UNIVERSITAS GADJAH MADA
**Sintesis SnSO? melalui Oksidasi Elektrokimia Timah Murni dalam Sel Dua Ruang Bersekat Membran Nafion 115 dan Kristalisasi Evaporatif**
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# INTISARI

Timah (Sn) merupakan komoditas strategis yang banyak dimanfaatkan dalam industri elektronik, katalisis, dan material fungsional. Salah satu turunannya, timah(II) sulfat ($SnSO_4$), berperan penting sebagai prekursor pada baterai, sensor gas, dan material optoelektronik. Namun, sintesis $SnSO_4$ konvensional umumnya masih melibatkan proses kimia multi-tahap, penggunaan reagen tambahan, serta waktu proses yang panjang. Penelitian ini bertujuan mengembangkan sintesis $SnSO_4$ melalui integrasi oksidasi elektrokimia timah dan kristalisasi evaporatif secara terkontrol. Penelitian dilakukan menggunakan sel elektrokimia dua ruang bersekat membran Nafion 115, dengan Sn sebagai anoda dan grafit sebagai katoda. Sel elektrokimia memiliki volume 175 mL pada masing-masing ruang anolit dan katolit, menggunakan larutan $H_2SO_4$ sebagai elektrolit, dengan volume anolit sebesar 150 mL. Parameter elektrokimia yang divariasikan meliputi konsentrasi $H_2SO_4$ 0,01–1,0 M, tegangan 0,5–3,0 V, dan waktu elektrolisis 4–8 jam. Optimasi efisiensi Faraday dilakukan menggunakan *Response Surface Methodology* (RSM) dengan desain Box–Behnken. Larutan konsentrat $SnSO_4$ hasil elektrolisis kemudian dikristalkan secara evaporatif pada suhu 55–85 °C dengan variasi *seed* 0–1,2 g/L. *Seed* $SnSO_4$ yang digunakan memiliki ukuran partikel rata-rata 2,2 µm. Produk dikarakterisasi menggunakan SEM-EDS, PSA, XRF, FTIR, dan XRD.

Hasil penelitian menunjukkan bahwa oksidasi elektrokimia Sn dalam media $H_2SO_4$ berhasil menghasilkan larutan konsentrat $SnSO_4$ sebagai prekursor kristalisasi. Peningkatan konsentrasi $H_2SO_4$ dan tegangan listrik mempercepat pelarutan Sn menjadi $Sn^{2+}$, tetapi kondisi berlebih dapat memicu pasivasi dan reaksi samping. Optimasi RSM menunjukkan kondisi optimum pada konsentrasi $H_2SO_4$ 0,64 M, tegangan 2,09 V, dan waktu elektrolisis 5,77 jam, dengan prediksi efisiensi Faraday maksimum. Kristalisasi evaporatif berhasil mengubah larutan konsentrat $SnSO_4$ menjadi kristal $SnSO_4$ anhidrat berstruktur *orthorhombic*. Suhu evaporasi menjadi parameter dominan, dengan kondisi 85 °C tanpa *seed* menghasilkan *yield* kristalisasi sekitar 95%. Penambahan *seed* 0,6–1,2 g/L hanya mempercepat tahap awal kristalisasi, tetapi tidak meningkatkan konversi akhir secara signifikan. Analisis pembentukan partikel menunjukkan bahwa kristalisasi $SnSO_4$ berlangsung melalui tahapan nukleasi, pertumbuhan kristal, aglomerasi, dan pematangan. Ukuran partikel meningkat dari sekitar 2,3 µm menjadi 5,5 µm setelah 6 jam, menunjukkan transformasi dari prekursor berbasis timah (Sn–O/Sn–O–$SO_4$) menjadi kristal $SnSO_4$ yang lebih stabil dan terorganisasi. Pendekatan momen distribusi ukuran partikel menunjukkan bahwa laju pertumbuhan kristal menurun mengikuti hubungan $G_L = 3{,}15 \times 10^{-8}\, t^{-0{,}486}$, sedangkan laju nukleasi dan kernel aglomerasi masing-masing dinyatakan dengan $B_0 = 1{,}77 \times 10^{5}\, G_L^{0{,}81}\, M_T^{-1{,}27}$ dan $\beta = 2{,}41 \times 10^{3}\, G_L^{0{,}02}\, B_0^{-1{,}19}\, t^{-2{,}18}$. Hubungan tersebut menunjukkan bahwa pertumbuhan awal dikendalikan oleh supersaturasi tinggi, kemudian bergeser menuju kontrol difusi ion dan integrasi permukaan, dengan aglomerasi yang dominan pada tahap awal kristalisasi. Secara keseluruhan, integrasi oksidasi elektrokimia dan kristalisasi evaporatif efektif menghasilkan $SnSO_4$ anhidrat *orthorhombic* dengan morfologi *prismatic*, kristalinitas tinggi, dan karakter yang mendekati $SnSO_4$ komersial/standar.

**Kata kunci:** efisiensi Faraday, kristalisasi evaporatif, membran Nafion 115, oksidasi elektrokimia, $SnSO_4$.

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# ABSTRACT

*Tin (Sn) is regarded as a strategic metal with extensive applications in electronics, catalysis, and functional material development. Among its important derivatives, tin(II) sulfate ($SnSO_4$) is widely recognized as a valuable precursor for batteries, gas sensors, and optoelectronic materials. Nevertheless, conventional routes for $SnSO_4$ production commonly require multi-step chemical procedures, additional reagents, and prolonged processing times. Therefore, this study was designed to develop a controlled synthesis route for $SnSO_4$ by integrating electrochemical oxidation of tin with evaporative crystallization. The synthesis was carried out in a two-compartment electrochemical cell separated by a Nafion 115 membrane, using Sn as the anode and graphite as the cathode. Each anolyte and catholyte chamber had a capacity of 175 mL, with $H_2SO_4$ solution employed as the electrolyte and an anolyte volume of 150 mL. The investigated electrochemical variables included $H_2SO_4$ concentration (0.01–1.0 M), applied voltage (0.5–3.0 V), and electrolysis duration (4–8 h). Faradaic efficiency was optimized using Response Surface Methodology (RSM) based on a Box–Behnken experimental design. The $SnSO_4$ concentrate obtained from electrolysis was further processed by evaporative crystallization at 55–85 °C with seed concentrations of 0–1.2 g/L. The $SnSO_4$ seed particles had an average size of 2.2 µm. The resulting products were characterized using SEM-EDS, PSA, XRF, FTIR, and XRD.*

*The findings demonstrate that electrochemical oxidation of Sn in $H_2SO_4$ medium effectively produced a $SnSO_4$ concentrate solution suitable as a precursor for crystallization. Increasing both the $H_2SO_4$ concentration and applied voltage enhanced the dissolution of Sn to $Sn^{2+}$; however, overly severe conditions promoted passivation and side reactions. The RSM optimization identified the optimum operating conditions as 0.64 M $H_2SO_4$, 2.09 V, and 5.77 h electrolysis time, under which maximum Faradaic efficiency was predicted. Subsequent evaporative crystallization successfully transformed the $SnSO_4$ concentrate into orthorhombic anhydrous $SnSO_4$ crystals. Evaporation temperature was found to be the most influential parameter, with crystallization at 85 °C without seed addition yielding approximately 95% crystallization efficiency. The addition of seed crystals at 0.6–1.2 g/L accelerated only the early crystallization stage and did not substantially improve the final conversion. Particle formation analysis revealed that $SnSO_4$ crystallization involved sequential nucleation, crystal growth, agglomeration, and ripening. The particle size increased from approximately 2.3 µm to 5.5 µm after 6 h, indicating the transformation of tin-based precursors (Sn–O/Sn–O–$SO_4$) into more stable and structurally ordered $SnSO_4$ crystals. The particle size distribution moments approach indicated that the crystal growth rate decreased according to $G_L = 3.15 \times 10^{-8}\, t^{-0.486}$, while the nucleation rate and agglomeration kernel were described by $B_0 = 1{,}77 \times 10^5\, G_L^{0,81}\, M_T^{-1,27}$ and $\beta = 2{,}41 \times 10^3\, G_L^{0,02}\, B_0^{-1,19}\, t^{-2,18}$, respectively. These correlations suggest that the initial growth stage was governed primarily by high supersaturation, followed by a transition toward ion diffusion and surface integration control, while agglomeration was dominant during the early crystallization period. Overall, the combined electrochemical oxidation–evaporative crystallization route proved effective for producing orthorhombic anhydrous $SnSO_4$ with prismatic morphology, high crystallinity, and properties closely comparable to those of commercial/standard $SnSO_4$.*

***Keywords**: electrochemical oxidation, evaporative crystallization, Faradaic efficiency, Nafion 115 membrane, $SnSO_4$.*