

# Polyaniline-carrageenan nanocomposite electrode as capacitor

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## Abstract

Energy is a requisite resource relied upon at a global scale by every household, business, and institution. Technological advancements have pushed the need for energy, particularly electrical energy, to skyrocket due to its increasing demand in different industries. Batteries have high energy density, but they also require constant recharging which takes up a significant amount of time. Batteries allow for high energy storage, but they break down over time as their chemicals lose potency over time from the recurrent charging and discharging. In this study, a more efficient way of storing electrical energy is explored through the production of supercapacitors using electrodes from the polymerization of polyaniline (PAni), a conducting polymer, and carrageenan, a polysaccharide. Essentially, supercapacitors are used in storing high amounts of electrical energy in small units. Unlike batteries, they have high power density which allows for quick bursts of electrical current. Supercapacitors not only charge faster than batteries do, but they also last longer.

**Keywords:** Polyaniline; carrageenan; nanocomposite electrode; supercapacitor.

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## Introduction

Energy is a requisite resource relied upon at a global scale by every household, business, and institution (OECD, 2012). Technological advancements have pushed the need for energy, particularly electrical energy, to skyrocket due to its increasing demand in different industries. The production and storage of electrical energy has long been a significant factor in technological advancements. Electrical energy can either be produced from hydroelectric power plants or from the transformation of geothermal energy. It is commonly stored in electrochemical cells such as dry cells or “batteries” (Crabtree et al., 2015).

Batteries have high energy density, but they also require constant recharging which takes up a significant amount of time (Colclasure et al., 2019; Larcher & Tarrascon, 2015). Batteries allow for high energy storage, but they break down over time as their chemicals lose potency over time from the recurrent charging and discharging. There are also safety concerns regarding them. Fires have been started due to battery malfunction and other electrical errors while charging (Sun et al., 2020).

In this study, a more efficient way of storing electrical energy is explored through the production of supercapacitors using electrodes from the polymerization of polyaniline (PAni), a conducting polymer, and carrageenan, a polysaccharide (Pasela et al., 2019). Essentially, supercapacitors are used in storing high amounts of electrical energy in small units (Dubal et al., 2015). Unlike batteries, they have high power density which allows for quick bursts of electrical current. Supercapacitors not only charge faster than batteries do, but they also last longer (Armelin et al., 2016).

In order to produce a supercapacitor through uncomplicated and inexpensive means, the supercapacitive properties of electrodeposited polyaniline-carrageenan were investigated. Since conducting polymers such as polyaniline (PAni) are generally easy to fabricate and carrageenan is an economical alternative electrolyte, this study is expected to offer another means of producing functional and commercial supercapacitors (Montalbo & Marquez, 2017).

## Objectives of the Study

This study aims to determine the feasibility of a polyaniline-carrageenan nanocomposite electrode for a supercapacitor by investigating what type and concentration of carrageenan in the electrolyte solution will give the most capacitance. It is to electrochemically synthesize a polyaniline-carrageenan nanocomposite electrode for supercapacitor application. Specifically, the objectives are to:

1. Structurally characterize the fabricated polyaniline-carrageenan nanocomposite using Fourier transform infrared (FTIR) spectroscopy;
2. Characterize the surface topography using Atomic Force Microscopy (AFM); and
3. Determine the electrochemical characteristics of the electrode by employing cyclic voltammetry (CV).

## Hypothesis

### Research Hypothesis

A polyaniline-carrageenan nanocomposite electrode can charge and discharge electrical energy using an electrolyte solution with the most amount of carrageenan containing the most ester sulfate groups to produce the most capacitive electrode.

### Statistical Hypothesis

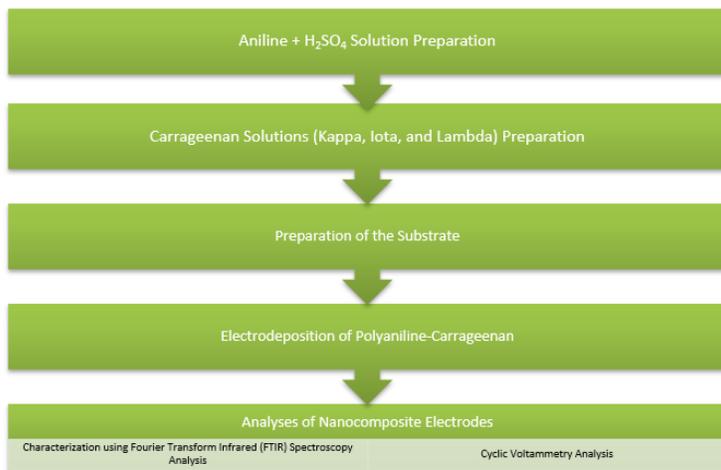
H<sub>0</sub>: The number of ester sulfate groups in the carrageenan does not affect the capacitance of the electrode.

H<sub>a</sub>: The more sulfated the carrageenan is, the more capacitive the electrode will be.

H<sub>0</sub>: The concentration of the carrageenan solution does not affect the capacitance of the electrode.

H<sub>a</sub>: The more concentrated the carrageenan solution is, the more capacitive the electrode will be.

## Methodology



**Figure 1.** Process for polyaniline-carrageenan nanocomposite electrode as capacitor

### Aniline + H<sub>2</sub>SO<sub>4</sub> Solution

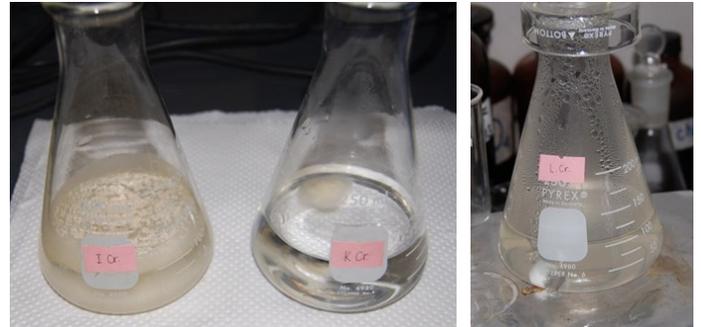
A 100 mL solution of 1 Molar H<sub>2</sub>SO<sub>4</sub> was prepared. 50 mL of 1M H<sub>2</sub>SO<sub>4</sub> was measured using a 100 mL graduated cylinder and poured in a 100 mL beaker. Using a micropipette, 1 mL of aniline was added to the beaker. The beaker was covered with Parafilm and the solution was sonicated for 10 minutes.



**Figure 2.** Aniline + H<sub>2</sub>SO<sub>4</sub> solution

### Carrageenan Solutions

For each type of carrageenan, 0.5 g of the powder was measured and placed in an Erlenmeyer flask. The flask was filled with water to produce a 100 mL solution. The solution was stirred continuously using a magnetic stirrer with the temperature set at 60° - 70° Celsius throughout the experiment.



**Figure 3.** Carrageenan solutions

### Substrate

The substrate used was an indium tinted oxide (ITO) coated PET. Using a pencil and a ruler, 1" x 1/2" was measured and cut. The cover was peeled off and the substrates were placed in a vial using a pair of tweezers. The vial was filled with propanol and placed in a sonicator for five (5) minutes.



**Figure 4.** Substrate

### Electrodeposition of Polyaniline-Carrageenan

The three electrode cell system was set up. Platinum (Pt) was used as the auxiliary (counter) electrode, the substrate was used as the working electrode, and Silver (Ag) / Silver chloride (AgCl) was used as the reference electrode (Ding et al., 2008). A potentiostat was used for the Electrochemistry System.

The substrate was taken from the vial using a pair of tweezers and the conducting side was determined by a multimeter set at Ohms. The substrate was attached to the working electrode with the conducting side facing the counter electrode. The electrolyte solution was prepared by getting 6 mL of the aniline-sulfuric acid solution using a micropipette and adding different amounts of the carrageenan solutions as it was being sonicated.

After sonication, the solution was placed on a platform in the electrochemistry set-up. For the electrodeposition, the electrodes were partially submerged in the solution. The recorder was set to galvanostat mode where a constant current of 4mA was applied to deposit PANi and PANi-carrageenan on the surface of the substrate. Afterwards, the substrate was taken and air dried.

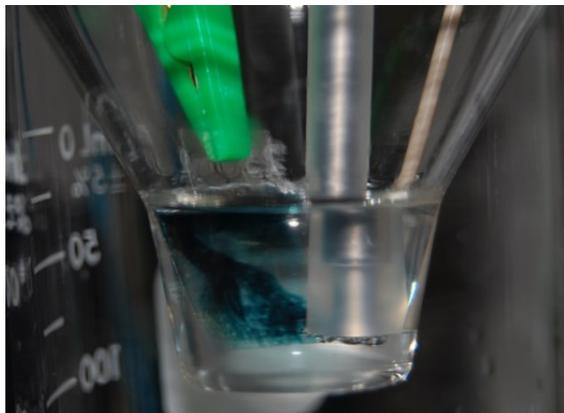


Figure 5. Electrodeposition of polyaniline-carrageenan

The experiment was done using the three types of carrageenan (kappa, iota, and lambda) with three trials each. Concentrations were indicated in Table 1 below.

Table 1. Concentrations of carrageenan solutions and aniline-sulfuric acid in the electrolyte solution

Carrageenan Solution	1% (0.06 mL)	5% (0.32 mL)	10% (0.67 mL)
Aniline + H <sub>2</sub> SO <sub>4</sub>	99% (6 mL)	95% (6 mL)	90% (6 mL)

#### Analyses of Nanocomposite Electrodes

The polyaniline-carrageenan composite was characterized using Fourier transform infrared (FTIR) spectroscopy to determine whether or not carrageenan was present in the nanocomposite electrode. Using the EChem Software Application, the capacitive and conductive property of each nanocomposite electrode was determined by applying voltages. Using cyclic voltammetry, the graphs of the redox reactions of the different electrodes are made. For each graph, a specific voltage range is used. The lower boundary is 0.2000 V and the upper boundary is 0.6020 V.

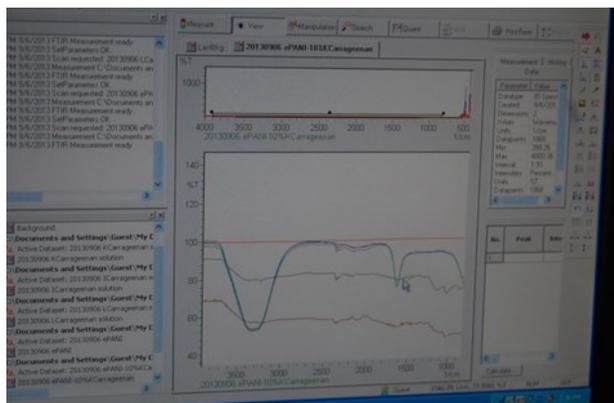


Figure 6. Characterization using Fourier Transform Infrared (FTIR) Spectroscopy analysis

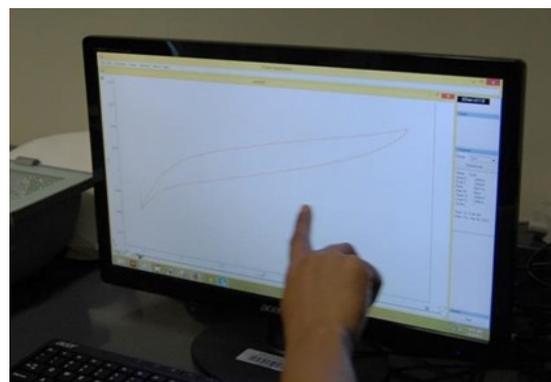


Figure 7. Cyclic Voltammetry (CV) analysis

## Results and Discussion

### Structural Characterization: Fourier Transform Infrared (FTIR) Spectroscopy

The production of a nanocomposite is an objective of this study (Shao et al., 2017). The presence of carrageenan in the electrode is determined through FTIR spectroscopy to validate its composite nature. The FTIR spectra of electrodeposited PANi and PANi-Carrageenan composites are presented in Figures 8 and 9.

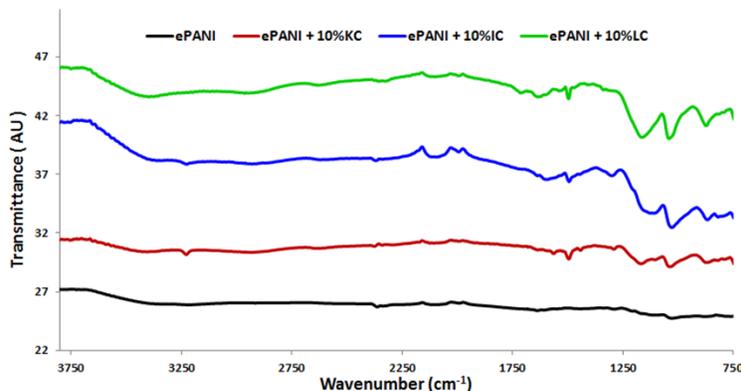


Figure 8. Overlay FTIR spectra of PANi and PANi-carrageenan composites

Based on the graph of the PANi, the PANi-carrageenan composites show different peaks at 750cm<sup>-1</sup> to 1750cm<sup>-1</sup>. There are peaks that occur only when carrageenan is present. This proves the presence of carrageenan in the electrode.

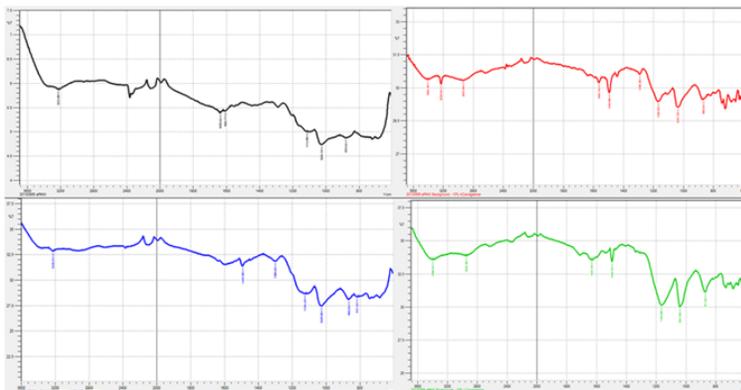


Figure 9. FTIR spectrum of A) PANi only, B) PANi + κ-carrageenan, C) PANi + ι-carrageenan and D) PANi + λ-carrageenan

Compared to PANi, the PANi-carrageenan composites exhibited peaks characteristic of carrageenan. These peaks are caused by functional groups present, with peaks inherent to carrageenan from a study by Prado-Fernández et al. (2002).

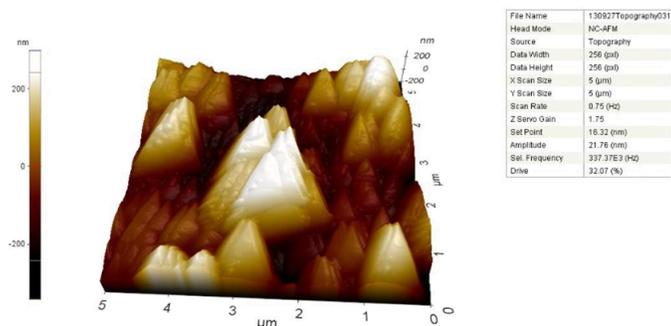
It also shows that characteristics of peaks of  $\kappa$ -carrageenan appear at  $3229\text{ cm}^{-1}$  and  $3404\text{ cm}^{-1}$ , and those for  $\iota$ -carrageenan, appear at  $3227\text{ cm}^{-1}$  and  $3380\text{ cm}^{-1}$ . Bands appear at  $1042\text{ cm}^{-1}$  and  $3397\text{ cm}^{-1}$  in the  $\lambda$ -carrageenan composite. The peaks shown in Table 2 prove that carrageenan was indeed deposited in the electrode. This confirms the composite nature of the electrode.

**Table 2.** Peaks of 10%  $\kappa$ -,  $\iota$ -, and  $\lambda$ -carrageenans

$\kappa$ -carrageenan			$\iota$ -carrageenan			$\lambda$ -carrageenan		
No.	Peak	Intensity	No.	Peak	Intensity	No.	Peak	Intensity
1	651.94	0.53	1	682.80	28.26	1	650.01	31.63
2	721.38	0.53	2	723.31	28.27	2	682.80	31.38
3	742.59	0.53	3	742.59	28.21	3	731.02	31.42
4	786.96	0.53	4	794.67	28.44	4	871.82	31.13
5	819.75	0.53	5	817.82	28.35	5	1041.56	30.02
6	866.04	0.53	6	866.04	28.11	6	1165.00	30.15
7	1037.70	0.54	7	1028.06	27.46	7	1336.67	34.18
8	1166.93	0.53	8	1126.43	28.69	8	1496.76	33.42
9	1290.38	0.51	9	1126.43	28.69	9	1537.27	34.09
10	1440.83	0.52	10	1300.02	31.88	10	1631.78	33.59
11	1492.90	0.53	11	1492.90	31.37	11	1710.86	33.91
12	1562.34	0.52	12	1595.13	31.55	12	2102.41	35.30
13	2931.80	0.52	13	1994.40	33.91	13	2326.15	34.92
14	3228.84	0.52	14	2110.12	33.47	14	2628.98	34.58
15	3404.36	0.52	15	2368.59	33.16	15	2945.30	33.91
			16	3226.91	32.85	16	3396.64	33.58
			17	3379.29	33.20			

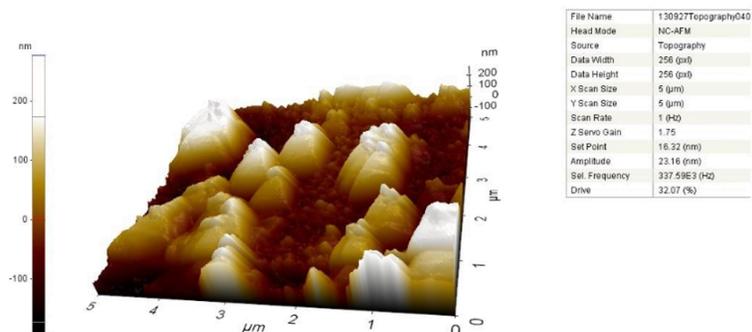
**Surface Topography: Atomic Force Microscopy**

The surface topography of the PANi +  $\kappa$ -carrageenan composite (shown in Figure 10) can be described as having mountain-like structures with pointed peaks. There are also distinct nadirs and zeniths. The different triangular structures are also grouped closely together. They seem to be grouped according to their relative sizes and are generally tall. There are also dark areas and light areas spread throughout the surface.



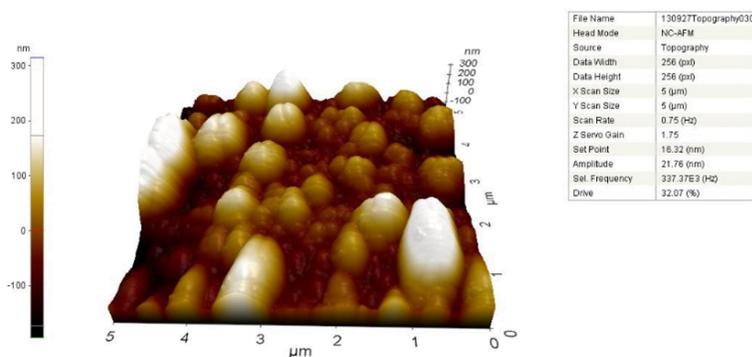
**Figure 10.** PANi with  $\kappa$ -carrageenan (in 3D)

The surface topography of the PANi +  $\iota$ -carrageenan composite (shown in Figure 11) can be described as having mountain-like structures which have uneven and slightly pointed peaks. The different triangular structures are not that high, with heights ranging from medium to low elevations. The surface is smoother compared to the surface of the PANi +  $\kappa$ -carrageenan composite. The difference in spacing between the mountain-like structures are neither grouped nor packed.



**Figure 11.** PANi with  $\iota$ -carrageenan (in 3D)

The surface topography of the PANi +  $\lambda$ -carrageenan composite (shown in Figure 12) can be described as having mountain-like structures which are dome-shaped and have rounded peaks or edges. The surface seems to be the smoothest among the three types of PANi-carrageenan composites. The different mountain-like structures differ slightly in height. They are also scattered and evenly distributed. The structures are not that high compared to the triangular structures in the PANi +  $\kappa$ -carrageenan composite.



**Figure 12.** PANi with  $\lambda$ -carrageenan (in 3D)

Figures 10, 11, and 12 show that the PANi-carrageenan composite is a nanostructure, since an image is seen through AFM, which is primarily used to show nanostructure, which means that the electrode is both a nanostructure and composite in nature.

**Electrochemical Characterization: Cyclic Voltammetry**

Cyclic voltammetry was employed to determine the capacitive characteristic of the PANi-carrageenan composites. The opening of the graph clearly demonstrates its capacitive capability as the redox reaction occurs. The concentrations of the PANi-carrageenan composites were kept at a constant 10% to eliminate any conflicting variables.

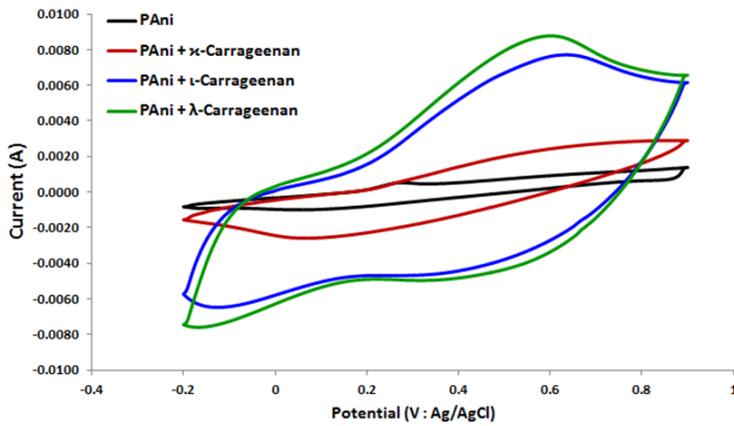


Figure 13. Overlay cyclic voltammogram of PANi and PANi-10 carrageenan composites

There is a distinct difference in the opening of the curve among PANi and its composites as shown in Fig. 8. It is clear that the 10% kappa composite showed a larger area enclosed in the redox curve compared to PANi alone, and that the 10% iota composite exhibited an even larger area. The 10% lambda composite had the largest area in the redox curve of them all and, therefore, has the highest capacitance. This is further proved by the computed mean capacitance of each composite. PANi + 10% λ-carrageenan had the highest capacitance compared to PANi and its kappa and iota composites.

### Capacitance of PANi and PANi-10% Carrageenan Composites

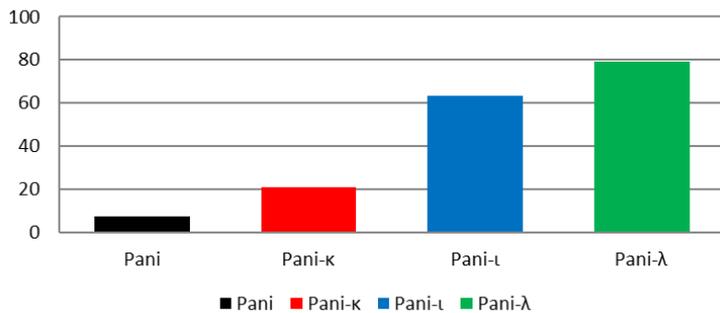


Figure 14. Computed mean capacitance (in Farad) of PANi and PANi-10% carrageenan composites

Using cyclic voltammetry, the graphs of the redox reactions of the different electrodes were made and for each graph, a specific voltage range was used. The lower boundary is 0.2000 V and the upper boundary is 0.6020 V. The area of the curve was calculated using the integral function. Since the x-axis parameter is time and the y-axis parameter is the current, the area under the curve equals the amount of charge. From this, the capacitance of each electrode is calculated using the formula  $C = [Q/(V_f - V_i)]$  where C is the capacitance, Q is the amount of charge and  $V_f - V_i$  is the change in voltage. Based on the results, the electrode involving λ-carrageenan gave the highest capacitance with an average of 78.97181 Farads. The electrode involving ι-carrageenan comes next, giving an average capacitance of 63.2089 Farads. The electrode involving κ-carrageenan comes third, giving an average capacitance of 20.93201 Farads. The electrode involving PANi comes last, giving an average capacitance of 7.256219 Farads.

Since the electrode involving λ-carrageenan gave the highest capacitance among the four types of electrodes, the capacitance of the different electrodes of λ-carrageenan based on their concentrations are calculated. Again the formula  $C = [Q/(V_f - V_i)]$  is used for each electrode. The same voltage range and the integral function are used. Based on the results, the electrode involving 10% carrageenan gave the highest capacitance, giving an average capacitance of 78.9781 Farads.

It was decided that only the PANi + λ-carrageenan composite would be tested for the electrodes with different concentrations of the carrageenan solution, since it was identified as the most capacitive electrode among the three PANi composites.

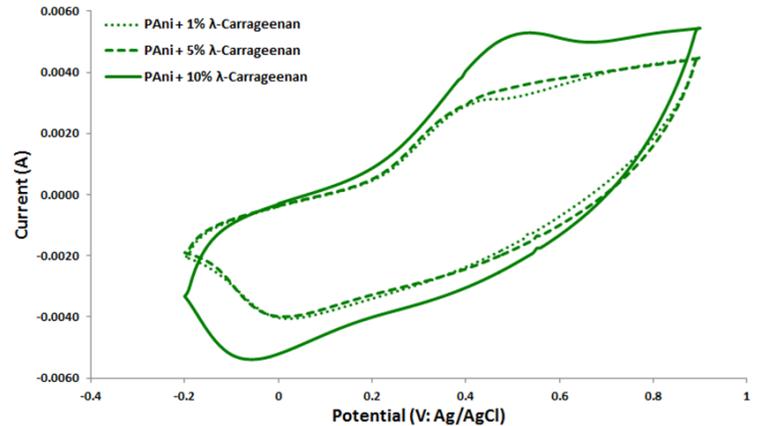


Figure 15. Overlay cyclic voltammogram of PANi + λ-carrageenan composites with different concentrations

An ideal capacitor has a rectangular shape. On the other hand, real capacitors often have a slow rise in current and some curves on the corners of the rectangle due to the effect of Equivalent Series Resistance.

Comparing the areas enclosed in the redox curves (Figure 15), it shows that the 1% composite has a smaller area compared to the 5% composite. Also, the opening of the redox curve is largest in the composite with the 10% concentration, further proving that it is the most capacitive electrode in terms of carrageenan solution concentration.

### Mathematical Analysis: T-Test

The capacitive value of each composite was determined by computing for the standard deviation and mean. Then, the capacitive capability of each nanocomposite was compared using a T-test to determine the most capacitive composite. Table 3 shows the computed capacitance values of PANi and each of its composites.

**Table 3.** Computed capacitance from three trials of different nano-composite electrodes

Concentrations	Trials	Capacitance in Farads	Standard Deviation	Mean
PAni	1st	6.9129353234	0.298	7.26
	2nd	7.447761194		
	3rd	7.407960199		
PAni + 10% $\kappa$ -carrageenan	1st	20.6592039801	0.260	20.93
	2nd	20.9601990049		
	3rd	21.1766169154		
PAni + 10% $\iota$ -carrageenan	1st	63.7562189054	0.510	63.21
	2nd	63.09845771144		
	3rd	62.7860696517		
PAni + 10% $\lambda$ -carrageenan	1st	78.5820895522	0.340	78.97
	2nd	79.1293532338		
	3rd	79.2039800995		

As shown in Table 4, the results of the T-test show that PAni + 10%  $\kappa$ -carrageenan, with the lowest farads, has the lowest capacitance. Since the results of the T-test showed that PAni + 10%  $\kappa$ -carrageenan has higher capacitance compared to PAni alone, the iota composite has higher capacitance than the kappa composite, and PAni + 10%  $\lambda$ -carrageenan has higher capacitance than PAni + 10%  $\iota$ -carrageenan; thus, the lambda composite appears to have the highest capacitance. This is backed up by the mean capacitances of each composite shown in Table 3. PAni + 10%  $\lambda$ -carrageenan had the highest mean capacitance as shown in Figure 12.

**Table 4.** T-test of the computed capacitance values of PAni and PAni-10% carrageenan composites

Composite	Null Hypothesis	Alternative Hypothesis	Critical Region	Degrees of Freedom	t	Decision
PAni vs. $\kappa$ -carrageenan	$\mu_P = \mu_\kappa$	$\mu_\kappa > \mu_P$	$t > 2.015$	$v = 5$	69.183	Reject Ho
$\kappa$ -carrageenan vs. $\lambda$ -carrageenan	$\mu_\kappa = \mu_\lambda$	$\mu_\lambda > \mu_\kappa$	$t > 1.943$	$v = 6$	273.078	Reject Ho
$\kappa$ -carrageenan vs. $\iota$ -carrageenan	$\mu_\kappa = \mu_\iota$	$\mu_\iota > \mu_\kappa$	$t > 2.132$	$v = 4$	147.770	Reject Ho
$\lambda$ -carrageenan vs. $\iota$ -carrageenan	$\mu_\lambda = \mu_\iota$	$\mu_\lambda > \mu_\iota$	$t > 2.015$	$v = 5$	52.585	Reject Ho

Note: Level of significance ( $\alpha$ ) = 0.05

**Table 5.** Computed capacitance of  $\lambda$ -carrageenan concentrations from three (3) trials of different nanocomposite electrodes

Concentrations	Trials	Capacitance in Farads	Standard Deviation	Mean
1% $\lambda$ -carrageenan concentration	1st	3.651741294	0.373	4.05
	2nd	4.109452736		
	3rd	4.390543264		
5% $\lambda$ -carrageenan concentration	1st	5.564676617	0.134	5.42
	2nd	5.3880559701		
	3rd	5.300995025		
10% $\lambda$ -carrageenan concentration	1st	78.5820895522	0.340	78.97
	2nd	79.1293532338		
	3rd	79.2039800995		

As shown in Table 6, the results of the T-test showed that 1%  $\lambda$ -carrageenan in the PAni composite, with the lowest farads, exhibited the lowest capacitance. Since the results of the t-test showed that the 5% composite has a higher capacitance (even with the slightest difference) compared to the composite with the 1% concentration, the 10% composite has a higher capacitance than both the 1% composite and the 5% composite; thus, the PAni + 10%  $\lambda$ -carrageenan composite appears to have the highest capacitance based on Table 5.

**Table 6.** T-test of the computed capacitance values of the different concentrations of  $\lambda$ -carrageenan in the PAni nanocomposite

Composite	Null Hypothesis	Alternative Hypothesis	Critical Region	Degrees of Freedom	t	Decision
1% and 5%	$\mu_A = \mu_B$	$\mu_B > \mu_A$	$t > 2.353$	$v = 3$	5.975	Reject Ho
1% and 10%	$\mu_A = \mu_C$	$\mu_C > \mu_A$	$t > 2.353$	$v = 3$	109.418	Reject Ho
5% and 10%	$\mu_B = \mu_C$	$\mu_C > \mu_B$	$t > 2.132$	$v = 4$	193.983	Reject Ho

Note: Level of significance ( $\alpha$ ) = 0.05

## Conclusions

This study was conducted in order to determine the supercapacitive properties of electrodeposited polyaniline-carrageenan composites that will validate its feasibility as a supercapacitor. A nanocomposite electrode was made by electrodepositing polyaniline and carrageenan. The FTIR spectra confirmed the composite nature of the electrode. Peaks characteristic of carrageenan were found in each FTIR spectrum of the polyaniline-carrageenan composite. The capacitive property of the composite electrodes was determined using cyclic voltammetry. The cyclic voltammogram of the composites according to the type of carrageenan present showed a significant difference in the capacitance of each composite with lambda exhibiting the highest values. Furthermore, comparison of the cyclic voltammograms of the composites in relation to the concentration of the carrageenan solution showed that the composite with the highest concentration, 10% PAni-carrageenan composite, had the highest capacitance. The results of the T-tests show that the more sulfated the carrageenan is and that the more concentrated the carrageenan solution is, the more capacitive the electrode will be. From the results, it can be deduced that the most capacitive electrode was the PAni + 10%  $\lambda$ -carrageenan. Moreover, the capacitance of each composite is within the range of supercapacitance which further proves that a supercapacitor can indeed be made using polyaniline-carrageenan nanocomposite electrodes.

## Recommendations

Recommendations were suggested by the researchers for further studies:

- Different combinations of kappa, iota, and lambda carrageenan can be applied (i.e. 15% kappa and 85% iota, etc.)
- Different characterization methods can be used instead of Fourier transform infrared spectroscopy.
- In making the carrageenan solution, different masses of carrageenan can be used for each type.
- Instead of making a nanocomposite electrode using polyaniline and carrageenan; carrageenan can be applied as a separate solid electrolyte.

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