorporated throughout the composite. The low intensity explains why the area crossed by the graphite X-ray beams is greater than that of the graphite in the polymer. The d-spacing values calculated from XRD peaks using Bragg's law ($n\lambda$ = 2d sinθ) show that pure graphite exhibits a d-spacing of 0.335 nm at 2θ = 26.6°. For Co/Gr 1% composite, the d-spacing increases to 0.336 nm (2θ = 26.53°), while Sp/Gr 1% shows 0.337 nm (2θ = 26.40°), suggesting slight intercalation.

From the diffractograms, characteristic graphite diffraction peaks in carrageenan increase with increasing levels of incorporated graphite, but do not change angle. The first conclusion is that graphite does not exfoliate in the polymer. However, partial exfoliation cannot be ruled out. The XRD diagram of Sp/Gr 1% shows a peak at 2θ = 26.40°, which characterises the graphite reflection and the hexagonal structure along the (200) orientation, as shown by the single peak, which also corresponds to an interreticular distance of $d_{200} = 0.34nm$ between the graphite planes [1, 25]. The intensity of this peak is much lower than that of the Co/Gr 1% peak, even though they have the same charge ratio. This may reflect the reduction of graphite throughout the polymer, and may confirm the existence of graphite exfoliation giving rise to graphenes. Moreover, the appearance of the sample helps to prove this finding. The test has been carried out 5 times and always gives similar results.

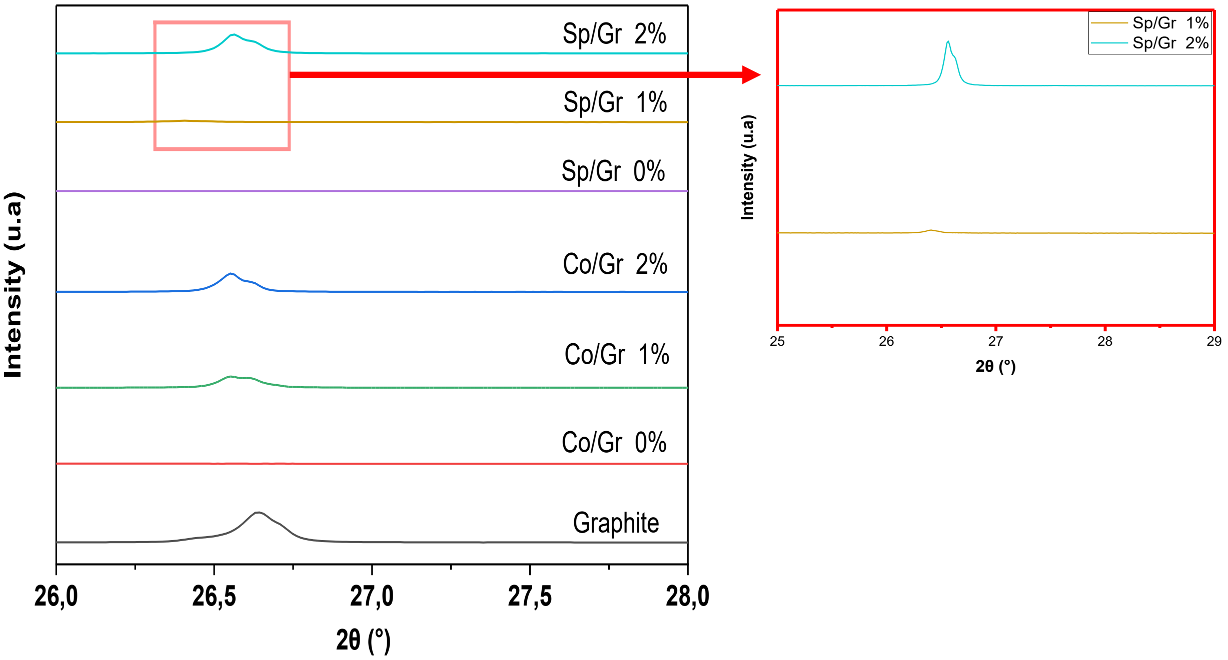
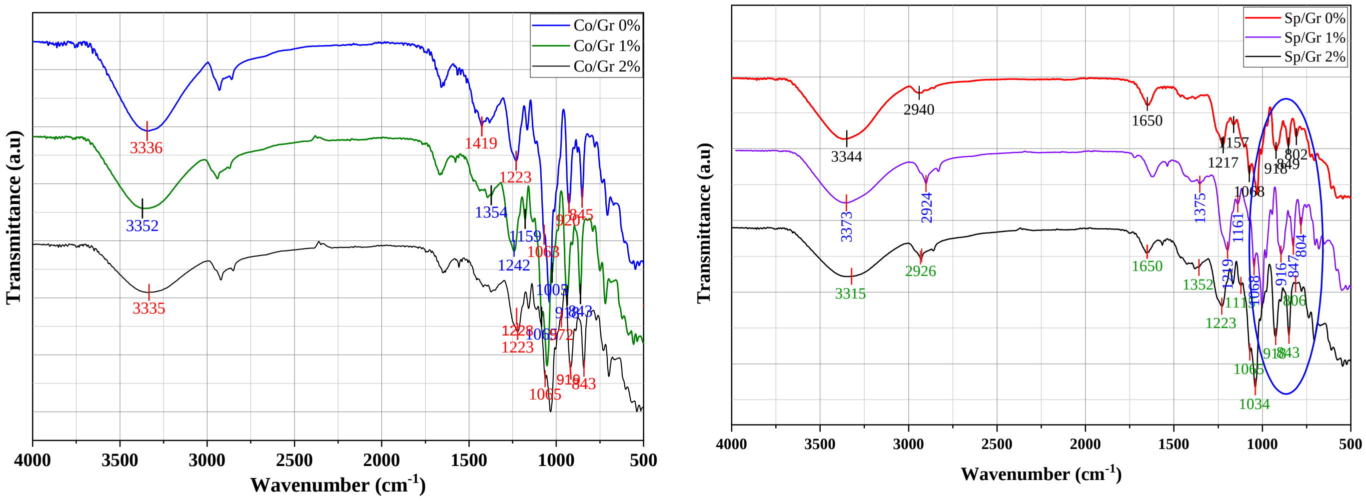


Figure 4. XRD diagram of Co/Gr and Sp/Gr.

3.3. Fourier Transform Infrared Analysis (FT-IR)

The FT-IR spectra of Co/Gr and Sp/Gr are shown in Figure 5. According to the figure, all peaks characteristic of carrageenan are present whether ι-carrageenan or κ-carrageenan. The peaks at 3336 and 1419 cm^{-1} respectively indicate the presence of the hydroxyl OH band $(CH_2 - OH)$ and $C - H$ at alkane bending vibration CH_2 [26, 27]. The bands at 1223 cm^{-1} and 845 cm^{-1} correspond to $C - O - S$ stretching vibration and ester sulfate groups(symmetrical vibration $O = S = O$), respectively. In addition, the characteristic $C - O - C$ band of the anhydrous-galactose residue and the $C - O$ bridge stretching peak at are visible at 920 cm^{-1} and 1115 cm^{-1} respectively. The peak at 845 cm^{-1} confirms the stretching vibration of the $C - O - SO_3$ group [28]. Frequencies between 845 cm^{-1} is associated with the $C - S$ bond bending vibration of D-galactose-sulfate, which is related to the amount of sulfate in κ-carrageenan [29] (Figure 5a). The peak observed at 918 cm^{-1} corresponds to the $C - O - C$ of 3,6 anhydrogalactose. The peaks at 802 and 849 cm^{-1} are attributed to the $-O - SO_3$ stretching vibration of D-galactose-2-sulfate(DA2S) and D-galactose-4-sulfate(G4S) [30]. The presence of these peaks confirms that we have ι-carrageenan, as Chitra et al have shown [31](Figure 5b).



(a) Spectre FIRT of Co/Gr

(b) Spectre FIRT of Sp/Gr

Figure 5. Spectre FIRT of Co/Gr and Sp/Gr.

4. Conclusion and Perspectives

Carrageenan-based films exhibit distinct physicochemical and thermal properties depending on their seaweed origin. The extraction of carrageenan from Cottonii and Spinosum seaweed, followed by composite formation with exfoliated graphite via solution mixing and casting-evaporation, yielded bio(nano)composite films with enhanced thermal stability (36°C increase in decomposition temperature) and structural integrity. XRD analysis confirmed graphite incorporation with d-spacing values of 0.336-0.337 nm, while FT-IR verified chemical interactions between carrageenan functional groups and graphite sheets.

These materials show promising potential for:

1) *Packaging applications*: Due to improved thermal stability and biodegradability
2) *Biomedical scaffolds*: Leveraging carrageenan's biocompatibility with graphite's electrical conductivity
3) *Biodegradable electronics*: Combining environmental sustainability with functional performance

Future research directions include:

1) Comprehensive mechanical characterization (tensile strength, elasticity, barrier properties)
2) Biodegradability assessment under various environmental conditions
3) Scale-up feasibility and economic analysis for industrial applications
4) Investigation of additional properties (electrical conductivity, antimicrobial activity)
5) Raman spectroscopy analysis to confirm graphene exfoliation efficiency

The preliminary results presented herein establish a foundation for further development of seaweed-based biocomposites, contributing to sustainable material innovation.

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Abbreviations

TGA	Thermogravimetric Analysis
XRD	X-ray Diffraction
FT-IR	Fourier Transform Infrared Spectroscopy
Carr-Co	Carrageenan from Kappaphycus Alvarezii (Cottonii)
Carr-Sp	Carrageenan from Eucheuma Denticulatum (Spinosum)
Co/Gr	Cottonii Carrageenan-graphite Composite
Sp/Gr	Spinosum Carrageenan-graphite Composite
DA	3,6-anhydrogalactose Unit
DA2S	D-galactose-2-sulfate
G4S	D-galactose-4-sulfate

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Olivier Dimbiniaina Maka: Writing – review & editing

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Maherizo Tiandrainy Gedice: Funding acquisition, Investigation

Heriarivelo Risite: Project administration, Supervision

Conflicts of Interest

The authors declare that there are no conflicts of interest regarding the publication of this paper. No financial or personal relationships with other people or organizations have inappropriately influenced this work.

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