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Synthesis and Characterization of Solid-State Electrolyte NASICON ($Na_3Zr_2Si_2PO_{12}$) from Different Precursor Sources

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INTRODUCTION & AIM

Introduction:

- Rapid growth of renewable energy and electric vehicles increases demand for safe and efficient energy storage.
- Lithium-ion batteries (LIBs) dominate but face issues like high cost, scarcity of Li, and safety risks.
- Sodium-ion batteries (SIBs) are promising due to low cost, abundant Na resources, and environmental friendliness.
- Finding a suitable solid-state electrolyte is crucial for improving SIB performance and safety.
- NASICON-type materials ($Na_{1+x}Zr_2Si_xP_{3-x}O_{12}$) offer high ionic conductivity (~10^{-3} S/cm), thermal stability, and chemical robustness.
- Among them, $Na_3Zr_2Si_2PO_{12}$ (NZSP) is a leading candidate for solid-state sodium-ion batteries.
- The purity of precursor chemicals strongly influences the phase formation, microstructure, and conductivity of NASICON.

Aim:

1. To synthesize NASICON (NZSP) using chemicals from high-purity-grade supplier and economical but low-purity-grade supplier.
2. To compare their structural and electrochemical properties.
3. To assess whether low-cost chemicals (Sample-B) can match high-purity chemicals (Sample-A).

METHOD

Synthesis Route: Solid-State Reaction Method

Steps:

1. **Precursors:** Na_2CO_3, ZrO_2, SiO_2, $NH_4H_2PO_4$
2. **Stoichiometry:** Mixed in molar ratio 3:4:4:2
3. **Grinding:** 3 hours with ethanol using mortar–pestle
4. **Pre-heating:** 100 °C for 12 h
5. **Calcination:** 1000 °C for 10 h
6. **Pelletizing:** 13 mm die under 250 kg/cm² for 15 min
7. **Sintering:** 1050 °C for 9.5 h
8. **Characterization:**
    1. **Electrochemical Impedance Spectroscopy (EIS)**
    2. **X-Ray Diffraction (XRD)**

RESULTS & DISCUSSION

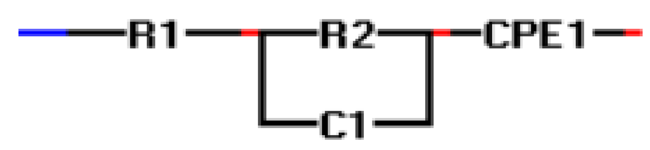
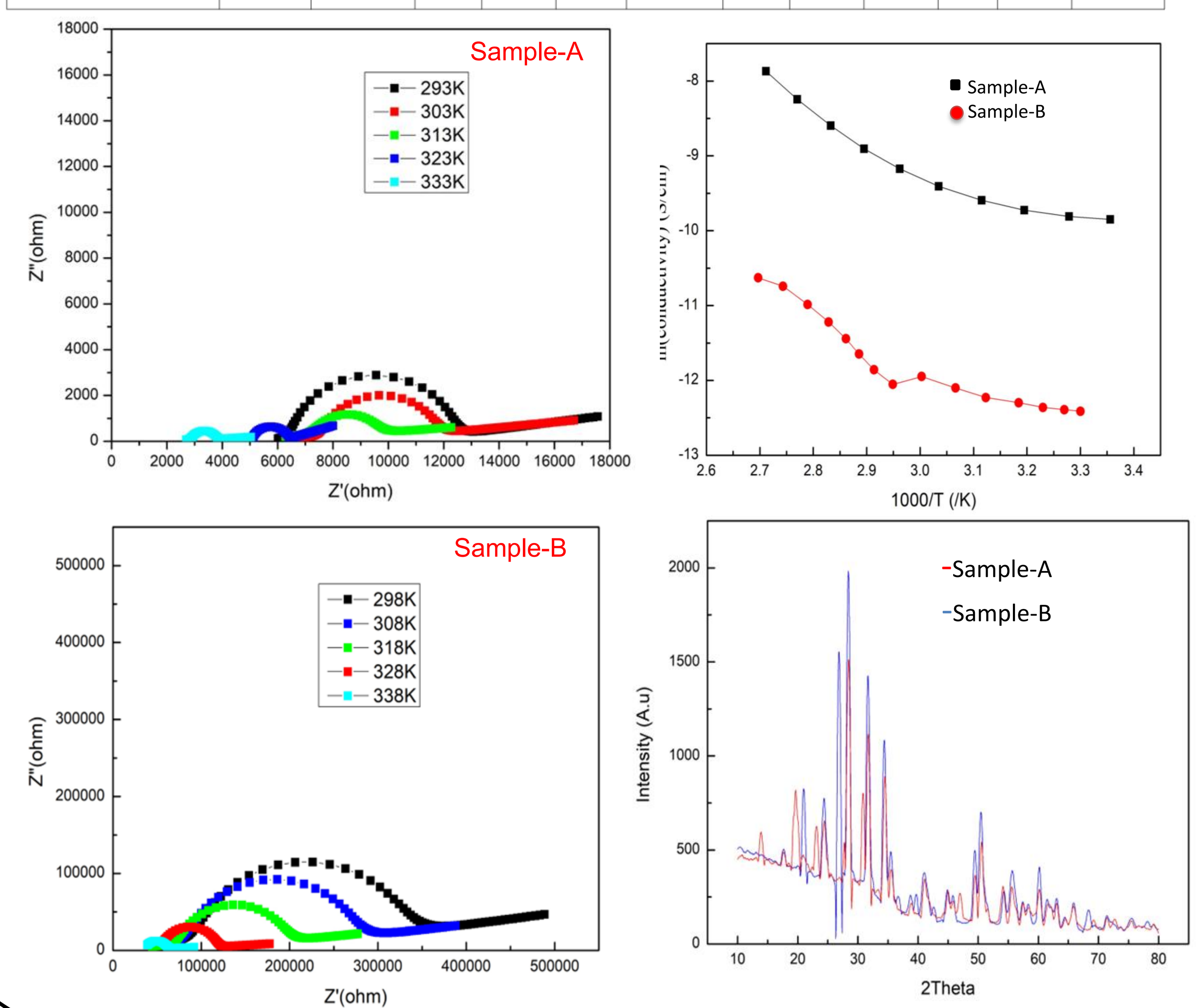


Fig 8: Equivalent circuit of Nyquist plot

Density of the pellets after sintered	
Sample-B	Sample-A
2.82 g/cm³	2.91 g/cm³

Cost of precursors to synthesize 2 mol NZSP

PRECURSORS	WEIGHT (g)	purity(%)	price	price/g	Weight	Sample-B (Rs.)	WEIGHT(g)	Purity (%)	price	price/g	Weight	Sample-A (Rs.)
SODIUM CARBONATE	500	99.5	298	0.596	318	189.528	500	99.50	6210	12.4	318	3950
AMMONIUM DIHYROGEN ORTHOPHOSPHATE	500	98	588	1.176	230	270.48	500	99.999	56820	114	230	26137
SILICON DIOXIDE	1000	99	1503	1.503	240.332	361.219	1000	99.999	46893	46.9	240.3	11272
ZIRCONIUM DIOXIDE	500	98.5	2853	5.706	492.88	2812.373	500	99	16480	33	492.9	16245
Total						3633.6						57603



CONCLUSION

- NASICON ($Na_3Zr_2Si_2PO_{12}$) was synthesized using high-purity source and low-purity source precursors via the solid-state reaction method.
- Sample-A showed lower grain boundary resistance, higher density (2.91 g/cm³), and purer NZSP–ZrO_2 phases.
- Sample-B exhibited higher resistance, lower density (2.82 g/cm³), and greater porosity with an impurity sodium silicate phase.
- These results confirm that precursor purity significantly affects microstructure, porosity, and ionic conductivity.
- Overall, high-purity precursors source yield denser, purer, and more conductive NASICON than low-purity source.

FUTURE WORK / REFERENCES

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