Express Polymer Letters Vol.18, No.8 (2024) 796–806
Available online at www.expresspolymlett.com
https://doi.org/10.3144/expresspolymlett.2024.59

Research article

Microwave-assisted sodium alginate extraction from *Dictyota menstrualis* and the fabrication of green thermal insulators

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Received 7 April 2024; accepted in revised form 9 May 2024

Abstract. This study investigates the enhanced extraction of sodium alginate (SA) from *Dictyota menstrualis* using microwave-assisted techniques and its subsequent application in fabricating green thermal insulators. Utilizing optimized microwave parameters, we achieved a notable increase in SA yield, peaking at 18.5%, significantly higher than the 14.2% obtained through conventional methods. This process not only underscores the efficiency of microwave-assisted extraction by improving yield by approximately 30% but also highlights its environmental sustainability due to reduced solvent use and shorter processing times. The study demonstrates that increasing sodium alginate concentration from 1 to 5% enhances the mechanical strength and thermal insulation properties of bioaerogel scaffolds, evidenced by an increase in density from 0.171 to 0.234 g/cm^3 and a decrease in porosity from 93.6 to 89.6%. Additionally, the thermal conductivity and diffusivity measurements of 0.065 W/(m·K) and 0.294 mm^2/s, respectively, affirm the excellent thermal insulation stability of these scaffolds. The study demonstrates the potential of microwave-assisted extraction as a scalable and eco-friendly approach for biopolymer recovery, and the feasibility of using the extracted SA in creating effective, green thermal insulators, marking a significant step towards sustainable material development.

Keywords: *sodium alginate, aerogels, thermal insulator, microwave assessed*

1. Introduction

The quest for sustainable and eco-friendly materials in the field of material science has led to a growing interest in marine-derived biopolymers. Sodium alginate, a natural polysaccharide extracted from brown seaweeds, has emerged as a pivotal material due to its unique properties such as biocompatibility, biodegradability, and its ability to form gels in the presence of divalent cations [1]. These characteristics make sodium alginate an excellent candidate for a myriad of applications, including drug delivery systems, wound dressings, and as scaffolds for tissue engineering [2]. Among the diverse sources of sodium alginate, *Dictyota menstrualis*, a brown alga,

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stands out for its high growth rate and alginate content [3]. However, the extraction efficiency and yield of sodium alginate from this species have not been fully optimized, presenting an opportunity for research and development. Conventional sodium alginate extraction techniques are typically time-consuming, requiring several hours to complete, and are characterized by high energy consumption due to the need for prolonged heating at elevated temperatures [4, 5]. These factors not only reduce efficiency but also often result in lower yields [6]. In the study of Łabowska *et al*. [7], the authors reported that the prolonged exposure to heat can degrade the alginate or fail to efficiently break down the cell walls, compromising the quality of the extract. In contrast, microwave-assisted techniques significantly reduce the extraction time, often completing the process within minutes, and are more energy-efficient as the microwave energy directly interacts with the material. This method enhances cell disruption and enables better penetration of solvents into the biomass, typically achieving higher yields and maintaining better quality of alginate due to reduced thermal degradation [5]. This technique employs microwave energy to expedite the extraction process, achieving rapid and efficient solvent heating [8]. The underlying mechanism is believed to involve microwave-induced heating causing water within the seaweed cells to vaporize, thereby building pressure against the cell walls [8, 9]. This pressure leads to the disruption of the cell walls, allowing for the efficient release of compounds housed within the cells into the solvent. Yuan and Macquarrie [10], in 2015, pioneered the adoption of microwave heating over conventional heat sources for extracting alginate, marking a significant advancement in extraction technologies. Thus, the current research focuses on enhancing the yield of sodium alginate from *Dictyota menstrualis* through an eco-friendly approach, consist of microwave treatment.

Bioaerogel scaffolds represent a cutting-edge innovation in the field of biomaterials, offering a unique combination of properties that are particularly suited for many applications [11]. Derived from natural biopolymers, such as sodium alginate, these scaffolds are characterized by their ultra-lightweight nature, high porosity, and extensive surface area, which facilitate cell adhesion, proliferation, and differentiation [12]. The interconnected porous architecture of bioaerogels not only allows for efficient nutrient and waste exchange but also enables the precise control of materials release kinetics, making them ideal carriers for therapeutic agents [2]. The development of bioaerogel scaffolds from sodium alginate emphasizes the sustainability aspect by utilizing renewable marine resources, thereby offering an eco-friendly alternative to conventional synthetic materials. This research lies in the dual approach of enhancing the extraction yield of sodium alginate from an underutilized source using eco-friendly approach and the innovative application of this biopolymer in creating bioaerogel scaffolds and determining its thermal properties. This study not only contributes to the efficient utilization of marine biomass but also opens new avenues in the development of biocompatible and sustainable materials for medical applications. Through this exploration, we aim to bridge the gap between marine biopolymer resources and their practical applications, highlighting the importance of sustainable material science in addressing contemporary challenges in the biomedical field.

2. Materials and methods

2.1. Sodium alginate extraction

Dictyota menstrualis seaweed were procured from E.Z Oceiana SDN BHD (Penang, Malaysia), rinsed, dried at 60 °C, and ground into a fine powder. 10 g were subjected to microwave-assisted extraction using a 2% (w/v) sodium carbonate Sigma–Aldrich (Schnelldorf, Germany) solution as the solvent, with a solid to solvent ratio of 1:20 g/ml. The microwave extraction parameters were optimized, with the process set at 600 W for different times including 2, 4, 6 and 8 min. For comparative analysis, the control sample was extracted using a conventional heating method without the application of microwave energy [13]. Following extraction, the mixture was allowed to cool to room temperature, filtered through a fine mesh to separate the liquid extract, and the sodium alginate was precipitated by adding an equal volume of 99% ethanol Sigma–Aldrich (Schnelldorf, Germany), followed by gentle stirring and overnight setting at 4 °C. The precipitated sodium alginate was then collected, washed with ethanol to remove impurities, and finally dried in an air-circulated oven at 50 °C to get pure sodium alginate (Figure 1).

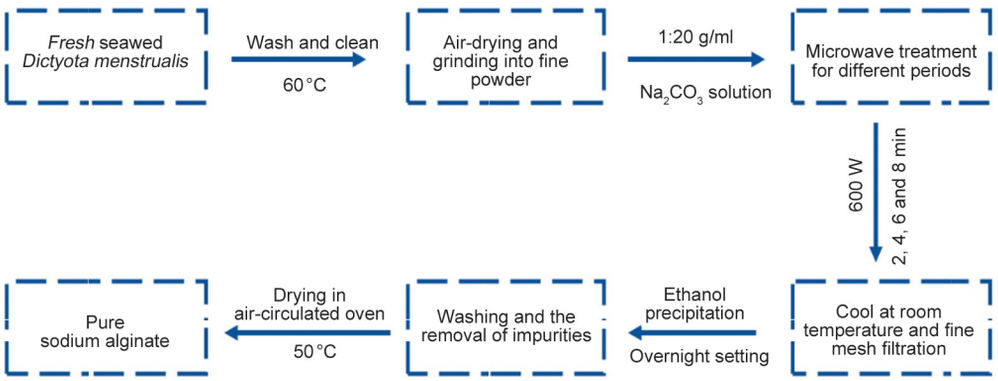


Figure 1. Illustration of microwave-assisted sodium alginate extraction approach from *Dictyota menstrualis.*

2.2. Characterization of sodium alginate

2.2.1. Yield sodium alginate of sodium alginate

The yield of sodium alginate extracted from *Dictyota menstrualis* was calculated by weighing the dried sodium alginate obtained after the microwave-assisted extraction process. This weight was divided by the dry weight of the seaweed powder used for the extraction to calculate the percentage yield. The formula for yield calculation was as Equation (1):

$$\text{Yield}\,[\%] = \\ = \frac{\text{Weight of dried sodium alginate}}{\text{Weight of dry seaweed power used}} \cdot 100 \quad (1)$$

2.2.2. FTIR analysis of sodium alginate

The chemical composition of the raw material and the extracted sodium alginate was determined using Fourier-transform infrared (FTIR) spectroscopy (Thermo Scientific model Nicolet I S10 spectrometer). The spectral analysis covered a range from 4000 to 400 cm^{-1}, and the samples were subjected to scanning with a precision of 4 cm^{-1}. Each sample was performed three times to ensure reproducibility of the results.

2.2.3. Thermal analysis of sodium alginate

Thermogravimetric analysis (TGA) and derivative thermogravimetry (DTG) using a TGA/SDTA 851e instrument manufactured by Mettler Toledo. The analysis was conducted within a consistent heating range from 25 to 800 °C at a rate of 10 °C/min to assess the thermal properties and behaviour of the extracted sodium alginate. Differential scanning calorimetry (DSC) analysis was also conducted using a DSC unit (Netzsch DSC-200 PC Phox, NETZSCH Holding, Selb, Germany). Approximately 10 mg of each sample was weighed and placed in a standard cup, while an empty cup served as a reference. The samples were subjected to heating from 30 to 800 °C at a rate of 20 °C/min under a nitrogen atmosphere. Measurements of weight loss and derivative weight loss corresponding to temperature changes were obtained.

2.3. Fabrication of bioaerogel scaffolds

Various concentrations of sodium alginate, ranging from 1 to 5%, were dissolved thoroughly in distilled water under continuous stirring for 3 h. until complete dissolution occurred. Subsequently, each solution was placed in the freezer overnight and underwent freeze-drying at –50 °C under 0.05 mbar pressure for a duration of 72 h to remove all water content from the suspension, resulting in the formation of sodium alginate bioaerogel scaffold.

2.4. Characterization of bioaerogel scaffolds

2.4.1. Surface morphology and physical properties

The surface morphology analysis was conducted using a field emission scanning electron microscope (FE-SEM), specifically the Leo Supra, 50 VP model by Carl Zeiss, SMT (Carl Zeiss Group, Oberkochen, Germany). The aerogel density was determined by maintaining a consistent shape (1 cm^3) for all the prepared aerogels, measuring their respective mass and volume. Porosity was calculated using the formula derived from the obtained density as mentioned in [14]. The water absorption capacity was assessed by submerging 1 cm^3 of each sample in 20 ml of distilled water, allowing saturation for a constant period of 60 s. After blotting excess water with filter paper, the saturated aerogel was weighed to determine the water absorption capacity.

2.4.2. FTIR analysis for bioaerogel scaffolds

Additionally, the investigation of functional groups present in the aerogel samples was performed using FT-IR spectroscopy. The spectroscopic analysis utilized the Thermo Scientific model Nicolet I S10 spectrometer, operated with a Perkin Elmer spectrum 1000 and samples prepared as KBr pellets to acquire the spectrum data. Measurements were conducted in the range of 4000–400 cm^{-1} with a resolution of 4 cm^{-1} and an accumulation of 16 scans per sample to ensure accuracy and repeatability.

2.4.3. Mechanical properties for bioaerogel scaffolds

The mechanical characteristics of the bioaerogel scaffolds were assessed via texture profile analysis conducted with a TA-HDi texture analyzer machine from Stable Micro Systems, based in Surrey, UK. The bioaerogels, standardized to dimensions of 2×2×1 cm (length, width, and height), underwent compression to 75% of their initial height, repeated twice for consistency. The analysis was executed in triplicate to ensure reliability and accuracy of the measurements.

2.4.4. Thermal analysis

The thermal properties and behaviour of all the prepared bioaerogel scaffolds were investigated through TGA and DTG utilizing a TGA/SDTA 851e instrument manufactured by Mettler Toledo. This analysis involved a consistent heating range from 25 to 800 °C, maintained at a rate of 10 °C/min. The thermal conductivity of the samples was determined using the Hot Disk TPS 2500 S Thermal Conductivity Meter, employing the Transient Plane Source technique. This method involves placing a nickel foil sensor, encapsulated in Kapton film, between two pieces of the aerogel sample. As the sensor simultaneously heats the sample and records the temperature increase, the instrument's software calculates the sample's thermal conductivity, thermal diffusivity, and specific heat capacity [15]. Measurements were conducted at room temperature, utilizing a standard solid sample holder, ensuring precise evaluation of the aerogels' thermal transport properties.

3. Results and discussion

3.1. Yield of sodium alginate

The findings of this research elucidate a substantial improvement in the extraction yield of sodium alginate from *Dictyota menstrualis* utilizing microwave-assisted extraction techniques. Notably, the application of microwave-assisted extraction for durations of 6 and 8 min significantly enhanced the yield of sodium alginate by approximately 30% compared to control samples processed via conventional extraction methods. Specifically, yields of 18.5% were achieved using the microwave-assisted approach for both 6 and 8 min, markedly higher than the 14.2% yield obtained through traditional extraction techniques. However, shorter microwave durations of 2 and 4 min resulted in comparatively lower yields of 14.9 and 16.2%, respectively. In recent research done by Torabi *et al*. [16], the authors investigated the influence of various parameters on the yield during the microwave-assisted extraction process targeting *Nizimuddinia zanardini*. The authors reported great enhancement in the yield, owing to the ability of microwave radiation's to penetrate the cell walls of effectively, facilitating the release of sodium alginate into the extraction solvent [16]. Additionally, the reduced extraction time and lower solvent consumption offered by microwave-assisted extraction contribute to its efficiency and environmental sustainability.

3.2. Chemical composition analysis

The FTIR analysis of the sodium alginate extracted from *Dictyota menstrualis* revealed distinct peaks indicative of its characteristic functional groups (Figure 2). Key absorption bands were observed at around 3420 cm^{-1}, attributed to the O–H stretching vibrations, indicating the presence of hydroxyl groups [17, 18]. The band at approximately 2920 cm^{-1} was assigned to C–H stretching vibrations, typical of aliphatic compounds. Significant peaks at around 1610 and 1420 cm^{-1} corresponded to the asymmetric and symmetric stretching of carboxylate ions (COO^-), respectively, confirming the alginate's polymeric uronic acid content [19]. Additionally, a peak near 1020 cm^{-1} was identified, which is characteristic of the C–O stretching vibrations, further validating the polysaccharide structure of the alginate. No notable variances were detected among the samples, suggesting pure sodium alginates in all of them without any significant impurities.

3.3. Thermal properties of sodium alginate

The thermal analysis of isolated sodium alginate is presented in Figure 3, in which we can see the TGA

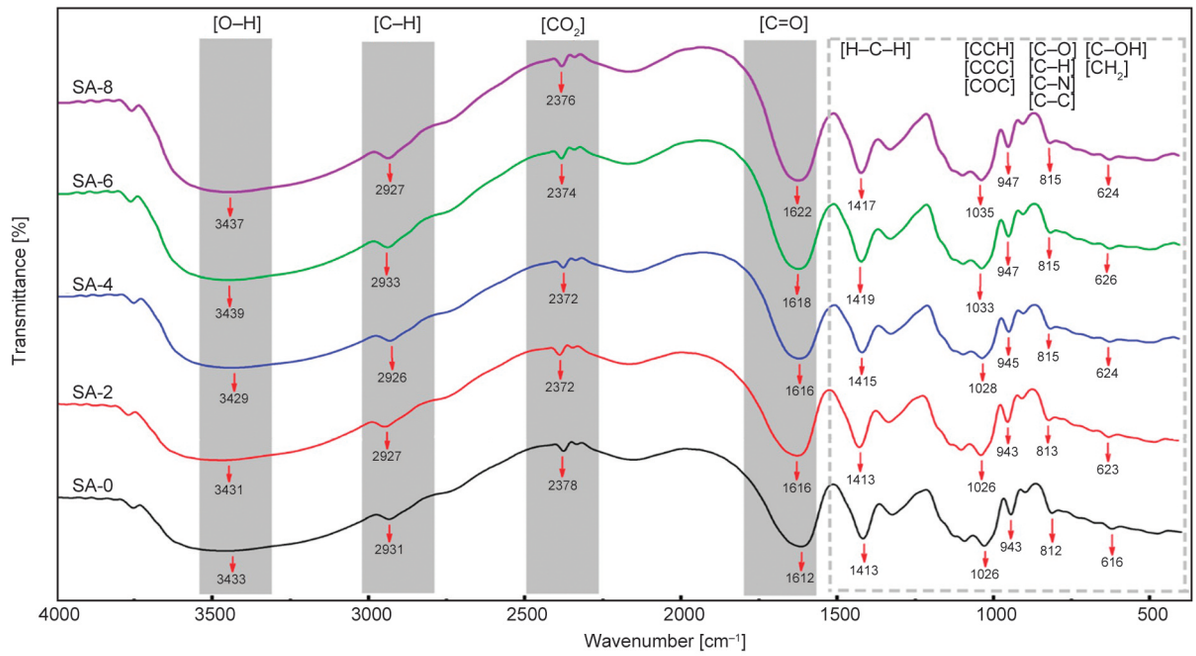


Figure 2. FTIR spectra for the isolated sodium alginate using different microwave treatment duration.

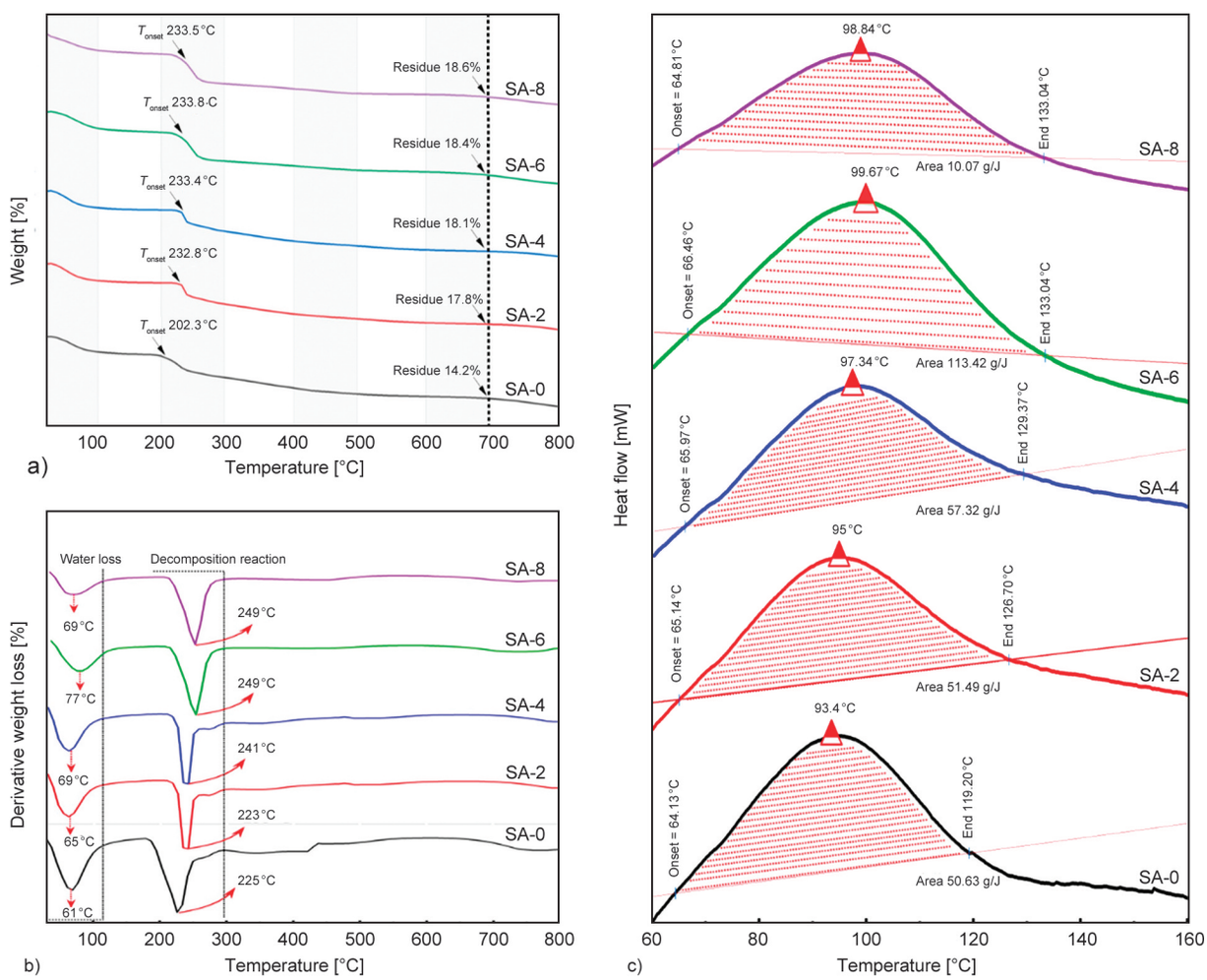


Figure 3. Thermal properties of the isolated sodium alginate using different microwave treatment duration: a) TGA b) DTG and c) DSC curves.

DTG curves, revealing insightful data on its thermal stability and degradation behaviour. The TGA curve show an initial weight loss observed below 100 °C, which could be attributed to the evaporation of water content. A significant degradation stage was noted between 200 and 300 °C, which is characteristic of the decomposition of the sodium alginate's saccharide rings, indicating the onset of thermal decomposition [20]. In a previous study by Xu *et al*. [21], the authors stated that alginate acted as a stabilizer to increase the thermal stability of materials during the thermal denaturation process, which support our results. In agreement with our results, a slight shift towards lower temperatures was also reported by Nguyen *et al*. [22]. The DTG curve provided a more detailed view of the degradation process, showing a prominent peak in this temperature range, which corresponds to the maximum rate of thermal decomposition of the alginate, which also related to depolymerization and pyrolysis decomposition of the main chain of chitosan polymer [23]. Following this peak, a gradual decline in weight loss rate was observed, suggesting the breakdown of more thermally stable components [24]. As the temperature approached 500 °C, a residual mass remained, indicating the formation of a carbonaceous residue. This behaviour is typical for polysaccharide-based materials, where complete degradation involves both the loss of organic components and the formation of a char residue [25]. The thermal analysis confirms that the sodium alginate extracted possesses typical thermal degradation characteristics of alginates, with the microwave-assisted extraction method not adversely affecting its thermal properties. This analysis underscores the potential of the extracted sodium alginate for use in applications where thermal processing is involved, such as the fabrication of bioaerogel scaffolds.

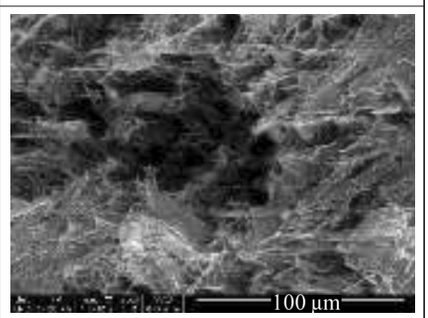
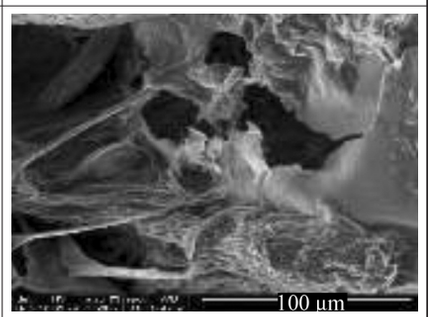
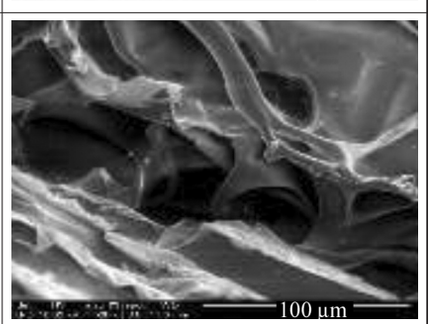
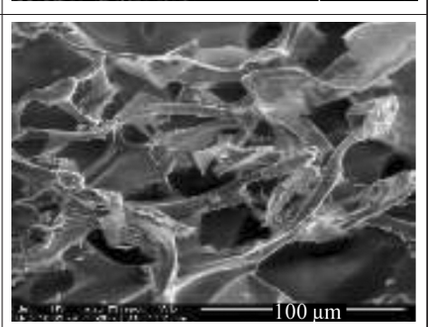
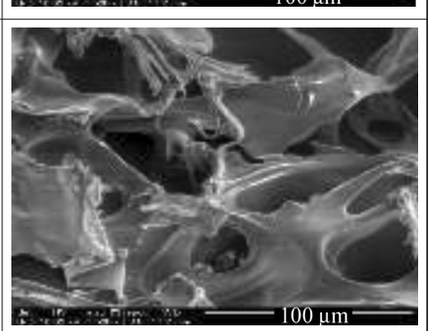
DSC analysis revealed distinct thermal transitions, consistent with the findings from TGA, which elucidates the relationship between thermal decomposition and the thermal stability of the samples according to previous studies [26, 27]. The DSC analysis highlighted notable endothermic and exothermic transitions, primarily associated with moisture loss and degradation. For the samples exposed to 2 min of microwave treatment, a lower onset of thermal degradation (T_{onset}) was observed, indicating a potentially less stable structure, possibly due to the inadequate extraction time [28]. As the microwave exposure time was extended to 4 and 6 min, there was a noticeable increase in both T_{onset} and the peak of degradation temperature (T_{peak}), suggesting an improvement in thermal stability. This improvement points to a more complete extraction process that likely facilitates the recovery of sodium alginate with a higher molecular weight and superior thermal properties. Notably, the sample treated for 8 min showed the highest thermal stability among the durations tested, likely due to the optimal extraction of high-quality sodium alginate, resulting in an enhanced yield of the biopolymer with improved thermal characteristics. However, it is essential to balance these results against the potential for thermal degradation at prolonged exposure times, which could compromise the integrity and molecular weight of the extracted alginate.

3.4. Characterization of bioaerogel scaffolds

The bioaerogel scaffolds were produced using sodium alginate extracted at the optimal microwave exposure time of 6 min. This duration was selected based on our findings that a 6 min microwave treatment significantly enhances the yield of sodium alginate while maintaining its structural integrity, compared to both shorter and longer microwave exposure times. Table 1 presents the results of density and porosity of the prepared bioaerogel samples together with the morphological analysis. The lowest density observed at 1% sodium alginate concentration (0.171 g/cm^3) corresponds to the highest porosity (93.6%), indicating an extremely lightweight and porous structure. As the sodium alginate concentration increases to 5%, the density progressively rises to 0.234 g/cm^3, while the porosity decreases to 89.6%. At a lower concentration, there is a reduced availability of polymer chains to form cross-links. This limitation results in a gel network with relatively fewer junction points and a larger volume of void spaces, hence a higher porosity [29]. The scarcity of cross-linking points also means that the gel matrix has less structural integrity and mass per unit volume, contributing to a lower density, which can be clearly seen in SEM images. A previous study reported a tubular unidirectional high porous structure in pure sodium alginate aerogel with smooth surface [30], which can be clearly seen in the concentrations higher than 3%. The increased number of polymer chains and cross-links within a given volume contributes to a higher mass per unit volume, thereby increasing

the density of the aerogel as sodium alginate concentration increases. The aerogel surfaces at this concentration exhibit a predominantly smooth texture punctuated by occasional, larger pores, underscoring the material's inherent lightweight and highly porous nature [31].

Table 1. Properties and photos of prepared bioaerogel scaffolds.

Sample	Density [g/cm^3)	Porosity [%]	Digital photos	SEM images
SA-1%	0.171±0.013	93.6		
SA-2%	0.186±0.019	92.8		
SA-3%	0.198±0.029	92.1		
SA-4%	0.208±0.026	90.3		
SA-5%	0.234±0.041	89.6		

3.5. Textile profile analysis of bioaerogel scaffolds

The mechanical and textural properties of sodium alginate bioaerogels prepared with varying concentrations (1 to 5%) exhibit a clear trend of enhancement in structural integrity and cohesiveness with increasing sodium alginate concentration as presented in Table 2. For instance, the strength of the aerogels shows a progressive increase from 0.209 N/mm^2 in the 1% SA sample to 0.368 N/mm^2 in the 5% SA sample, indicating improved mechanical robustness. This could be attributed to a denser cross-linked polymer network formation. This network increases the intermolecular forces within the aerogel, thereby enhancing its structural integrity and mechanical robustness [32]. Resilience also increases with concentration, from 37.0 to 54.2%, this trend is mirrored in the gumminess and chewiness values, where SA-5% exhibits the highest gumminess (3.11 g) and chewiness (31.43 g·mm), suggesting a denser, more cohesive network. The slight decrease in springiness from 2.02 mm in SA-1% to 1.89 mm in SA-5% might indicate a tighter network that is less prone to elastic deformation. Cohesiveness also shows an upward trend, from 68.4% in SA-1% to 83.7% in SA-5%, highlighting an increase in the internal bonding of the aerogel matrix. These results underline the significant impact of sodium alginate concentration on the structural and mechanical properties of bioaerogels.

3.6. Thermal insulator properties

Figure 4 presents the results of thermal insulation properties together with the porosity of the prepared samples. Thermal conductivity values increase progressively from 0.065 W/(m·K) for the 1% SA sample to 0.078 W/(m·K) for the 5% SA sample. This increase is attributed to the denser structure of higher concentration bioaerogels, which facilitates better heat transfer. However, all values are within a low range, underscoring the bioaerogels' potential as effective thermal insulators. The results of thermal conductivity are higher than those obtained by Xu *et al.* [33], which could be attributed to the use of tunable double-crosslinked network skeleton structure construction of SA composite with Al_2O_3 fibers. However, their pure SA aerogel results are closed to those obtained in our study. Thermal diffusivity shows a slight increase from 0.294 mm^2/s for the 1% SA bioaerogel to 0.301 mm^2/s for the 5% SA bioaerogel. This increment suggests a marginally faster

Table 2. The results of textile profile analysis of prepared bioaerogel scaffolds

Sample	Strength [N/mm^2]	Resilience [%]	Gumminess [g]	Chewiness [g·mm]	Springiness [mm]	Cohesiveness [%]
SA-1%	0.209±0.08	37.0±2.0	1.19±0.4	12.34±1.4	2.02±0.08	68.4±3.5
SA-2%	0.270±0.09	43.8±3.3	1.47±0.3	18.74±2.0	2.00±0.08	71.2±2.7
SA-3%	0.311±0.09	46.1±4.8	2.01±0.2	24.80±2.7	2.01±0.12	78.6±7.3
SA-4%	0.324±0.02	51.6±4.5	2.91±0.1	28.30±1.6	1.96±0.09	80.5±5.6
SA-5%	0.368±0.05	54.2±8.2	3.11±0.2	31.43±2.5	1.89±0.10	83.7±4.2

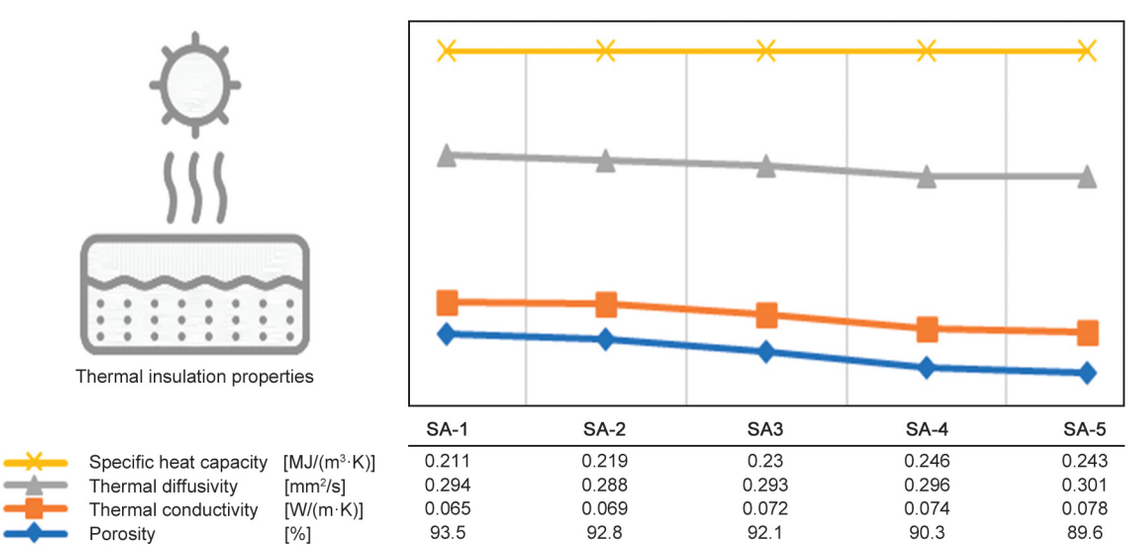


Figure 4. Thermal insulator properties of prepared bioaerogel scaffolds.

thermal response in higher concentration bioaerogels [34], possibly due to their denser structure and lower porosity, which can slightly enhance thermal transport within the material. These results are in-lines with those obtained by Akinshilo *et al*. [35], who prepared SA film. The behaviour of thermal management is intricately linked to the pore architecture within the aerogel scaffold [36]. Specific heat capacity, which indicates the amount of heat required to change the temperature of a material by one degree Celsius, also increased with the concentration of sodium alginate, peaking at 0.246 MJ/(m^3·K) for the 4% SA sample before a slight reduction at 5% SA concentration. The increase in specific heat capacity with concentration can be attributed to the greater mass of material available to absorb heat. The slight reduction observed in the 5% SA sample could be due to the densification of the aerogel structure, which may limit the material's ability to store heat. The anisotropic thermal insulation characteristics originated from a distinctly oriented lamellar/honeycomb configuration, while the elevated thermal conductivity stemmed from convection and thermal radiation processes [36]. The results of thermal conductivity and thermal diffusivity indicate an excellent thermal insulation stability of the prepared bioaerogel scaffolds.

4. Conclusions

In conclusion, we successfully established optimized microwave-assisted extraction parameters for the efficient isolation of sodium alginate from *Dictyota menstrualis*, setting a precedent for a novel and sustainable approach in biopolymer extraction. We discerned a marked enhancement in the yield of sodium alginate, significantly surpassing that obtained through conventional extraction methodologies. The pivotal achievement at 6 min extraction time underscores the efficacy of microwave-assisted extraction in bolstering the yield, thereby streamlining the extraction process while concurrently mitigating the environmental footprint associated with traditional extraction techniques. Building upon the foundation of this optimized extraction process, the study further delved into the innovative fabrication of bioaerogel scaffolds employing the extracted sodium alginate. These bioaerogels were characterized by their high porosity and remarkably low density, attributes that are quintessential for applications necessitating lightweight materials with substantial surface area. Additionally, comparing these results with existing literature confirms the similar thermal behavior of sodium alginate, aligning with documented benchmarks and underscoring the reliability of our findings. This research not only highlights the potential of microwave-assisted extraction as a green and efficient alternative to conventional extraction methods but also underscores the versatility and applicability of sodium alginate bioaerogels in advancing the fields of biotechnology and materials science. The findings from this study pave the way for future investigations into the scalability of microwave-assisted extraction for industrial applications, the further functionalization of bioaerogels for enhanced performance in specific applications, and the exploration of their biocompatibility *in vivo*.

Acknowledgements

Special thanks to Ministry of Higher Education (MOHE) for funding under the Fundamental Research Grant Scheme (FRGS), No. FRGS/1/2023/STGO5/USM/01/2. The authors would like to thank School of Industrial Technology, Universiti Sains Malaysia and Department of Chemical Engineering, Engineering Faculty, Universitas Sumatera Utara, Medan, Indonesia, for making this collaboration possible under the Adjunct Professor Senior Research Fellow Program for the year of 2024.

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