

Polysulphide-based Coating for Liquefied Petroleum Gas Sensor Improvement

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Abstract

Sulphur can be found in various applications such as fertilizer, pesticides, cosmetic, etc. However, due to the toxicity and unpleasant odour of sulphur, its use is limited, leading to an abundance of unutilized sulphur especially from petroleum refining process. Therefore, this study intends to apply the unutilized sulphur for other application by developing a polymeric sulphur-based coating for gas sensor enhancement in term of detection of liquefied petroleum gas (LPG). The polysulphide coating was fabricated using green strategy-based reverse vulcanization process approach by employing palm oil as a medium for the sulphur modification process with a 10:4:2 ratio of sulphur, epoxy resins, and palm oil, respectively. The analyses of surface morphology and functional group of resulting modified sulphur were characterized by using Scanning electron microscopy (SEM) and Fourier-transform infrared spectroscopy (FTIR) respectively. The sensitivity of prepared modified sulphur as a coated on MQ-6 gas sensor was evaluated and compared to an uncoated gas sensor. The feasibility of the coating was justified where coated gas sensor detects gas at 0.94 seconds while uncoated gas sensor detects at 3.00 seconds which also increased the hydrophobicity to protect the sensor.

1. Introduction

One of the sources of sulphur production comes from petroleum refinery process, which was from the production of hydrogen sulphide and sulphuric acid [1]. Despite being used in many applications such as fertilizer [2][3], cosmetics [4], pesticide[5] etc., sulphur has been known to be toxic and corrosive [6]. This has led to decreasing demand for sulphur where there was huge amount of sulphur which required to be disposed or treated as chemical waste which were estimated to be around 107 tonnes per year [7]. Many approaches have been implemented to counter this issue such as by developing the unused sulphur into various alternative applications as in to be used as starting materials for polymer fabrication or being modified to be a safer and eco-friendly material[1][8][9][3].

Recently, the use of sulphur-rich based polymers or polysulphide has attracted significant interest due to their flexibility in structures and their functional properties[10][8][11][12]. Several studies have been attempted with the intention to utilize the octa sulphur (S_8) for the preparation of polymeric materials based on the ring opening polymerization of sulphur. As the temperature of the reaction reached $> 160^\circ\text{C}$, the octameric structure will break and free di-radicals will attack another S_8 molecules [8]. The process was repeated several times until a linear polymer with an S-S bond backbone was discovered [13]. However, the polymer fabricated is unstable since the reaction of terminal free radicals is vulnerable and lead to depolymerize into original cyclic sulphur form.

Therefore, the chain requires to be stabilized by preventing free radicals from reacting with dienes [19]. Several approaches of using natural dienes including cardanol benzoxazines [14], limonene [15], canola oil [16] and myrcene [12], as well as synthetic dienes like styrene [17], 1,4-diphenylbutadiene [18] and 1,3-diisopropenylbenzene (DIB)[1] have been attempted and shows significant impact in retaining the linear polymer properties.

Epoxy resins on the other hand are a class of thermosetting polymers widely used for structural application [19]. Due to its high chemical resistance and good mechanical and physical properties [19], it is widely used in many applications such as coatings, adhesives, matrix materials for composites, and electronic insulators [20]. However, as epoxy resins are inherently brittle because of their highly cross-linked structure, a great effort has been made to improve the fracture toughness [19]. In order to improve brittleness, polysulfide rubber can be added in liquid form, as it is compatible with epoxy resins [21].

Due to its unsaturated fatty acid portion, unsaturated palm oil is used as a comonomer in an inverse vulcanization process [22][23]. As a result, terminal free radicals cannot depolymerize into their original cyclic forms [20]. This double bond contributes to the unsaturation of the oil. The presence of the double bonds makes the polymer more resistant to depolymerization by providing cross-linking within the polymer structure, thus enhancing its stability [24]. In this way, the polymer chains are prevented from easily breaking apart due to the cross-linking. By copolymerizing palm oil with elemental sulphur, it is possible to discover new applications for sulphur, as well as to produce cheap polymers that can replace petro-based polymers in numerous applications [25].

Previously, there have been several attempts to enhance the sensitivity of the gas sensor especially to be used for detection of liquified gas petroleum (LPG) such as butane or propane. These gases are flammable and prone to create explosion if reacted with fire. Previous studies have shown significant impact of using sulphuric based polymeric material as coating to enhance the sensitivity of the sensor [26]. It was discovered that by having the sulphur, the sensitivity of the sensor was increased due to their large surface area, resulting in higher sensitivity of the gas sensor [27]. In addition, by having a coating it can prevent the sensor material from oxidizing at high temperatures, which can adversely affect its performance [28].

Hence, this study was intended to investigate the performance of the developed polysulphide coating for gas sensor modified from epoxy resin as the source of polymer and was treated by using unsaturated palm oil as solvent. The development of polysulphide by using dimethylsulfoxide (DMSO) as solvent also was done to compare the feasibility of using synthetic solvent to the synthesis of the polysulphide. The coating was then characterised to investigate the morphology, the functional group which impacted the stability of the added sulphur to the epoxy resin as well as the performance of coating to enhance the sensitivity of the gas sensor of LPG.

2. Material and Method

Analytical grade of sulphur (TamCahaya, Malaysia), epoxy resins (X'traseal, Malaysia) as polymer source material, commercial palm oil (Saji, Malaysia) and dimethyl sulfoxide (DMSO) (QREC, Malaysia) as medium for sulphur modification were used.

2.1 Preparation of Polysulfide

10g of sulphur was melted for 10 minutes at 170°C on a hot plate (IKA, Germany). Then, 4mL of palm oil was added to the molten sulphur with vigorous mechanical stirring at 250 rpm on a magnetic stirrer (IKA, Germany) while maintaining the temperature at 160°C until the solution turned into an orange hue indicating ring-opening chains has reacted with the palm oil [20]. Then, 2mL of epoxy resins was added to the sulphur-oil mixture at about 150°C until it became a viscous texture. The mixture of polysulfide solution obtained was then left to cool at room temperature [7]. The steps for the synthesis of polysulphide by using DMSO was repeated by similar method as aforementioned to compare the solubility of the sulphur by using synthetic solvent [31].

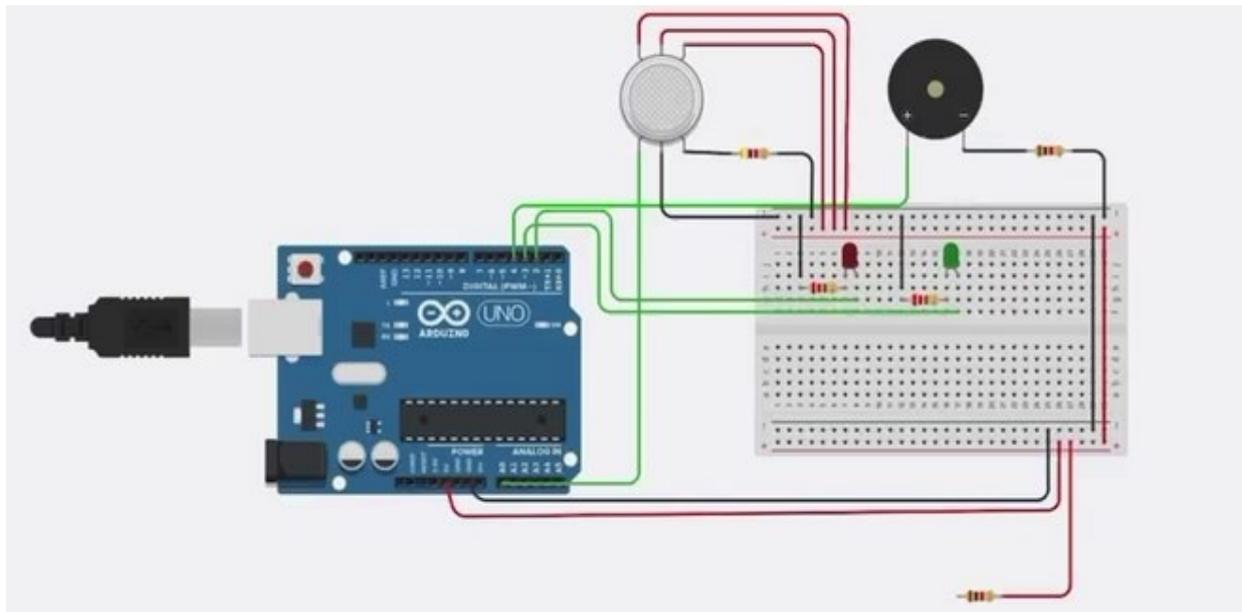
2.2 Characterization of Polysulphide

Attenuated Total Reflectance-Fourier Transform Infrared (ATR-FTIR) (Perkin Elmer, U.S.A) was used to investigate the organic functional group of the prepared polymeric sulphur and was carried out in the 500 – 4000 cm^{-1} wavenumber range [7]. The morphology of polysulfide was determined by using Scanning Electron Microscopy (SEM) (COXEM DVIA-T56, South Korea) at 20kV with 1000 magnifications and was sputtered with gold.

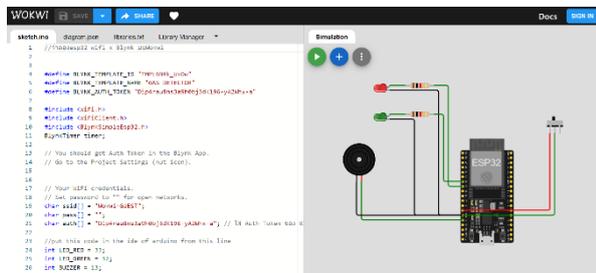
2.3 Preparation of Sensor

MQ-6 gas sensor was purchased from IconTech (Malaysia) and was connected to Arduino UNO board (Italy) directly where the Voltage Common Collector (VCC) pin, ground (GND) pin and analogue input 0 (AO) pin were

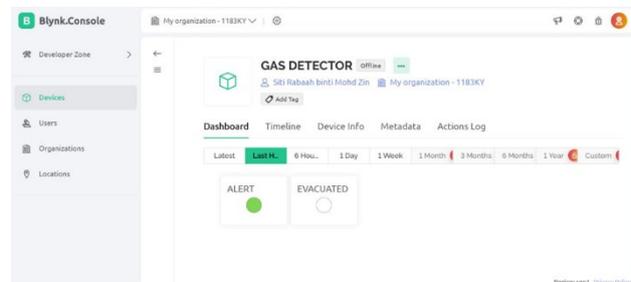
also connected to the board according to their respective port [29] as shown in Figure 1. The prepared circuit setup was then connected to open-source Arduino software according to Arduino ESP32 feature. The completed sensor circuit was then connected to open-source internet of thing (IoT) software which were Wokwi ESP32 to simulate the installation and Blynk to control the function of the sensor.



(a)



(b)



(c)

Fig. 1 (a) Illustration of the gas sensor connected to Arduino ESP32; (b) Wokwi, open-source internet of thing (IoT) source interface feature of using ESP32; and (c) Blynk, open-source application to control the sensor activity on mobile device

2.4 Preparation of Sulphur-based Polymer Coating

A few thin layers of polymeric sulphur were applied to the surface of the gas sensor by using a brush. The coating thickness was controlled to optimize the sensor performance. An ideal coating is thin and uniform to increase the surface area available for gas adsorption, which is crucial for detecting low gas concentrations [30].

3. Results and Discussion

Upon synthesis process, the sulphur-based polymer, polysulphide, were undergone to several characterization to determine the characteristics of the material and compatibility to be used as coating for the gas sensor.

3.1 Characterization of Polysulfide

The proposed schematic of the polysulphide synthesis is depicted in Figure 2. Based on the mechanism, it can be seen that the formation ring opening of cyclic sulphur occurs as the reaction reached at 170°C. This process was repeated several times until an S-S bond backbone was found in a linear polymer. Due to the vulnerable reaction of terminal free radicals, the polymer produced is unstable and leads to depolymerization into the original cyclic sulphur form [20]. As a result, unsaturated palm oil was used as a comonomer in an inverse vulcanization process because it contained unsaturated fatty acids [22][23]. The presence of the double bonds makes the polymer more

resistant to depolymerization by providing cross-linking within the polymer structure, thus enhancing its stability.

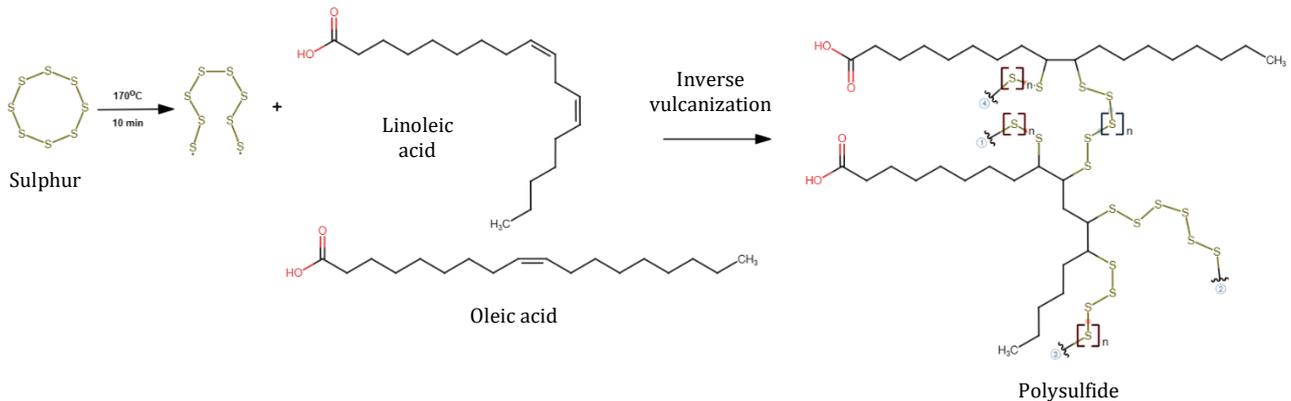


Fig. 2 Proposed schematic for the synthesis of polysulphide

The functional group of polysulphide was determined using ATR-FTIR as depicted in Figure 3. The spectra show the appearance of peak at 3500 cm⁻¹ indicating the presence of OH group. The disappearance of a peak at 3005 cm⁻¹ followed by 1650 cm⁻¹, which corresponds to the stretching vibration of vinyl groups C=C-H and C=C respectively. The functional group of C=O is presence at peak 1750 cm⁻¹. A significant peak was also observed at 966 cm⁻¹, indicating the polymerization of free radicals that caused the vibration of the C-S bond. Furthermore, there is a reduction in intensity associated with the *cis*-di-substituted alkene C-H bending in signals at around 717 cm⁻¹, which justifies the process polymerization was occurred [20]. These three spectra were analysed in order to determine the chemical structure of polysulphide as proposed in Figure 2. There is a significant difference between the polysulphide and raw palm oil at peak at 3005 cm⁻¹, which resembles the vinylic bond of C=C and is transformed to C-C during inverse vulcanisation to form the C-S polymer chain [31]. The formation of S-S opening bond was indicated at 488 cm⁻¹ of the polysulphide oil spectra which is similar to previous study by Biswas et al., (2007) [32].

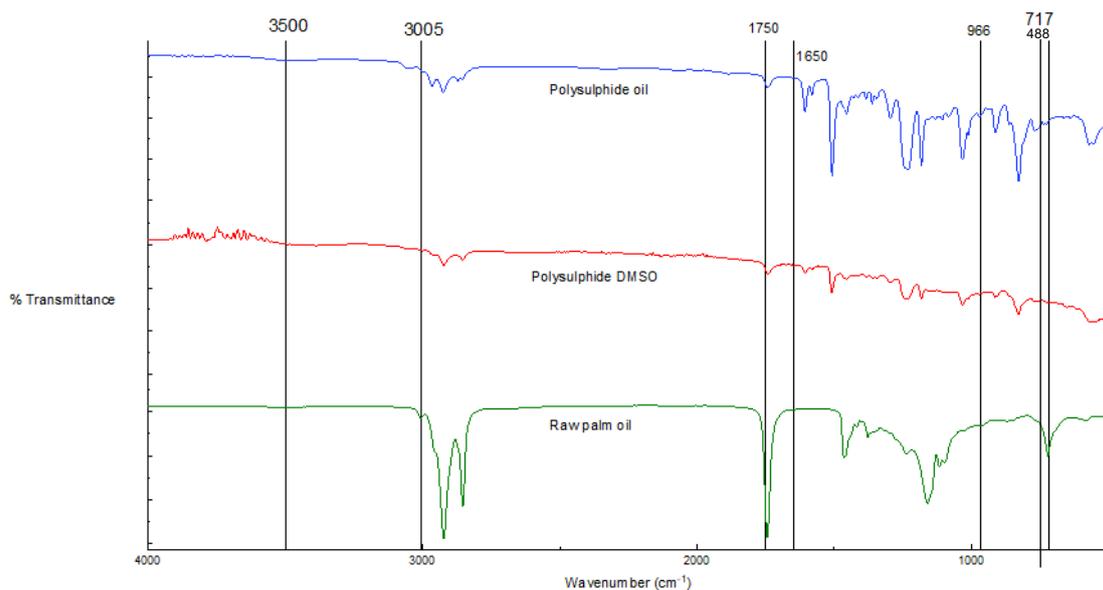


Fig. 3 ATR-FTIR spectra of polysulphide and palm oil

The morphology of different synthesis of polysulphide was evaluated by using SEM as depicted in Figure 4. It can be observed that the polysulphide exhibited different surface morphologies at different synthesis. Based on Figure 4 (a) of raw sulphur, polysulphide treated with palm oil (b) and polysulphide treated with DMSO (c). Polysulphide-DMSO was also prepared and compared with polysulphide-palm oil to evaluate the morphology effect between green strategy-based vulcanization process with conventional chemical vulcanization process. According to Figure 4 (b) and (c), the polysulphide treated respectively with palm oil and DMSO significantly differ from raw sulphur. Polysulphide-DMSO morphology surfaces appeared smooth, which may have contributed to the low

fracture toughness of epoxy resins. While, the morphology surfaces of polysulfide-oil are rougher, which indicates that it requires a great deal of energy to fracture the sample[5]. Oils create a heterogeneous material with areas of different mechanical properties, leading to a rougher fracture when the material breaks [13].

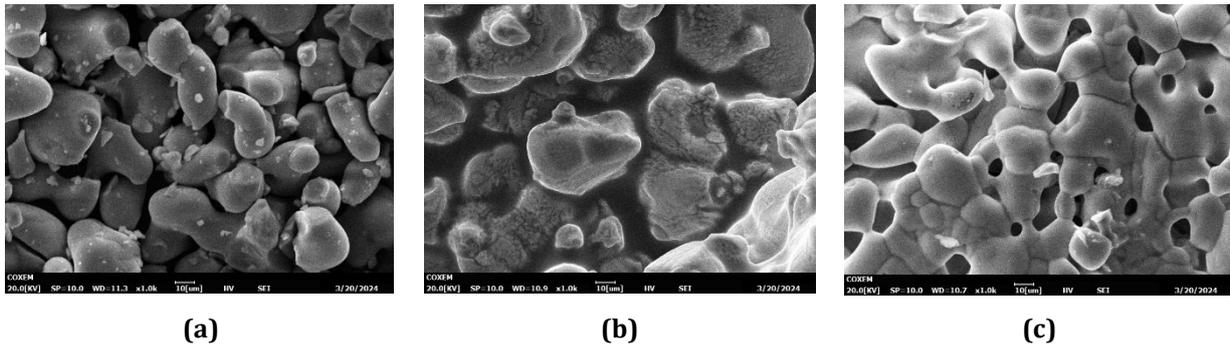


Fig. 4 SEM images of (a) raw sulphur; (b) polysulfide-palm oil; (c) polysulfide-DMSO with magnification 1000x

3.2 Evaluation Sensitivity of Gas Sensor

The application of prepared polysulfide-palm oil as coating agent for gas detection was applied on the gas sensor as shown in Figure 5. The obvious observation can be made after coating was the present of yellowish layer on the surface of the sensor as shown in Fig.5 (b). Coated gas sensor detects gas at 0.94 seconds while uncoated gas sensor detects at 3.00 seconds. Apart from decreased time, coating layers exhibit hydrophobic characteristics and thermal stability on the exterior surface of the sensor because the sulphur can resist high temperature up to 160°C [32]. Polysulfide-palm oil form heterojunctions with the host gas sensing materials to enhance electron transport thus effectively improving the responsivity and response speed [33].

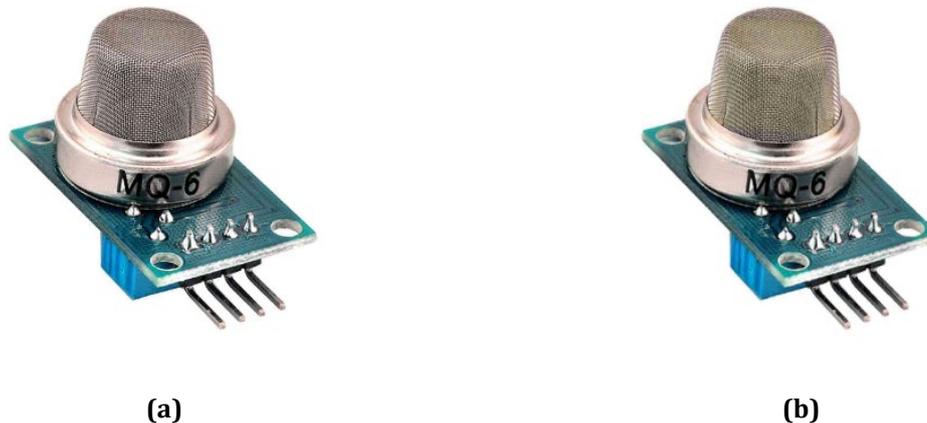


Fig. 5 (a) Gas sensor without coated; (b) Polymer coated gas sensor

4. Conclusion

The development of sulphuric-based polymer coating for gas sensor enhancement from waste of sulphur with green strategy-based reverse vulcanization process. The characterization of the synthesised polysulphide using ATR-FTIR has shown the presence of C-S bonding at 966 cm^{-1} indicating the successful reaction of the sulphur and oil. The morphology of the polysulphide coating was also analysed by using SEM has shown the gas sensor with polysulfide has higher sensitivity in terms of time taken to detect gas compared to the uncoated gas sensor. Based on the finding, it can be concluded that the prepared polysulfide as a compatible and effective coating agent improves the sensitivity of the gas sensor. Again, the innovative green polymer-gas sensor not only enhances the sensitivity of the gas sensor but lead to utilization of environmental-friendly material.

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Conflict of Interest

Authors declare that there is no conflict of interests regarding the publication of the paper.

Author Contribution

The authors confirm contribution to the paper as follows: **study conception and design:** Siti Rabaah Mohd Zin, Nurul Fatihah Hisamuddin, Siti Balqis Mohammad Shah, Wan Muhammad Faris Hadi Wan Sulaimi, Mohammad Arif Budiman Pauzan; **data collection:** Siti Rabaah Mohd Zin, Nurul Fatihah Hisamuddin, Siti Balqis Mohammad Shah, Wan Muhammad Faris Hadi Wan Sulaimi; **analysis and interpretation of results:** Siti Rabaah Mohd Zin, Nurul Fatihah Hisamuddin, Siti Balqis Mohammad Shah, Wan Muhammad Faris Hadi Wan Sulaimi, Mohammad Arif Budiman Pauzan; **draft manuscript preparation:** Siti Rabaah Mohd Zin, Nurul Fatihah Hisamuddin, Siti Balqis Mohammad Shah, Wan Muhammad Faris Hadi Wan Sulaimi, Mohammad Arif Budiman Pauzan, Zalilah Murni Mat Ali @ Yunus, Nurun Najwa Ruslan. All authors reviewed the results and approved the final version of the manuscript.

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