

Modified Polystyrene Waste as Cation Exchange Material and Its Characterization

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Abstract

Modification of polystyrene (PS) waste into sulfonated polystyrene (PSS) as cation exchange material and its characterization has been carried out, the polystyrene waste used is styrofoam from air conditioner packaging. Modification is carried out through the sulfonation process of styrofoam using acetyl sulfate as a sulfonating agent in tetrahydrofuran (THF) solution. Sulfonation is carried out at a temperature of 40 °C for 3.5 hours under nitrogen gas flow. The modified product is then characterized using FTIR, SEM, and testing the degree of sulfonation, water absorption capacity, and cation exchange capacity (CEC). The results show that modification has occurred in the presence of a sulfonate group ($-\text{SO}_3\text{H}$) which is characterized by the appearance of characteristic peaks of 1033 cm^{-1} , 1162 cm^{-1} and 3329 cm^{-1} in the FTIR spectrum and SEM results show that the PS structure which is not rough and becomes a rougher and non-uniform structure after sulfonation. The degree of sulfonation obtained reached 58%, the maximum water absorption achieved was 39% after soaking for 72 hours, and the highest measured cation exchange capacity was 1.87 meq/g in 1.5 N NaCl solution. These results indicate that styrofoam waste has the potential to be used as a basic material for efficient and environmentally friendly cation exchange resins.

Keywords: Polystyrene, Sulfonation, Acetyl sulfate, Cation exchange capacity, Activation energy

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Modifikasi limbah polistirena (PS) menjadi polistirena tersulfonasi (PSS) sebagai material penukar kation dan karakterisasinya telah dilakukan. Limbah polistirena yang digunakan adalah styrofoam dari kemasan pendingin udara. Modifikasi dilakukan melalui proses sulfonasi styrofoam menggunakan asetil sulfat sebagai agen sulfonasi dalam larutan tetrahidrofuran (THF). Sulfonasi dilakukan pada suhu 40 °C selama 3,5 jam di bawah aliran gas nitrogen. Produk hasil modifikasi kemudian dikarakterisasi menggunakan FTIR, SEM, dan pengujian derajat sulfonasi, kapasitas penyerapan air, dan kapasitas penukar kation (CEC). Hasil penelitian menunjukkan bahwa modifikasi telah terjadi dengan adanya gugus sulfonat ($-\text{SO}_3\text{H}$) yang ditandai dengan munculnya puncak karakteristik pada 1033 cm^{-1} , 1162 cm^{-1} dan 3329 cm^{-1} pada spektrum FTIR dan hasil SEM menunjukkan bahwa struktur PS yang tidak kasar menjadi lebih kasar dan tidak seragam setelah sulfonasi. Derajat sulfonasi yang diperoleh mencapai 58%, penyerapan air maksimum yang dicapai adalah 39% setelah perendaman selama 72 jam, dan kapasitas pertukaran kation tertinggi yang diukur adalah 1,87 meq/g dalam larutan NaCl 1,5 N. Hasil ini menunjukkan bahwa limbah styrofoam berpotensi digunakan sebagai bahan dasar untuk resin penukar kation yang efisien dan ramah lingkungan.

Kata Kunci: Polistirena, Sulfonasi, Asetil sulfat, Kapasitas pertukaran kation, Energi aktivasi

INTRODUCTION

Polystyrene (PS) is one of the most sought-after thermoplastics globally, ranking behind polyethylene, polypropylene, and polyvinyl chloride. The high demand for polystyrene is driven by its superior overall characteristics, ease of processing, and sustainable production practices [1]. The disadvantages of polystyrene are that it is slow to biodegrade and is not widely recyclable. The “single-use” nature of many of its packaging end-uses and its presence as waste in the environment mean that its use in some of these contexts, such as takeaway food containers, is controversial. As a result of these concerns, as well as due to competition from other types of plastics such as polypropylene and polyethylene terephthalate, the PS market in some industrial sectors has experienced declining interest, overcapacity, and underutilization. [2-5].

Due to the very slow degradation of PS, another option that has been considered is PS pyrolysis. This has also been reported to be very good for pyrolysis, produce the highest liquid oil production, more than 90%, start to degrade at the lowest temperature of 350 °C, have the highest calorific among all plastics (above 40 MJ/kg), have catalysts that improve the quality and quantity of oil, and are suitable for copyrolysis with biomass wood [6]. The downside is that the waste is in the form of gas, which is difficult to control. This is because impurities in PS can poison the surrounding environment if handled improperly. Because it is difficult to degrade, another way to utilize polystyrene is to convert it into sulfonated polystyrene.

In fact, many studies related to polystyrene sulfonation have been reported, such as superplasticizers to improve fluidity and workability based on partially sulfonated polystyrene for calcium sulfate hemihydrate [7]. Other researchers have also reported on polystyrene sulfonation and its characterization using Raman spectroscopy [8]. Previous researchers have also reported on the preparation and characterization of sulfonated polystyrene/magnetite nanocomposites for the adsorption of organic dyes [9], apart from that, there are also researchers who reported the synthesis of sulfonated polystyrene-based porous activated carbon for organic dyes removal from aqueous solutions [10].

The latest developments have been reported by many researchers regarding polystyrene sulfonation as a proton-conducting membrane material, membranes for fuel cells [11]. Research on the use of sulfonated polystyrene as a cation exchange has been reported, but in the form of a membrane where this membrane

will be used as a membrane in fuel cells [12]. There is another research report, namely Optimization of the sulfonation process of polystyrene waste for hardness and heavy metal removal [13].

Previous research reports [14] have carried out sulfonation of polystyrene with a maximum sulfonation degree of 1.96%, in contrast to previous research reports which have not reported activation energy. From measuring the activation energy of sulfonated polystyrene, it is hoped that it can improve performance and optimize the utilization of sulfonated polystyrene and hoped that this research will provide additional information regarding the activation energy of cation exchange. As mentioned before, PS degradation takes a long time. PS's use is broad, ranging from the food industry and electronics to packaging for chemicals and cosmetics, which results in polystyrene waste. To address this issue, it is proposed to modify polystyrene into sulfonated polystyrene for other uses, such as ion exchange.

MATERIALS AND METHODS

Materials

The materials used in this study were: Styrofoam waste collected from electronic product packaging, dichloromethane, acetic anhydride, sulfuric acid, tetrahydrofuran, methanol, sodium chloride, and sodium hydroxide.

Polystyrene Preparation

Sample preparation begins by crushing polystyrene (Styrofoam) into small pieces, then the polystyrene is soaked in 300 mL of acetone to clean it from impurities, then dried to evaporate the remaining solvent and change the physical form of the polystyrene to become more compact. After that, the polystyrene is dried in an oven at a temperature of 80 °C for 1 hour, and the sample is stored in a desiccator.

The molecular mass of polystyrene was calculated using the viscometry method. Polystyrene weighing 0.05 g, 0.1 g, 0.15 g, 0.2 g, and 0.25 g was dissolved in 5 mL THF solution to obtain concentrations of 1 g/dL, 2 g/dL, 3 g/dL, 4 g/dL, and 5 g/dL. Viscosity was measured using an Ostwald Viscometer at 25 °C by calculating the flow time of THF as a solvent and the flow time of polystyrene solution as a concentration. Relative viscosity was determined by comparing the flow time of polystyrene solution with the flow time of solvent. First, the specific viscosity of each concentration was determined, followed by determining the reduced viscosity, and finally determining the intrinsic viscosity by creating a curve of the relationship

between reduced viscosity vs. C (concentration). The point that intersects the y-axis is the intrinsic viscosity, namely the reduction viscosity when C=0. If the intrinsic viscosity has been obtained, the M can be determined using the Mark-Houwink equation, namely as follows:

$$[\eta]=K \times M^a \dots\dots\dots(1)$$

where:

- [η] = intrinsic viscosity (dL/g)
- M = relative molecular mass of polystyrene
- K and a = couple constant between solvent and polystyrene.

Synthesis of Sulfonated Polystyrene (PSS)

The sulfonation agent used was acetyl sulfate solution. This solution was prepared by mixing 10 mL of dichloromethane and 4.1 mL of acetic anhydride in a three-necked flask under inert conditions using nitrogen gas (N₂). The solution was cooled to 0 °C and then 6.5 mL of 98% H₂SO₄ was added with careful stirring until a homogeneous solution was obtained. The clean polystyrene was dissolved with the solvent in the reactor while stirring at a constant speed using a magnetic stirrer until the polystyrene was completely dissolved in the solvent (homogeneous). The solvent used was THF. 11 g of PS was dissolved in 100 mL of THF until homogeneous. After homogeneity, acetyl sulfate as a sulfonation agent was added to the solution as much as 5 mL at temperature of 40 °C. The sulfonation process was carried out under a stream of nitrogen gas and the flow was maintained throughout the sulfonation reaction.

After 3.5 hours of reaction, the product was taken and precipitated into 100 mL of methanol while stirring. The methanol was then evaporated by letting it dry for \pm 7 days. After the evaporation process was complete, the sulfonated polystyrene was oven-dried at 80°C until it became dry and hard, then stored in a desiccator. The dried PSS was ground using a mortar and pestle, then sieved using a 100mesh sieve to obtain a uniform particle size.

The degree of sulfonation of sulfonated polystyrene was determined by the titration method. 0.1 g of sulfonated polystyrene was first weighed and then dissolved in 20 mL of THF solvent. Before titrating the PSS solution, 2-3 drops of phenolphthalein indicator were added. Then, it was titrated with standardized 0.01 N NaOH and stopped when the titration endpoint was reached, which was indicated by a color change of the indicator [15].

Water absorption capacity and cation exchange capacity

Water absorption capacity was performed by measuring the weight of water absorbed by the resin after being immersed in demineralized water for a specified period of time. A 0.1 g sample of resin was weighed and then immersed in demineralized water for 24 hours, stirring with a magnetic stirrer to allow water absorption. After the soaking period was complete, the resin was removed, drained, and reweighed to determine its weight after absorbing water [16]. The above steps were also repeated for the 48 and 72 hours.

Cation Exchange Capacity

The cation exchange capacity of PSS was determined using the acid-base titration method. 0.2 g of PSS was weighed and added to a 0.5 N NaCl solution and stirred for 48 hours to allow the exchange of H⁺ protons with Na⁺. The proton content of the solution was then determined by titration with a standardized 0.01 N NaOH solution. Before titration, 2-3 drops of phenolphthalein indicator were added to the PSS mixture with the salt solution. The steps were also carried out for variations in NaCl concentration of 1 N and 1.5 N.

Determination of activation energy (Ea) is carried out through CEC analysis based on the Arrhenius equation. CEC measurements of PSS are carried out at various temperatures ranging from 25 °C, 50 °C, and 75 °C. The Arrhenius equation follows the following formula:

$$K = A \exp Ea/RT \dots\dots\dots(2)$$

Then the equation is integrated to:

$$\ln (k) = \ln (A) - Ea/R \times 1/T \dots\dots\dots(3)$$

where:

- k = rate constant (cation exchange capacity, CEC)
- A = pre-exponential factor
- Ea = activation energy
- T = temperature
- R = universal constant (= 8.314 J/mol.K)

Functional groups were analyzed using Fourier transform infrared spectroscopy (FTIR) (Bruker Alpha II, Platinum ATR) in the range of 4000-400 cm⁻¹. Surface morphology was examined using a scanning electron microscope (SEM, Thermo Scientific).

RESULTS AND DISCUSSION

Polystyrene Preparation

After soaking, significant changes were found in the physical properties of polystyrene. Before soaking, polystyrene had a soft texture and low flexibility. After soaking in acetone, polystyrene underwent changes, polystyrene which originally had a porous structure turned into a solid mass, its density increased, with a plastic and somewhat elastic texture when still wet. The volume of polystyrene experienced a very drastic shrinkage, leaving only a small portion of its initial volume. The changes in the physical form of polystyrene are shown in **Figure 1**.

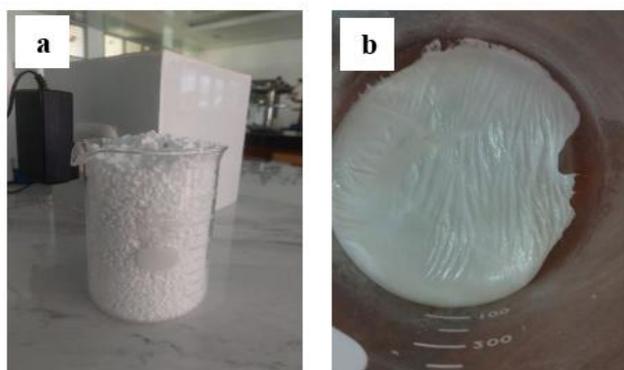


Figure 1. Physical changes of polystyrene a) before being soaked in acetone and b) after being soaked in acetone

The change process shows that immersion of polystyrene in acetone triggers the release of gas from the pores and the dissolution of the polystyrene structure as its main polymer. This result is in line with previous research [17], which shows that immersion of PS in acetone causes a drastic defoaming process due to the release of gases such as n-butane or n-pentane, followed by changes in the physical properties of the material from elastic solid to viscous plastic.

The determination of the molecular mass of polystyrene was carried out by measuring the flow time of the polymer in THF solvent using several concentration variations, namely 1 g/dL, 2 g/dL, 3 g/dL, 4 g/dL, and 5 g/dL. In the application of the Mark-Houwink equation, the values of a and K are constants for polystyrene with THF solvent at a temperature of 25°C, namely $a = 0.70$ and $K = 14.1 \times 10^{-3}$. The flow time of the THF solvent is 2.12 seconds, while the flow time of each polymer solution at a concentration of 1 g/dL, 2 g/dL, 3 g/dL, 4 g/dL, 5 g/dL are 3.55, 5.45, 7.92, 11.26, and 14.11 seconds, respectively.

From the **Figure 2**, an intercept of 0.5538 dL/g or 5538 mL/g is obtained which is the intrinsic

viscosity $[\eta]$ of polystyrene in THF solvent. This is then entered into the Mark-Houwink equation, so that the polystyrene molecular mass can be determined. The results of the calculation obtained the relative molecular mass of polystyrene, namely 134959.98 g/mol.

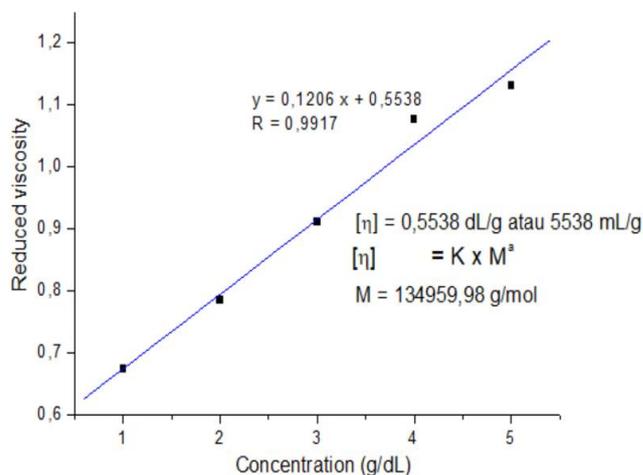


Figure 2. Concentration - Reduced Viscosity Relationship Curve.

Synthesis of Sulfonated Polystyrene (PSS)

The synthesis of polystyrene into sulfonated polystyrene is carried out by adding a sulfonating agent, namely acetyl sulfate. Acetyl sulfate acts as a sulfonating agent formed through the reaction between acetic anhydride and concentrated sulfuric acid in dichloromethane solvent. In this reaction, acetic anhydride functions to bind water so that it does not participate in the reaction, because of its hygroscopic nature. The process of forming acetyl sulfate must be carried out under inert conditions, to avoid unwanted reactions. Therefore, during the reaction, nitrogen gas (N_2) is circulated to maintain an inert atmosphere. In addition, the reaction temperature is maintained at 0 °C to avoid uncontrolled boiling (bumping) which can cause splashing during the addition of concentrated sulfuric acid to the reaction mixture. Immersing the reactor with ice water aims to maintain the temperature during the reaction, preparation of acetyl sulfate have shown in **Figure 3**. The reaction of acetyl sulfate and polystyrene is shown in **Figure 4**.

The sulfonation reaction is a substitution reaction that aims to replace the H atom with the $-SO_3H$ group in polystyrene. The polymer and sulfonating agent must be in the same phase. Sulfonated polystyrene will have a $-SO_3H$ group in polystyrene causing sulfonated polystyrene to easily release H^+ ions.



Figure 3. a) Preparation of acetyl sulfate and b) acetyl sulfate solution

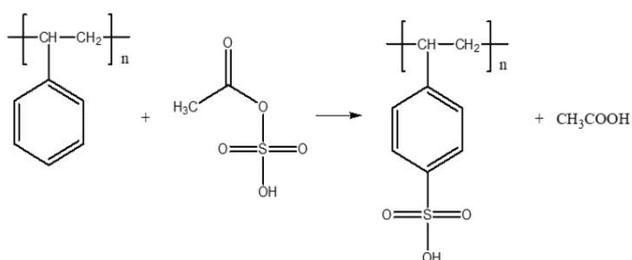


Figure 4. Sulfonation reaction of polystyrene with acetyl sulfate

Sulfonated polystyrene has a sulfonate group that can exchange cations, so it is included in the category of cationic ion exchange resins. After the sulfonation process is complete, the reaction product is then precipitated using methanol while stirring. The purpose of precipitation is to precipitate the reaction product into a solid on the surface of the container. The reaction product is air-dried until only the precipitate remains, which is then heated using an oven at 60 °C for 1 hour to evaporate the remaining methanol in the precipitate, leaving only the PSS precipitate. The PSS precipitate was dried again at 80 °C for 4 hours to allow the byproducts of the sulfonation reaction to completely evaporate. Due to its hygroscopic nature, the sulfonation product, sulfonated polystyrene (PSS) or resin, must be stored in a desiccator after drying. The PSS was then ground using a mortar and pestle until smooth, then sieved through a 100-mesh sieve to obtain uniform PSS resin particle sizes. The resulting PSS resin was brownish in color.

The degree of sulfonation in this study reached 58%, indicating that the sulfonation process successfully bound the sulfonate group (-SO₃H) of more than half of the polymer chain as a whole, which showed a higher degree of sulfonation compared to previous studies that used acetyl sulfate as a sulfonation agent of 36.7%. This difference in the

degree of sulfonation could possibly occur due to the type of polystyrene, the solvent used, and the use of N₂ gas flow during the sulfonation process which was different from the study [18].

Water absorption capacity

The initial mass of PSS is 0.1 g, water absorption capacity with different immersion times showed an increase in mass of 0.124 g, 0.130 g, and 0.139 g, respectively. Based on the results of the water absorption test over time, PSS resin had the highest water absorption capacity at 39% at 72 hours and the lowest at 24 hours at 24%, as calculated using the water absorption formula. The increase in resin mass during the immersion process indicates that the resin is able to absorb water gradually. This indicates that the resin has a porous structure that can absorb and retain water molecules, due to the presence of sulfonate groups (-SO₃H) from sulfonation in the polystyrene chains, which are hydrophilic. These groups interact with water through hydrogen bonding, allowing water to penetrate the resin matrix. The higher the sulfonate group content, the higher the water absorption capacity of the resin. The degree of sulfonation of 58% allows the interaction of the -OH group from -SO₃H to interact with water, but this does not happen optimally. This can be made possible by the nature of the internal structure of the sulfonated polystyrene produced. The higher the sulfonate group content, the higher the water absorption capacity of the resin, this is different from the truth. The absorption capacity of 39% does not correlate with the degree of sulfonation of 58%. This water absorption capacity does not occur optimally. This is made possible by the nature of the internal structure of the sulfonated polystyrene produced.

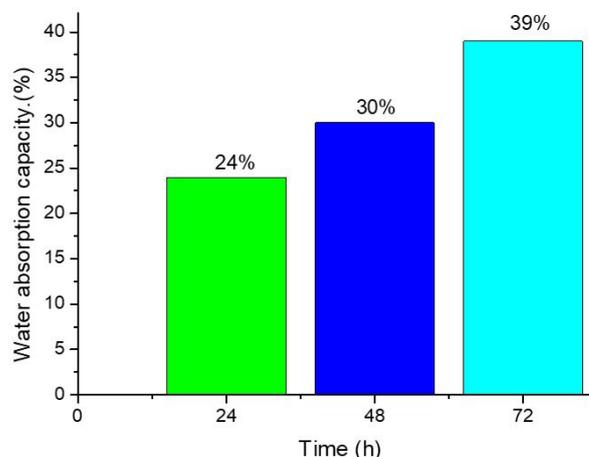


Figure 5. Curve of the relationship between soaking time and water absorption capacity

From **Figure 5**, shows the relationship between immersion time and water absorption. The relatively water absorption capacity of the PSS resin indicates that the sulfonation process was successful and resulted in a resin structure capable of interacting with water. The water absorption capacity was 39% for 72 hours. The water absorption result of this study at 24 hours was 24%, this is higher than that reported in previous studies with water absorption for 24 hours of 9.04% [14]. This behavior of PSS towards water absorption is similar to the general behavior of resins that have been reported [19] with different materials, that the resin shows an increase in water absorption over time without damaging the resin structure. From research behavior [19] it is possible that there will be an increase in water absorption if the absorption time is increased to 96 hours, up to the maximum capacity limit.

Cation Exchange Capacity and activation energy

From **Figure 6** shows an increase in the CEC, indicating that the concentration of the NaCl solution plays an important role in increasing the efficiency of ion exchange between the PSS sulfonate group and sodium ions from the solution. The increase in the CEC is caused by the increase in the number of Na⁺ ions present in the system with increasing solution concentration, resulting in a faster exchange of H⁺ ions bound to the sulfonate group with Na⁺ ions. The cation exchange capacity is greatly influenced by the degree of sulfonation of polystyrene. In contrast to research [20], the sulfonation agent used previously was trimethylsilyl chlorosulfonate which produced a degree of sulfonation of 73.61% so that the cation exchange capacity was quite high.

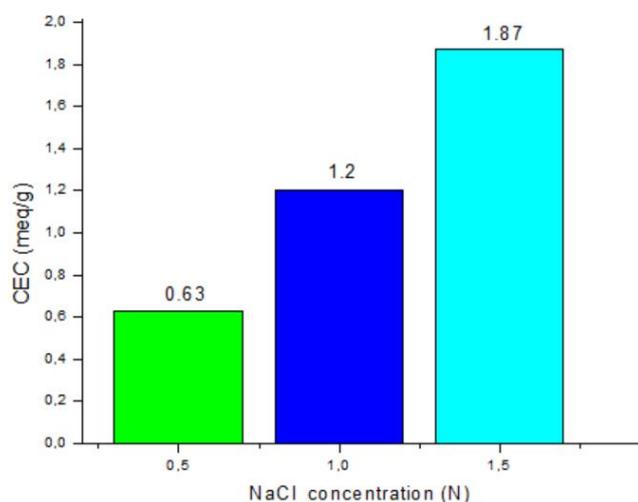


Figure 6. Effect of NaCl concentration on CEC of sulfonated polystyrene

In cation exchange, activation energy is the energy required for ions to overcome the energy barrier and interact with the active site of the cation exchange material. The activation energy obtained from the calculation provides information about the sensitivity of the cation exchange process to temperature changes. The determination of activation energy uses the Arrhenius equation, which relates $\ln(\text{CEC})$ to temperature and activation energy.

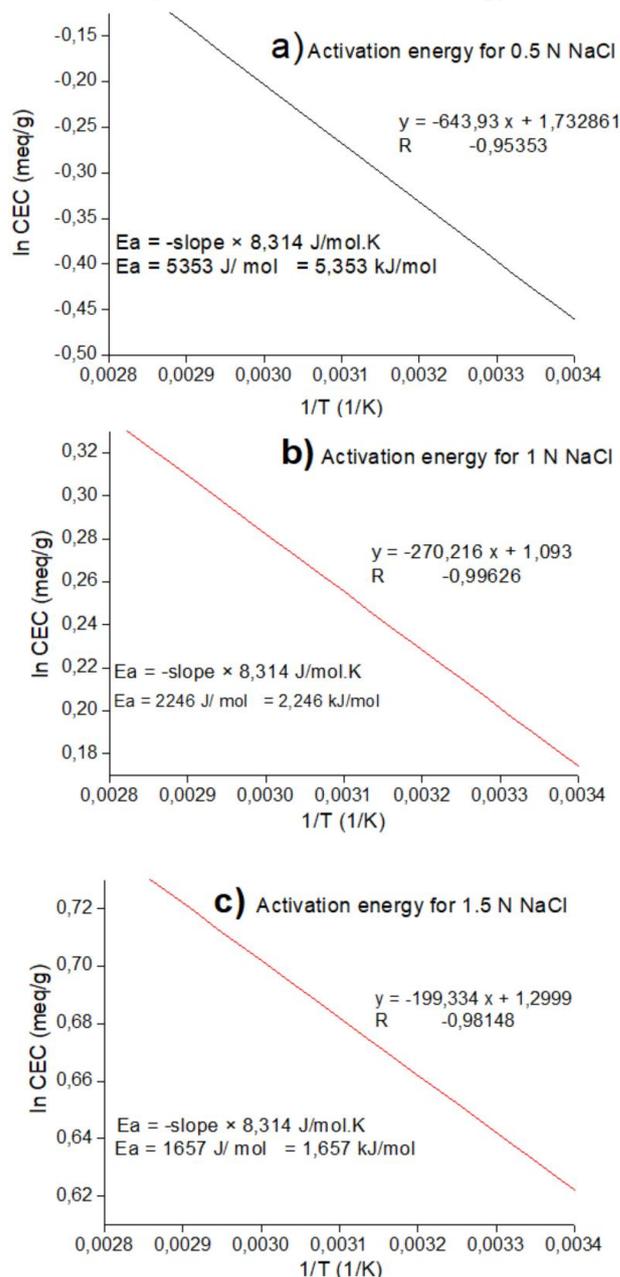


Figure 7. The relationship between $\ln(\text{CEC})$ and $1/T$ for a) 0.5 N NaCl b) 1 N NaCl and c) 1.5 N NaCl

The relationship between $\ln(\text{CEC})$ and $1/T$ at various NaCl concentrations including 0.5 N NaCl, 1

N NaCl, and 1.5 N NaCl, as shown in **Figure 7**, is used to analyze the effect of temperature on cation exchange capacity and subsequently calculate the activation energy.

The slope of E_a/R obtained is used to find the activation energy (E_a) with R being the ideal gas constant. Based on the graphical analysis of the relationship between $\ln(\text{CEC})$ and $1/T$, different slopes are obtained for each NaCl variation. At $\ln \text{CEC}$ of 0.5 N and $1/T$, the slope of the line is -643.93 (**Figure 7 a**), while the slope of the $\ln \text{CEC}$ curve of 1 N and $1/T$ is -270.22 (**Figure 7 b**), and there is a difference in the slope in the graph of the relationship between $\ln \text{CEC}$ of 1.5 N and $1/T$ which shows of -199.33 (**Figure 7 c**) By using the slope and the ideal gas constant ($R = 8.314 \text{ J/mol.K}$), the activation energy (E_a) for the cation exchange process produces E_a 5.353 kJ/mol, 2.246 kJ/mol, and 1.657 kJ/mol, respectively. The relatively low activation energy indicates that the cation exchange reaction in the resulting material does not require a large amount of energy to occur, so the cation exchange process can occur relatively easily.

Activation energy can arise when there are collisions among molecules. The greater the probability of intermolecular collisions, the more likely it is that intermolecular interactions will exceed the activation energy, meaning a lower activation energy. This finding aligns with earlier studies [21], which indicate that a higher concentration enhances the chance of molecular collisions, thereby resulting in decreased activation energy.

Analysis of PS and PSS Functional Groups Using FTIR

The functional groups of PS and PSS were analyzed by comparing the wavenumber absorption recorded in the spectra of each polymer functional group before and after sulfonation. The FTIR spectrum analysis of PS in **Figure 8** shows characteristic peaks for functional groups found in specific wavebands. The absorption at wavenumber 3026 cm^{-1} indicates aromatic C-H stretching vibrations, the peak at approximately 2916 cm^{-1} indicates aliphatic C-H stretching vibrations of the phenyl group and the polystyrene main chain, and the absorption at approximately 1598 cm^{-1} and 1447 cm^{-1} are associated with aromatic C=C stretching vibrations.

After sulfonation, the FTIR spectrum of PSS shows new peaks, characterized by absorption peaks at wavenumbers 1033 cm^{-1} and 1162 cm^{-1} . The appearance of peaks indicates the formation of

sulfonate groups ($-\text{SO}_3\text{H}$) in the polystyrene structure, which confirms the success of the sulfonation process. The peak at 1033 cm^{-1} is a symmetric stretching vibration of S=O, while the peak at 1162 cm^{-1} is related to the asymmetric stretching vibration of S=O or S-O stretching vibration. The FTIR spectrum of PSS shows a wave number at 3329 cm^{-1} with a broad peak indicating the presence of O-H stretching vibrations. The results obtained are in line with previous reports [22], that the FTIR spectrum of PS, shows peaks at 3025 cm^{-1} , 2916 cm^{-1} , 1600 cm^{-1} , and 1492 cm^{-1} , indicating the presence of aromatic C-H and C=C vibrations. After the sulfonation process, new peaks appeared at 1034 cm^{-1} and 1163 cm^{-1} which indicated the presence of sulfonate groups (S=O).

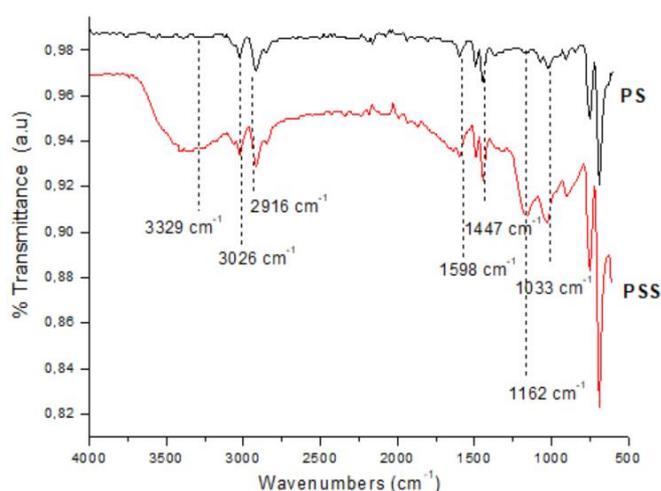


Figure 8. FTIR spectra of PS and PSS

Morphological Analysis of PS and PSS Using SEM

Figure 9a shows a relatively homogeneous and not rough surface morphology of PS. The PS surface appears to show pores or cracks throughout the observed area. This surface corresponds to the characteristics of pure polystyrene, namely hydrophobic and having a dense and compact polymer structure. The homogeneity of the PS surface indicates the polymerization process of styrene, which produces linear semi-crystalline polymer chains. The absence of protrusions and the presence of pores indicate that in this observation there are no phases or other components that are separated from the main polystyrene matrix.

The SEM results of sulfonated polystyrene (**Figure 9b**) show morphological changes. The PSS surface becomes rougher compared to PS and contains pores, with irregularly shaped protrusions and the formation of small clumps scattered throughout the surface area.

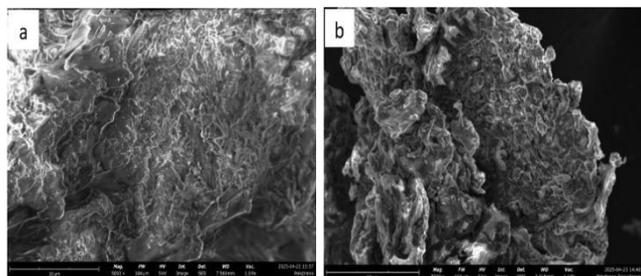


Figure 9. Morphology of a) polystyrene and b) sulfonated polystyrene

This non-uniform surface texture indicates modifications to the polymer structure due to sulfonation. The sulfonation process, which involves the addition of hydrophilic sulfonic groups ($-SO_3H$) to polystyrene chains, causes modifications to the physical properties of the polystyrene surface. The addition of these polar groups can disrupt the hydrophobic interactions between the PS polymer chains. This result is in line with research [23], where sulfonation causes changes in the surface morphology of polystyrene.

CONCLUSION

Sulfonated polystyrene (PSS) derived from electronic packaging waste was successfully obtained with a sulfonation degree of 58% using the sulfonating agent acetyl sulfate. The substitution of H atoms in polystyrene by sulfonate groups increased the hydrophilicity of polymer, as demonstrated by the maximum water absorption of 39% after 72 hours. The cation exchange capacity increased from 0.63 to 1.87 meq/g with increasing NaCl concentration, confirming the enhanced ion exchange performance of PSS and its suitability as a cation exchange resin. The higher the NaCl concentration, the lower the activation energy, a logical consequence of the higher frequency of chemical species collisions.

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