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Synthesis, Characterization of Ag_2s from AgCl Waste of Argentometry Titration with Heating Temperature Variations and Its Application as a Temperature Sensor Based on Negative Temperature Coefficient (NTC)

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Abstract

Synthesis of Ag_2S from AgCl waste of argentometric titration with heating temperature variations as a temperature sensor has been done. This study aims to synthesize Ag_2S and examine the effect of heating temperature on crystal quality and electrical characteristics as a temperature sensor based on the Negative Temperature Coefficient (NTC). Ag_2S synthesis was carried out by precipitation in a water bath with various heating temperatures of 40°C, 60°C, and 80°C. The success of the synthesis was confirmed by X-Ray Diffraction (XRD) with a typical peak of 2θ from Ag_2S standard at 29.07°, 31.60°, 36.97°, 37.81°, and the highest crystallinity was obtained at a heating temperature of 60°C. Meanwhile, UV-Vis Diffuse Reflectance Spectroscopy (DRS UV-Vis) showed a band gap corresponding to Ag_2S (0.9-1.05 eV). Furthermore, the Ag_2S powder was made into pellets and applied as a temperature sensor. Then the resistance value and the electrical characteristics of the resulting sensor were measured. The best resistance was obtained from Ag_2S synthesized at a temperature of 60°C with constant (B) and sensitivity (α) values of 2974 K and -3.35%, respectively. This indicated that Ag_2S had been successfully synthesized, and the best sensor quality was obtained from Ag_2S heated at a temperature of 60°C.

1. Introduction

Argentometric titration has been widely used for analyzing the concentration of analytes accurately. However, on the other hand, it poses a problem with large amounts of AgCl precipitate residue [1]. AgCl waste residue, if directly disposed of, will be harmful to the environment, so it is necessary to reuse AgCl waste for other valuable materials. This is in line with the goals of Green Chemistry: minimizing waste and recovering material benefits [2]. Therefore, in this study, AgCl waste will be recycled into silver sulfide (Ag_2S).

Several studies related to Ag_2S synthesis were conducted using Chemical Bath Deposition (CBD) [3], hydrothermal [4], spray pyrolysis (SPD) deposition [5], sonochemistry [6], sol-gel [7], and precipitation. CBD requires many chemicals, and it is an unsustainable process. Meanwhile, hydrothermal and sonochemical preparations require a vacuum and high temperature, and sol-gel takes a long time. In this study, the precipitation method will be employed because the preparation is easy, can be done at low temperatures of 25–90°C [8], has good composition control [9], and can be prepared in situ directly on the titration waste.

Several dissolution/precipitation processes of Ag_2S have been done successfully from natural phosphates for photocatalyst applications [9]. Precipitation of Ag_2S with L-cysteine (as a source of sulfur) [10], Ag and sulfurization of H_2S with Ag-Ag_2S nanohybrid NHS [11]. However, H_2S has high toxicity, and L-cysteine is difficult to obtain. Meanwhile, in this study, Ag_2S was obtained by AgCl precipitation with sulfurization using thiourea, which is non-toxic and environmentally friendly [12].

Silver sulfide is a semiconductor material with a band gap of 0.9–1.05 eV [13], good chemical stability, and optical properties. Ag_2S is widely used in various fields such as photocatalyst [14], antifungal [15], antibacterial [16], detector and temperature sensor [13].

The temperature sensor is one of the sensors to detect symptoms of temperature changes in specific dimensions with a thermistor [17]. One often used thermistor is the negative temperature-coefficient (NTC), with good sensitivity and response [18]. Most of the NTC materials are solid solutions of transition metal oxides, such as NiO, Mn_3O_4, and Co_3O_4, with spinel-type crystal structures often exhibiting poor stability and reproducibility due to high porosity and incomplete intergranular contact [19].

Utilization of Ag_2S as an NTC thermistor is still rarely done. Research by Yu et al. [20] successfully fabricated NTC using Ag_2S-Ag films from annealed Ag/MPA nanoparticle films showing a reversible change in resistivity when the temperature increased from 25 to 170°C. Therefore, in this research, Ag_2S synthesis from AgCl waste from argentometric titration was done at various temperatures of 40°C, 60°C, and 80°C. The synthesized Ag_2S powder was characterized using XRD and DRS UV-Vis. In addition, the quality of the synthesized Ag_2S as a temperature sensor based on the Negative Temperature Coefficient (NTC) was also studied.

2. Materials and Methods

The research consisted of Ag_2S synthesis and preparation of NTC temperature thermistor pellets. The synthesis was conducted by the deposition method, and pellets preparation was done by combining Ag_2S powder with two wires as electrodes, which were then pressed and molded, as shown in Figure 1.

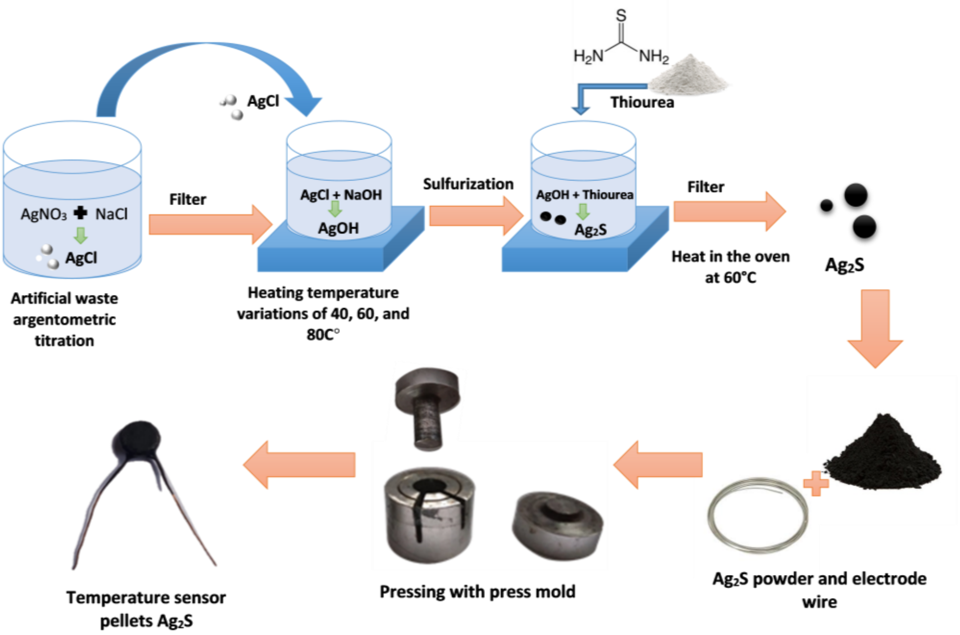

Figure 1. Synthesis of Ag_2S scheme and preparation of NTC thermistor pellet

2.1. Materials and equipment

Materials used were thiourea (Merck), $AgNO_3$ (Merck), NaCl (Merck), NaOH p.a (Merck), frying oil (Tropicana slim), and paint for iron (Weldon gloss). The equipment used were glassware (Herma, Pyrex, Iwaki), analytical balance (Ohaus), hotplate stirrer (Thermo-scientific), multitester (Krisbow), water bath (handmade), thermometer, pH indicator (Macherey-Nagel), press mold, oven (Faithful), DRS UV-Vis (Shimadzu UV–2450), and X-ray diffraction (Shimadzu XRD – 7000).

2.2. Ag_2S synthesis with the variation of heating temperature

Ag_2S synthesis was done in a water bath. Previously, AgCl artificial waste was prepared by dissolving $AgNO_3$ and NaCl in distilled water with different beakers, then mixed to form a white precipitate of silver chloride. Furthermore, the silver chloride precipitate was filtered and dissolved in 0.1 M NaOH and heated in a water bath to a certain temperature variation of 40°C, 60°C, and 80°C. Thiourea powder was added when the solution reached that temperature, and a black Ag_2S precipitate was formed. After constant temperature, the precipitate was filtered and dried in an oven at 60°C for 60 minutes.

2.3. Characterization of Ag_2S

Two instruments, XRD and DRS UV-Vis, were employed to characterize Ag_2S powder. X-ray Diffraction (XRD) was used to determine the crystallinity of Ag_2S and Diffuse Reflectance Spectroscopy UV-Vis (DRS UV-Vis) was done to measure the band gap of the Ag_2S semiconductor.

2.4. Pellet preparation of Ag_2S for temperature sensor

The pellet preparation was conducted by pressing the synthesized Ag_2S powder and iron wire as electrodes using a pellet press. The pressed temperature sensor pellets were then coated with iron paint and dried at room temperature for 24 hours.

2.5. Temperature sensor resistance measurement in oil

The resulting temperature sensor pellets were determined for their electrical characteristics by measuring their resistance in the oil using a multitester. Measurements were performed in a temperature range of 25–50°C. This is in line with the thermistor sensor application for measurements between 20 to 50°C, where this measurement range is the normal working air temperature to the operating air temperature around the equipment/machine [21]. The resistance measurement results were then processed to produce a graph with a relationship between temperature and resistance to obtain the electrical characteristics of the temperature sensor. Another graph with a relationship between ln ρ and 1/T was obtained to determine the electrical parameter value and the quality of the resulting temperature sensor. The resistivity value depends on temperature so that it can be calculated by equation (1) [22]:

$$\rho_{(T)} = \rho_{\infty} \, exp \left(\frac{Ea}{kT}\right) \quad (1)$$

From the graph depicting the relationship between ln to 1/T, the electrical parameter values of the temperature sensor, namely constant (B) and sensitivity (α), can be calculated using equation (2) and (3) [23, 24].

$$\mathrm{B} = \frac{\mathrm{Ea}}{\mathrm{k}} \quad (2)$$

$$\alpha = -\frac{\mathrm{B}}{\mathrm{T}^2} \times 100\% \quad (3)$$

3. Results and Discussion

3.1. Ag_2S synthesis with varied heating temperature

The synthesis of silver sulfide (Ag_2S) was done in an alkaline medium (NaOH) by utilizing silver chloride waste from argentometric titration using thiourea reagent as a sulfur source [25]. In this study, the synthesis of AgS was conducted in a water bath system by varying the heating temperature. The heating was varied (40°C, 60°C, and 80°C) to determine the effect of heating temperature on crystals and the characteristics of silver sulfide as a temperature sensor.

In this synthesis, artificial waste silver chloride was dissolved in NaOH to produce a brown AgOH solution with a pH = 12 value. After the temperature reached the predetermined variation, thiourea solids were added, which served as a source of sulfur, and the solution turned black with pH = 7, indicating the formation of Ag_2S precipitate as equations (4), (5), and (6) [26].

$$\mathrm{NaCl + AgNO3 \rightarrow AgCl\downarrow + NaNO_3} \quad (4)$$

$$\mathrm{AgCl_{(s)} + NaOH_{(aq)} \rightarrow Ag(OH)_{(aq)} + NaCl_{(s)}} \quad (5)$$

$$\mathrm{2AgOH_{(aq)} + CS(NH_2)_{2(aq)} \rightarrow Ag_2S\downarrow + H_2NCN_{(aq)} + 2H_2O} \quad (6)$$

Since the Ksp value of Ag_2S is lower than that of AgOH, it is more likely to precipitate because the OH^- ions will be replaced by S^{2-} ions and produce Ag_2S (K_{sp} AgOH = 2×10^{-8}, K_{sp} Ag_2S = 1×10^{-49}). The obtained Ag_2S precipitate was then dried in an oven to remove the water it contained [27]. The reaction mechanism of thiourea hydrolysis in an alkaline medium is shown in equations (7), (8), and (9) [28].

$$\mathrm{(NH_2)_2CS + OH^- \rightarrow HS^- + H_2NCN + H_2O} \quad (7)$$

$$\mathrm{HS^- + OH^- \rightarrow S^{2-} + H_2O} \quad (8)$$

$$\mathrm{2AgOH + (NH_2)_2CS \rightarrow Ag_2S\downarrow + H_2NCN + 2H_2O} \quad (9)$$

3.2. Characterization of Ag_2S Powder

The crystal structure of the Ag_2S synthesized with variations in heating temperature was confirmed using X-ray Diffraction (XRD), as shown in Figure 2. X-ray characterization was carried out with Cu $K_{\alpha1}$ radiation (1.5406) at 2θ from 20 to 70° to determine crystallinity and the size of the crystal. The results show the peaks of the XRD spectra of Ag_2S powder samples with variations in heating temperatures of 40°C, 60°C, and 80°C. At a heating temperature of 40°C, the Ag_2S peak appears at 2θ of 29.05, 31.57, 34.44, 36.93, and 37.80°. At a heating temperature of 80°C, Ag_2S peaks at 2θ of 29.05, 31.61, 34.48, 36.96, and 37.84°. Meanwhile, at a heating temperature of 60°C, Ag_2S peaks at 2θ of 29.07, 31.60, 34.47, 36.97, and 37.81°. This result is in agreement with another study group on silver sulfide [29], and the Ag_2S phase in this study can be determined according to the data reported for Ag_2S (acanthite) (JCPDS: 00-014-0072) [30]. This is confirmed by the dominant peak pattern, which corresponds to the RRUFF standard for Ag_2S data shown at 2θ peak 22° (-102), 29° (110), 31° (-113), 34.48° (-121), 36.97° (-122), 37.8° (-104), and 41° (031) [31] and indexed to the monoclinic. It can be concluded that Ag_2S has been formed and successfully synthesized.

Variations in heating temperature affected the resulting crystallinity. This can be seen from the difference in the intensity of the curve obtained. The best crystallinity was obtained at a heating temperature of 60°C. In addition, at the peaks of this synthesis, the Ag_2S produced is impure because there are still residual impurities from reactions such as NaCl at 2θ of 32.36 and 46.47° at the peak yields of Ag_2S at heating temperatures of 60 and 80°C.

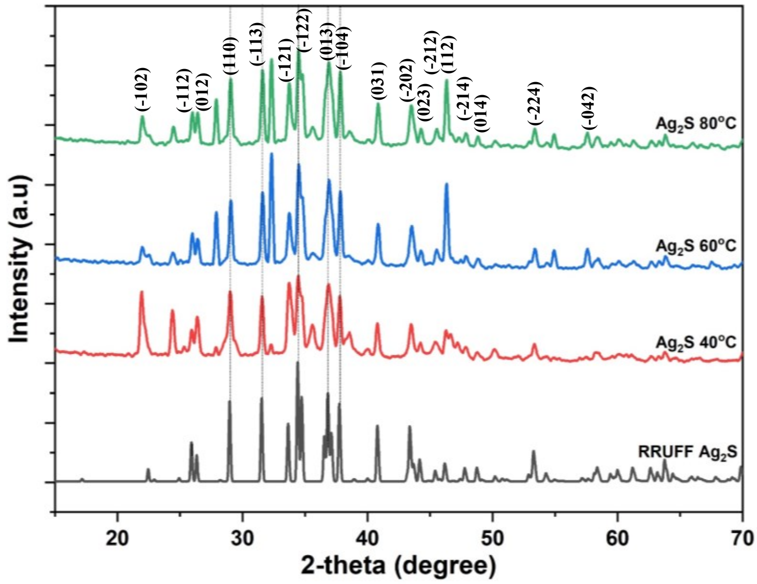

Figure 2. Diffractogram of RRUFF Ag_2S with sample with varied heating temperature

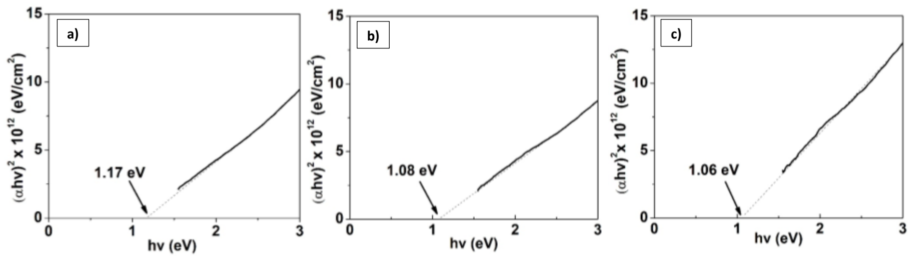

Figure 3. The band gap of Ag_2S at heating temperatures of (a) 40, (b) 60, and (c) 80°C

Meanwhile, the impurities originating from different reaction residues, such as AgOH and H_2NCN at 2θ of 21.94 and 24.48°, were present at a heating temperature of 40°C. The determination of Ag_2S crystal grain size was estimated using the Debye-Scherrer formula [32, 33] in equation (10).

$$G = \frac{k\,\lambda}{D\cos\theta} \tag{10}$$

Where G is the crystal size, k = 0.9 is the formation factor, λ is the wavelength of the CuK_α line, D is the FWHM in radians, and θ is the Bragg angle. From the calculation results in Table 1, it is known that the smaller the FWHM, the larger the crystal grain size and the smaller the diffraction pattern. On the contrary, the larger the FWHM value, the smaller the crystal size and the larger the diffraction pattern [33].

The average grain sizes of Ag_2S crystals at heating temperatures of 40°C, 60°C, and 80°C determined by the Debye-Scherrer equation were 33.18, 33.81, and 31.62 nm. The average grain size of the sample crystals decreased when the heating temperature was 80°C. This is because the heating temperature was extremely high during the synthesis, causing the sulfide ion thiourea to evaporate and produce a smaller average crystal grain size. Crystals with high crystallinity values and an average large crystal grain size were obtained at a heating temperature of 60°C.

Table 1. The analysis results of Ag_2S crystal size

Sample	2-theta (o)	Theta (o)	FWHM (rad)	D (nm)
	29.05	14.52	0.196	40.82
	31.57	15.78	0.236	33.81
Ag_2S 40°C	34.44	17.22	0.196	40.27
	36.93	18.46	0.708	11.11
	37.80	18.90	0.196	39.89
		Average		33.18
	29.07	14.53	0.196	40.82
	31.60	15.80	0.236	33.80
Ag_2S 60°C	34.47	17.23	0.196	40.27
	36.97	18.49	0.551	14.28
	37.81	18.91	0.196	39.89
		Average		33.81
	29.05	14.53	0.196	40.82
	31.61	15.81	0.236	33.80
Ag_2S 80°C	34.48	17.24	0.196	40.27
	36.96	18.48	0.787	9.99
	37.84	18.92	0.236	33.23
		Average		31.62

Characterization with a DRS UV-Vis spectrophotometer was performed to determine the band gap of the Ag_2S powder sample with variations in heating temperature. The energy band gap of Ag_2S powder with variations in heating temperature was obtained by plotting the data using the direct band gap equation. The graph plot of Ag_2S with variations in heating temperature between $(\alpha h\nu)^2$ vs. $h\nu$ is shown in Figure 3. The Tauc Plot method with a tolerance of ±5% shows the graph intersection, and the flat axis shows the energy band gap width. Based on Figure 3, the band gap value obtained by all samples is around 1 eV, precisely the same as the band gap of the Ag_2S semiconductor studied by Sahraoui et al. [5]. The resulting band gap value also decreases with increasing heating temperature. This is due to the formation of larger aggregates that caused a decrease in the band gap value [5].

3.3. Results of Ag_2S temperature sensor

Temperature sensor pellets were made using powder from Ag_2S synthesis with varied heating temperatures. The three Ag_2S temperature sensor pellets have the same dimensions, 0.5 cm in diameter with 0.2 cm thickness and 3 cm in wire length. The temperature sensors can be seen in Figure 4. The temperature sensors made from Ag_2S semiconductor material have the property of NTC thermistor characteristics. The working principle of NTC is the resistance change with a change in temperature, where the resistance value and temperature in the NTC type are inversely proportional [17].

3.4. Measurement of temperature sensor resistance in oil

Resistance measurement of the Ag_2S temperature sensor with variations in heating temperature was conducted in oil to determine the electrical characteristics of the Ag_2S semiconductor as a Negative Temperature Coefficient (NTC) temperature sensor. Since oil has the property of heating up more quickly, the temperature sensor was applied to oil to observe how quickly the resulting temperature sensor responds to changes in temperature (heat) based on this electrical property. Measurement of temperature sensor resistance was measured using a multitester with the output generated by the temperature sensor (thermistor) known as resistance [34]. The results of measuring the resistance of the Ag_2S temperature sensor with variations in heating temperature are presented in Figure 5.

Figure 5 shows that the temperature sensor decreases the resistance value with the higher heated oil temperature. This is in line with the characteristics of the NTC temperature sensor, where the resistance value will change if there is a change in the surrounding temperature, and the resistance value will be smaller if the surrounding temperature is getting hotter/higher or vice versa [35]. The resistance measurement results obtained are smaller than some other NTC studies, such as Liu et al. [36], Yu et al. [20], and Trung et al. [37]. The resistance value affects energy and stability. The higher the resistance, the greater the energy required and the more stable the material. In contrast, the smaller the resistance, the smaller the energy required and the lower stability. Therefore, the value of the resistance should not be too large or too small. In this study, the resistance value of 10^6 Ω can be used as NTC.

The oil resistance measurement results are used to test the electrical parameters of the Ag_2S temperature sensor. The main electrical parameters in the temperature sensor are constant (B) and sensitivity (α). These two parameters determine the electrical characteristics and quality of the resulting temperature sensor [38]. The electrical characteristics of the temperature sensor are determined through the graph with the relationship between ln ρ and 1/T so that the linear equation y = mx + c is obtained, as shown in Figure 6.

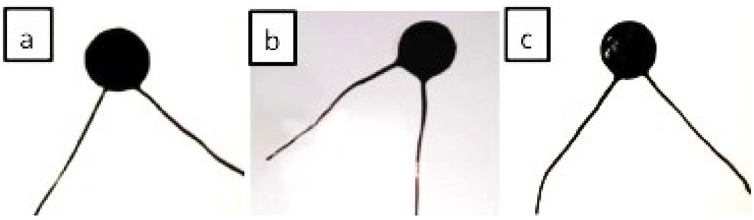

Figure 4. Temperature sensor pellets from Ag_2S powder heated at (a) 40°C, (b) 60°C, and (c) 80°C

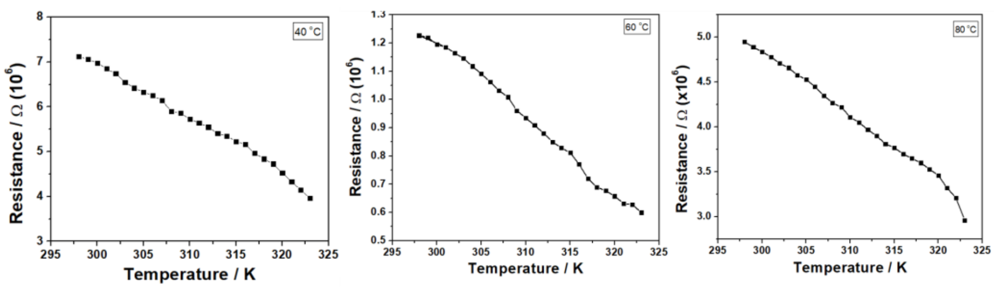

Figure 5. The relationship graph between resistance and temperature at heating temperatures of (a) 40°C, (b) 60°C, and (c) 80°C

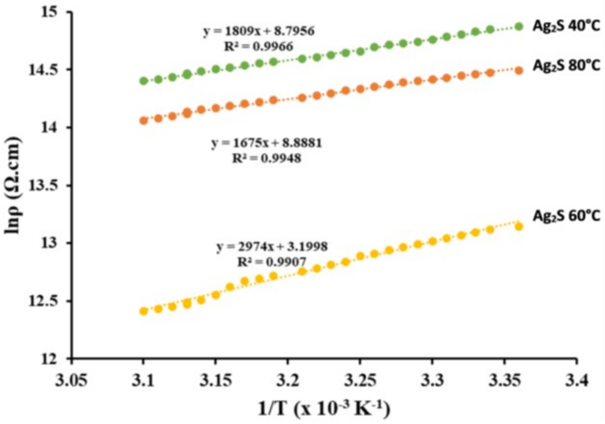

Figure 6. The relationship graph of ln ρ vs. 1/T on Ag_2S temperature sensor with varied heating temperature

The temperature sensor constant (B) value is derived from the slope of the graph ln ρ vs. 1/T, and the sensitivity value of the Ag_2S temperature sensor was calculated using equation 3. The results of the electrical characteristics of the Ag_2S temperature sensor with variations in heating temperature are presented in Table 2.

Table 2. Electrical parameter values of Ag_2S temperature sensor

Temperature Sensor	B (K)	α (%)
Ag_2S 40°C	1809	-2.04
Ag_2S 60°C	2974	-3.35
Ag_2S 80°C	1675	-1.89

Based on the data in Table 2, it can be seen that the greater the constant value, the greater the sensitivity value obtained. The greater the value of the electrical parameters obtained, the better the quality of the temperature sensor produced. It can be concluded that the 60°C Ag_2S temperature sensor is a temperature sensor with better quality than 40°C and 80°C.

Table 3. Comparison of thermal constant (B) with commercial NTC thermistors

Materials	B (K)	References
rGO	572	[37]
Graphene	945	[39]
Graphene-P(VDF-TrFE)	250	[39]
CNT	474	[40]
$Ca_{0.05}Zn_{0.05}TiO_3$	2242	[41]
Ag_2S-Ag	2684	[20]
This work (Ag_2S)	2974	-

In addition, the value of the electrical characteristics produced by the 60°C Ag_2S temperature sensor showed better results than Yu et al. [20], whereas the value of the Ag_2S temperature sensor constant ranges from 1250–2684 K, and the sensitivity ranges from -1.41 to -3.02%. This result is still better than the previous studies on commercial NTC materials, as shown in Table 3.

4. Conclusion

Synthesis of Ag_2S can be done by using waste silver chloride reacted with thiourea in an alkaline medium using the precipitation method in a water bath. Variations in the heating temperature of silver sulfide affect the crystal and electrical characteristics of Ag_2S as a temperature sensor. The results of this study with heating temperature variations of 40°C, 60°C, and 80°C showed optimal results at 60°C characterized by high crystallinity with a band gap value of 1.08 eV and good temperature sensor quality. It was indicated by the high electrical parameter values of constant (B) and sensitivity, which are 2974 K and -35%, respectively.

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