Scanning electron microscopy of soxhlet extracted aloe vera gel for electrolyte application

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Abstract: Organic materials such as aloe vera gel are now being used in a wide range of solar cells application because of their properties, environmental safety and low cost. The characteristics of the aloe vera gel are discussed in this paper to determine the preferable characteristics of aloe vera gel which make it suitable to act as gel electrolyte in DSSCs. The study was started by selecting the best aloe vera gel properties between younger and older aloe vera gel through the refractive index and viscosity analysis. Surprisingly, older aloe vera gel has suitable characteristics which is lower refractive index and consists higher value of viscosity, which needed in electrolyte application for DSSCs when being extracted by using soxhlet extraction methods. The extraction process was carried out in a series of solvents which is hexane and the mixture of hexane and ethanol at specific ratio. The functional group of the unfiltered and filtered aloe vera gel were determined by the Fourier Transform Infrared Spectroscopy (FTIR) while the surface morphology and spatial distribution elements of the samples were examined by using Scanning Electron Microscopy (SEM) and Energy Dispersive X-Ray Analysis (EDX).

1. Introduction
Aloe vera (Aloe barbadensis Miller) had a long history of providing a myriad of health benefits and being one of the herbal remedies most frequently used throughout the world [1]. It is known as a tropical or subtropical plant with a turgid green leaves joined at the stem in a rosette pattern. These leaves can be separated into three layers which are the inner gel, the yellow sap known as latex, and the outer thick layer, called the rind [2].

Currently, aloe vera has become one of the most important raw materials in the food industry since it represents an emerging source of bioactive components. The potential use of aloe vera gel in the food industry is mainly focused on the development of functional foods due to its beneficial properties in treating constipation, coughs, diabetes, headaches, arthritis and immune-system deficiency [2]. However, in the past few years, researchers have found another usage of aloe vera gel where it can be applied in the electronics. It appears to be a promising candidates for memory application because of the demonstrations of electrical signals in living aloe vera plants.

In this research, aloe vera gel has been chosen to act as a major component in electrolyte for the dye-sensitized solar cells (DSSCs) due to several properties that belong to polysaccharides such as good ionic conductivity, typically 2.75×10⁻⁵ S/cm, consists of charge trapping/de-trapping process, and have a lower value of dielectric permittivity, k which is around 3.39 to 3.6 [3-5]. Therefore, several analysis have been studied in this research for aloe vera gel based on their ages between 2 and 4 years old in order to determine the suitability of which aloe vera gel acquired to fulfill the requirement to act...
as an electrolyte. Age is being studied in this paper based on it being a factor that causes changes of the properties in aloe vera gel. After that, extraction process can be done based on the selected aloe vera gel that contained preferred characteristics in order to act as an electrolyte. Thus, soxhlet extraction methods known as solid-liquid extraction being used to get the aloe gel.

For a decades, soxhlet extraction have been used everywhere for many different purposes. In terms of the efficiency, soxhlet extraction is described as the universal chemical extraction process. The researchers usually used this method with the purpose of effortlessly extracting major and minor compounds. However, this process required large extraction time and quantity of solvents [6].

2. Materials and Methods

2.1. Materials
Two types of fresh aloe vera, in between 15 to 35 cm and 45 to 55 cm length which is around 2 and 4 years old were used. Two types of solvent were used for extraction process which are hexane and ethanol.

2.2. Sample preparation for viscosity and refractive index analysis
Two types of aloe vera leaves have been used which are younger and older aloe vera. First and foremost, the leaves were washed with fresh water. Then, the outer skin and the exudates of the leaves were removed manually by using sharp knife in order to form fillet. After that, the fillets were ground using a blender to obtain homogenized pulp. The 40 ml pulp on volume basis was centrifuged in cooling type centrifuge at 10°C and 5000 rpm of centrifuge speed for 30 min for separation of crude gel and fibre. After centrifuge step was finished, the pulp was filtered by using filtration vacuum pump to obtain pure gel from crude gel through the Whatman sterile membrane filters paper. The pure gel was collected in test tubes for further analysis while crude gel have been dried in an oven at 50°C for 2h.

2.3. Preparation of aloe vera gel
Figure 1 shows the step by step in order to get the aloe vera gel.

![Flow chart of aloe vera gel processing.](image)

2.4. Soxhlet extraction

Table 1. Solvents used during the soxhlet extraction with their physicochemical characteristics [7].

| Polarity    | Solvent | Polarity Index | Boiling Point (°C) |
|-------------|---------|----------------|--------------------|
| Non polar   | Hexane  | 0              | 68.85              |
| Polar       | Ethanol | 5.2            | 78.40              |
As shown in Table 1, two types of solvent were used which were hexane and ethanol. In order to identify the different extracted bioactive compound, both hexane and ethanol were combined in specific ratio. The extraction process was describe in the Figure 2.

**Figure 2.** Flow chart of soxhlet extraction processing.

### 2.5. Quality parameter measurement

Aloe vera gel quality is adjudged by two parameters which is refractive index and viscosity analysis. Therefore, both of these analysis have been discussed in this paper.

#### 2.5.1. Refractive index of aloe vera gel

The ATAGO Refractometer was used for the measurement of refractive index having range of refractive Indices between 1.3000 and 1.7000 with an accuracy of ±0.0002. Aloe vera gel have been dropped on the refractometer prism surface and was closed carefully. Then, click the ‘START’ button and take the reading when the machine stop run. These steps were repeated three times in order to get the average value of the refractive index for both younger and older aloe gel.

#### 2.5.2. Viscosity of aloe vera gel

The viscosity of aloe vera gel is a very important property in the biochemical analysis as it is the indicator of active biological constituents. The Brookfield CAP 2000+ Viscometer was used for the measurement of viscosity of the aloe vera gel. Firstly, a little amount of the aloe vera gel was dropped onto the viscometer prism and closed with the suitable spindle. After that, the excess of aloe vera gel was clean and the machine was run. Then, the value of centipoise, cP and its percentage was taken after the machine stop running. In this analysis, various temperature was recorded in order to determine the better results.

### 2.6. Fourier Transform Infrared (FTIR) spectroscopy

Fourier Transform Infrared was used to identify the chemical bonds by producing an infrared absorption spectrum and it is an effective analytical instrument for detecting the presence of certain functional groups in a liquid aloe vera gel. Liquid aloe vera gel was directly placed in the spectrometer. The samples were recorded in transmittance mode from 4000 to 450 cm⁻¹.

### 2.7. Scanning Electron Microscopy (SEM) and Energy Dispersive X-Ray analysis (EDX)

Scanning electron microscopy was used to observe physical changes in the aloe vera gel samples dried at 65°C while an energy dispersive x-ray was used to provide an elemental identification and
quantitative of the compositional information. All micrograph and element were taken from the surface of the samples by using Zeiss Leo Supra 55 VP. Before the test was performed, the samples were placed on sample holders using conductive carbon tape and coated with gold particles. The samples were observed at different magnifications ranging from 75X to 1000X.

3. Results and Discussion

3.1. Refractive index of aloe gel

| Types of aloe vera gel | Refractive Index |
|-----------------------|-----------------|
| Younger               | 1.3337±0.0002   |
| Older                 | 1.3334±0.0002   |

Table 2 presents the average value of the refractive index of aloe vera gel which are 1.3337±0.0002 for the younger and 1.3334±0.0002 for the older. In this case, gel with lowest refractive index is the best treatment for extraction process. It is because refractive index indicates the impurities in the extracted gel [8]. Thus, it can be concluded that older aloe vera gel is the best treatment for the extraction process.

3.2. Viscosity of aloe gel

| Temperature (°C) | Younger aloe gel | Older aloe gel |
|-----------------|-----------------|---------------|
|                 | centipoise (cP) | Pure Gel Recovery (%) | centipoise (cP) | Pure Gel Recovery (%) |
| 25              | 29              | 2.2            | 38              | 2.9            |
| 23              | 32              | 2.4            | 45              | 3.4            |
| 21              | 32              | 2.4            | 51              | 3.9            |

The viscosity of gel recorded at various temperature are presented in Table 3. Chandegara and Varshney have stated in previous paper that aloe vera is known as a non-Newtonian fluid, thus all the test parameter will be affected [9]. Therefore, various temperature have been chosen in this study to determine the better viscosity. From the result, it shows that when the temperature decreased from 25°C to 21°C, the value of the centipoise and percentage of pure gel recovery for both younger and older aloe gel were increased.

Younger aloe gel shows that the value of centipoise and pure gel recovery increased during the temperature changing in between 25°C to 23°C from 29 cP and 2.2% to 32 cP and 2.4%. However, the values does not change when the temperature was decreased to 21°C. Whilst for older aloe gel, it is clearly shows that the value of centipoise and pure gel recovery also increased when the temperature decreased. But both of the centipoise and pure gel recovery values for older aloe gel are larger compared to younger aloe gel. At temperature 25°C, the value of centipoise and pure gel recovery is 38 cP and 2.9% higher than younger aloe gel. When the temperature decreased to 23°C, centipoise value becomes 45 cP and pure recovery gel increased to 3.4%. While at 21°C, the value of centipoise is 51 cP and the pure recovery gel is 3.9%.

Thus, it can be concluded that older aloe vera gel possesses better quality of the biological active component due to the higher amount of the viscosity [9]. Previously, the average values of the dynamic viscosity for gel was found around 35.33±0.21 to 36.45±0.34 cP [8].

3.3. Fourier Transform Infrared (FTIR) spectroscopy

Figure 3 shows the FTIR spectra of unfiltered and filtered of 4 years old aloe vera gel. For the unfiltered aloe vera gel, it exhibits a broad absorption band at 3223 cm⁻¹ due to the stretching of –OH groups. It also shows the symmetrical and asymmetrical C-H stretching at 2127 cm⁻¹ and C=O stretching at 1652 cm⁻¹.
After that, filtered aloe vera gel displayed the –OH groups at 3399 cm⁻¹, symmetrical and asymmetrical C-H stretching at 2132 cm⁻¹, C=O stretching at 1646 cm⁻¹, C-O stretches of alkoxy at 1228 cm⁻¹ and C-Br stretching at 578 cm⁻¹. For both unfiltered and filtered aloe vera gel, it shows the C-H bond presents at 735 cm⁻¹, 780 cm⁻¹ and 617 cm⁻¹. This bond is out of plane deformation of carbohydrate monomers which shows that mannose and uronic acids presence along with their carbohydrate polymers such as acemannan and pectins [3].

![FTIR spectrum](image)

**Figure 3.** FTIR for (a) Unfiltered (b) filtered of 4 years old aloe vera gel.

From this result, it can be concluded that both of unfiltered and filtered aloe vera gel dwell a similar bonding. It also can be stated that the unfiltered and filtered aloe vera gel does not give an effect to the bonding contained in aloe vera gel. Based on Lim and Cheong, they stated that –OH group that shown in both unfiltered and filtered aloe vera gel was due to the characteristic of carbohydrate monomers including mannose and uronic acid while C=O indicates the presences of the carbonyl groups in the aloe vera gel. After that, C-O stretching was associated with the rhamnogalacturonan, a side-chain constituent of pectins [3].

3.4. **Scanning Electron Microscopy (SEM) and Energy Dispersive X-Ray analysis (EDX)**

Microstructure images of processed aloe vera gel are reported in Figure 4. Figure 4 (a) and (b) shows the SEM microstructure images of surface aloe vera gel. It was shown that the layer of aloe vera gel consists of organic fibers and particles with irregular and crushed shapes while Figure 4 (c) and (d) shows the image of uniformly lamella structure. This may be due to the degradation of the pectinaceous materials during the extraction process [10].

![SEM images](image)

**Figure 4.** Aloe vera gel examined with SEM (x1000): (a) younger aloe vera gel, (b) older aloe vera gel, (c) aloe gel extracted with hexane and (d) aloe gel extracted with mixture of hexane and ethanol.
EDX analysis shows that the younger aloe vera gel and extracted of aloe vera gel by hexane contained of carbon, oxygen, magnesium, chlorine, potassium and calcium while older aloe vera gel layers showed the addition of the elements that being detected which is sodium. However, the extracted aloe vera gel by mixture of hexane and ethanol does not detect the element of calcium but it being replaced with the element of phosphorus. Table 4 presents summary of the weight percentage of detected elements in the aloe vera gel. From the Table 4, it can be assumed that calcium and potassium are present in the oxide form due to the higher weight percentage of the oxygen in the gels [11].

### Table 4. EDX results for younger, older and extracted aloe vera gel.

| Elements (K) | Younger aloe gel | Older aloe gel | Aloe gel extracted by hexane | Aloe gel extracted by mixture of hexane and ethanol |
|-------------|-----------------|----------------|-----------------------------|--------------------------------------------------|
|             | Wt.% At.%       | Wt.% At.%      | Wt.% At.%                   | Wt.% At.%                                         |
| C           | 57.34 66.52     | 53.65 65.20    | 55.06 64.42                 | 55.04 66.21                                       |
| O           | 35.40 30.82     | 31.99 29.18    | 37.24 32.71                 | 31.90 28.82                                       |
| Mg          | 0.32 0.19       | 0.38 0.23      | 0.49 0.28                   | 0.21 0.13                                        |
| Cl          | 0.47 0.19       | 2.05 0.84      | 0.55 0.22                   | 2.04 0.83                                        |
| K           | 4.77 1.70       | 10.21 3.81     | 4.45 1.60                   | 10.63 3.93                                       |
| Ca          | 1.70 0.59       | 1.33 0.48      | 2.22 0.78                   | - -                                              |
| P           | - - -           | - - -          | 0.17 0.08                   | - -                                              |
| Na          | - - -           | 0.39 0.25      | - - -                       | - -                                              |

4. Conclusion

Characterizations of aloe gel samples were conducted to determine which aloe gel consists the preferred characteristics that make it suitable to act as gel electrolyte in DSSCs. Therefore, two parameters have been discussed in this paper which is refractive index and viscosity of aloe gel. Younger and older aloe vera gel were subjected in this analysis. Older aloe vera gel was selected because it possesses the lower refractive index and consists higher viscosity value which means, older aloe vera gel acquires the characteristics that suitable for electrolyte application.

From the FTIR results, it shows both unfiltered and filtered aloe vera gel contained the similar bonding. –OH groups determine the characteristics of carbohydrate monomers including mannose and uronic acid. Then, C=O indicates the presences of the carbonyl group and C-O stretching indicates the presences of carbohydrate and polymer such as acemannan and pectin [3].

After that, SEM result shows that older and younger aloe vera gel consists of irregular cellulose structure while aloe vera gel extracted by hexane and also extracted by the mixture of hexane and ethanol at specific ratio shows the uniform structure of the fibres. Other than that, EDX analysis result determines the element that contain in both younger and older, also in extracted aloe vera gel. The elements that have been found are carbon, oxygen, magnesium, chlorine, potassium, calcium, sodium and phosphorus. Some of these elements also been founded by Wu and his friends in the aloe gel such as calcium, magnesium, zinc, chromium and selenium. They stated that aloe gel contains 20 minerals in it and it was able to generate electricity due to the electrolysis process that take place in the gel [12].

Therefore, aloe vera gel can be classified as a preferable organic material to act as an electrolyte in the DSSCs based on elements possesses that being shown in EDX results and also aloe vera gel properties itself. This aloe vera gel will be used in the electrolyte application for DSSCs.

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