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PRODUCTION OF SODIUM SULFIDE FROM ELEMENTAL SULFUR AND SODIUM HYDROXIDE: PHYSICOCHEMICAL AND THERMOANALYTIC INVESTIGATION

***Abstract**: Sodium sulfide (Na_2S) is an industrially important compound widely used in metallurgy, leather processing, textile dyeing, and chemical synthesis. Conventional production based on high-temperature reduction of sodium sulfate with coke in shaft furnaces is environmentally outdated, energy-intensive, and unable to utilize sulfate-containing liquid wastes. This study proposes and experimentally validates an alternative low-temperature method for producing sodium sulfide through the reaction of elemental sulfur with aqueous sodium hydroxide. NaOH solutions of 30%, 40%, and 50% concentration were reacted with sulfur under controlled stirring at temperatures above 130 °C. The physicochemical properties of the resulting Na_2S/Na_2S_x mixtures—including pH, density, and viscosity—were measured as functions of sulfur content and temperature. Results show a monotonic decrease in pH with increasing sulfur concentration, while density and viscosity increase with sulfur content and decrease with temperature. Thermal analysis (TG/DTA/DTG) revealed a three-stage mass loss totaling 36.12% and an exothermic transition at 218.9 °C indicative of structural rearrangements in polysulfide species. The findings demonstrate that the sulfur–NaOH method provides a cleaner, waste-free, and economically viable alternative to traditional sodium sulfide production technologies.*

***Key words**: Sodium sulfide; Na_2S; polysulfides; sodium hydroxide; sulfur; thermal analysis; TG/DTA; viscosity; density; pH; green technology; waste-free process; sulfate-containing wastes; chemical synthesis.*

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Introduction

Sodium sulfide (Na_2S) is a strategically important inorganic compound widely used in non-ferrous metallurgy for the beneficiation of copper, lead–zinc, and molybdenum ores; in the leather industry for dehairing hides; in the textile industry for dyeing; and in chemical manufacturing for producing sulfur dyes and for reduction processes. Currently, industrial Na_2S is obtained by reducing briquetted sodium sulfate with metallurgical coke in shaft furnaces at **800–1000 °C**. Although this method was technologically advanced in the 1960s, it has become **environmentally outdated, energy-intensive, and economically non-competitive** compared to foreign technologies.

A major drawback of the conventional method is its inability to utilize **sulfate-containing liquid waste streams**, which are produced by many industries.

Environmental regulations and market competition require **waste-free, energy-efficient, and cleaner technologies** for Na_2S production. Consequently, developing an alternative method using **elemental sulfur and sodium hydroxide** is essential for restoring the competitiveness of domestic sodium sulfide production.

The purpose of this research was to develop, experimentally validate, and characterize a novel method for producing sodium sulfide from **elemental sulfur and aqueous sodium hydroxide**, followed by a detailed analysis of the **pH behavior, density, viscosity, and thermal properties** of the resulting mixtures.

2. Materials and Methods

2.1. Raw materials

- **Sodium hydroxide (NaOH)** solutions with concentrations of **30%, 40%, and 50%** (wt.%).
- **Elemental sulfur (S_8)** in powdered form.
- Distilled water.

2.2. Experimental procedure

Experiments were performed in a **thermostated reactor equipped with a mechanical stirrer**. The required amount of NaOH solution was charged into the reactor. Elemental sulfur was added **gradually** under continuous stirring. The reaction temperature was maintained at **not less than 130 °C**, ensuring efficient dissolution of sulfur and formation of sodium sulfide/polysulfide species.

Three sets of NaOH solutions were prepared:

Table 1.

NaOH concentration	Added sulfur (g per 100 g solution)
30%	12, 24, 36 g
40%	16, 32, 48 g
50%	20, 40, 60 g

2.3. Analytical methods

- **pH measurement:** pH meter at **18 °C**.
- **Density (ρ):** measured at **50, 70, and 90 °C**.
- **Viscosity (μ):** measured using viscometry at **50, 70, 90 °C**.
- **Thermal analysis (TG/DTG/DTA):** Performed using **Netzsch STA 409 PG** (Germany), K-type thermocouple, aluminum crucibles. Heating range: **20–300 °C**.

3. Results

3.1. pH, density, and viscosity data

Table 2. Physicochemical properties of NaOH–S mixtures

№	NaOH (wt.%)	S added (g)	pH (18°C)	Density ρ (g/cm³) at 50/70/90°C	Viscosity μ (cP) at 50/70/90°C
1	30	12	12.38 / 11.88 / 11.71 / 9.70	1.360 / 1.348 / 1.333	1.39 / 1.00 / 0.75
2	30	24	12.03 / 11.97 / 11.91 / 10.41	1.392 / 1.384 / 1.370	1.46 / 1.60 / 0.80
3	30	36	11.50 / 11.36 / 11.33 / 10.32	1.400 / 1.396 / 1.409	1.50 / 2.86 / 0.93
4	40	16	12.28 / 11.95 / 11.47 / 9.37	1.390 / 1.376 / 1.342	1.42 / 1.32 / 0.78
5	40	32	12.20 / 12.00 / 11.40 / 10.26	1.396 / 1.388 / 1.372	1.45 / 2.45 / 0.89
6	40	48	11.48 / 11.40 / 11.25 / 10.28	1.403 / 1.401 / 1.422	1.68 / 2.87 / 1.02
7	50	20	12.18 / 11.81 / 10.90 / 8.96	1.378 / 1.380 / 1.351	1.44 / 1.52 / 0.77
8	50	40	12.05 / 11.74 / 11.17 / 9.87	1.436 / 1.412 / 1.419	1.60 / 2.77 / 0.99
9	50	60	11.98 / 11.78 / 11.19 / 9.96	1.503 / 1.423 / 1.515	1.88 / 3.11 / 1.37

3.2. pH behavior

- pH **decreases with increasing sulfur content** within each NaOH concentration series.
- For 30% NaOH, pH decreases from **12.38 → 11.5** as sulfur increases from 12 g → 36 g.
- When solutions are diluted to 10%, 20%, 50%, pH decreases monotonically.

3.3. Density

- Density **decreases with temperature** for all samples.
- Increasing sulfur amount leads to **higher density**.
- Maximum density: **1.545 g/cm³** observed for the 50% NaOH solution with 60 g sulfur.

3.4. Viscosity

- Viscosity **decreases** as temperature increases (50 → 90 °C).
- Viscosity **increases strongly with sulfur concentration**, indicating longer polysulfide chains.
- Example:

30% NaOH + 12 g S: μ = 1.39 cP (50 °C)

30% NaOH + 36 g S: μ = 2.86 cP (70 °C)

4. Discussion

4.1. Influence of sulfur on pH

Adding sulfur to NaOH leads to the formation of **sodium sulfide (Na_2S)** and **polysulfides (Na_2S_x)**. The reaction consumes hydroxide ions, causing a **decrease in pH**:

$$2NaOH + xS \rightarrow Na2Sx + H2O$$

Thus, a direct correlation exists between sulfur content and acidity shift.

4.2. Temperature dependence of density

As temperature increases, the density decreases due to **thermal expansion**. Conversely, sulfur-rich mixtures show higher density because polysulfide structures have greater molecular mass.

4.3. Viscosity behavior

Viscosity reduction at elevated temperatures is expected due to decreased intermolecular interactions. The strong viscosity increase with sulfur content reflects formation of **long-chain polysulfides (S_x^{2-})**, which significantly alter rheological behavior.

4.4. Advantages of the sulfur–NaOH method

Compared to traditional Na_2SO_4 reduction with coke:

- No high-temperature furnaces required
- No harmful gaseous emissions (SO_2, dust)
- Ability to use **liquid sulfate-containing industrial wastes**
- Lower energy consumption
- Higher product purity
- Waste-free technology

This method creates a competitive and sustainable alternative to outdated industrial processes.

5. Thermal Analysis (TG/DTA/DTG)

A 30% NaOH mixture with a mass ratio NaOH:S = **4:1** was thermoanalyzed.

5.1. Mass loss

Total mass loss: **36.12%**, occurring in three stages:

1. **70–80 °C:**
   - Δm = **13.28%**
   - Rate: 2.2%/min
   - Enthalpy: –383.9 J/g
2. **92–130 °C:**
   - Δm = **11.22%**
   - Rate: 2%/min
   - Enthalpy: –137 J/g
3. **130–200 °C:**
   - Δm = **11.62%**
   - Peak rate: 4%/min
   - Enthalpy: –185.2 J/g

5.2. Thermal events

- At **218.9 °C**, a distinct **exothermic peak (8.9 J/g)** is observed, indicating a **phase transition** or structural rearrangement of polysulfide species.

These results confirm that the synthesized Na_2S–Na_2S_x mixtures undergo multi-stage dehydration and structural decomposition.

Conclusion

A new, environmentally friendly method for producing sodium sulfide from **elemental sulfur and sodium hydroxide** was experimentally validated. Detailed physicochemical studies demonstrated that:

- **pH decreases** with increasing sulfur content and dilution.
- **Density and viscosity** increase with sulfur concentration but decrease with temperature.
- **Thermal analysis** revealed a three-stage decomposition pattern with characteristic endo- and exothermic effects.
- The proposed method eliminates high-temperature processes, allows processing of **sulfate-containing liquid wastes**, and achieves higher ecological and economic efficiency.

Thus, the sulfur–NaOH method represents a **promising, competitive, and sustainable technology** for sodium sulfide production.

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