WORLD JOURNAL OF PHARMACEUTICAL RESEARCH
Coden USA: WJPRAP Impact Factor 8.453
Volume 14, Issue 21, 1682-1687. Research Article ISSN 2277–7105

THE PREPARATION, CHARACTERIZATION AND ELECTRICAL PROPERTIES OF SODIUM-BASED DIPHOSPHATES AND DIARSENATES

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Article Received on 10 October 2025,
Article Revised on 30 October 2025,
Article Published on 01 Nov. 2025,
https://doi.org/10.5281/zenodo.17541322

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How to cite this Article: *1Dr. Masheir Ebrahim Baleil, 2Dr. Mohammed Salem Abd Elfadil (2025 THE PREPARATION, CHARACTERIZATION AND ELECTRICAL PROPERTIES OF SODIUM-BASED DIPHOSPHATES AND DIARSENATES. "World Journal of Pharmaceutical Research, 14(21), 1682–1687.

ABSTRACT

This study aims to prepare to substitute lithium cation by sodium ions as cathode in rechargeable ion-batteries. Particularly, new materials in the Na-M -As-O systems (M= transition metal) was synthesized by solid-state reaction route or sol-gel process. X-ray diffraction was used to determine the crystal structure of each compound. In order to obtain $Na_2MX_2O_7$ dense ceramics, the optimal sintering temperature was determined. Their microstructure of Na-M- As-O was characterized by scanning electron microscopy. Electrical measurements were carried out using impedance spectroscopy. In theoretical studies, the bond valence site Maps (BVSM) model or Bond Valence Site Energy (BVSE) was used to simulate the conduction pathways of the monovalent cations, allowing a better correlation between the electrical properties and structure.

INTRODUCTION

The $Na_2MP_2O_7$ systems (M= transition metal)[1-2] in particular have an anionic layered framework $[MP_2O_7]$n 2n-, with sodium cations localized in between its layers. Because the open framework's layers are so far apart, these compounds' ionic conductivity performances are noteworthy. Enhancing ion mobility in $Na_2MP_2O_7$ systems (M=transition metal) is our aim. According to this method, arsenic is suggested as a partial and complete replacement for phosphorus. It is true that arsenic has a larger ionic radius than phosphorus, and this replacement can make the openings larger and improve ionic conduction. In contrast to $Na_2CoP_2O_7$ (σ300°C = 2×10-5 Scm-1; Ea = 0.63 eV), recent studies really demonstrate the intriguing electrical conductivity of the Na2CoP1.5As0.5O7 material[3], with an electrical conductivity value of σ240°C = 7.91×10-5 Scm-1 and an activation energy Ea = 0.56 eV.[4] We investigated the Na2MP2-xAsxO7 systems in this context (x = 0.5; 1.0), where M = Zn, Cu, Mn, Co, and Ni elements. The physicochemical and structural investigations of P/As doped sodium cobalt diphosphate materials are the focus of this work.

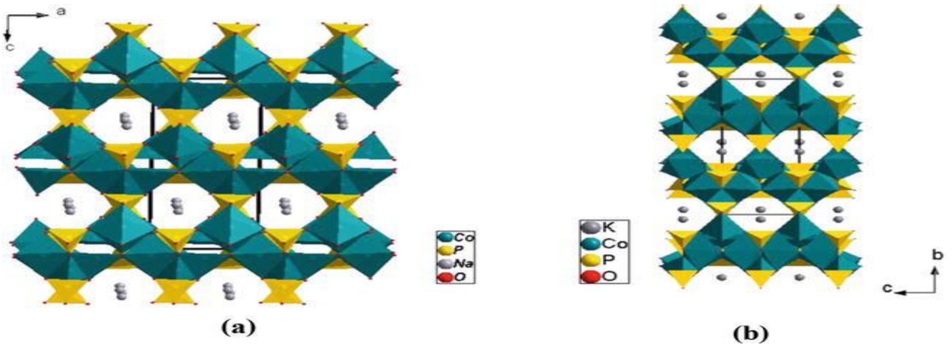

Figure (1.3): Projection of $KCo_4(PO_4)_3$ structure along [010] direction.

MATERIALS AND METHODS

The material and methods chapter is divided into three parts; description of the solid-state method, reagents characteristics description of sample synthesis and the experimental techniques of characterization.

2.1. Solid State Reaction route

the solid-state reaction method is the most adopted technique to synthesis polycrystalline powder and single crystals materials. We can describe the essential steps as:

- Grinding the mixture of the solid reagents by (agate, porcelain, etc.) mortar or mechanical grinder (planetary grinder).
- Calcination: heating the mixture for a few hours at 300-400 ^{o}C to eliminate the volatile products such as H2O, CO2, NH3, etc.
- Thermal heating of the residue at a temperature close to the melting temperature of the compounds. The equipment and glassware necessary in the solid-state method are:
- Crucibles or nacelles, usually in porcelain or sometimes in silica or platinum.
- Muffle furnace that is preferable automatic for heating.

- Porcelain or agate mortar for the fine grinding of samples. The mechanical grinding is also preferable.

2.2. Synthesis

Two mixture series of Metal M +II nitrate or acetate hydrate, NH4H2PO4, Na2HAsO4.7H2O, and NaNO3 (if needed) in two different molar rations Na: M: P: As equal to 2:1:1.5:0.5 and 2:1:1:1 were placed in porcelain crucibles and heated to 350°C during 24 h to eliminate the volatile products H2O, NO2, CO2 and NH3. Each obtained powder was grounded manually using agate mortar and shaped as cylindrical pellet by uniaxial press. The obtained pellets were heated progressively to 600 °C. After 72h, the sample was cooled slowly with the rate of 10°C/h down to the room temperature.

2.3. Characterization

In this section, we present the different characterization techniques and data processing software used for the realization of this work. We distinguish the following characterization techniques: Powder X-ray diffraction (XRD), Infrared spectroscopy (IR), Differential Scanning Calorimetry (DSC), Scanning Electron Microscope (SEM).

Complex impedance spectroscopy and Software for processing results.

Table 1: Structure refinement results of the $Na_2CuP_{1.5}As_{0.5}O_7$ compound.

Crystallographic Data	
Empirical Formula	$Na_2CuP_{1.5}As_{0.5}O_7$
Formula Weight; ρ_{cal}	305.44 g mol^{-1}; 3.220 g cm^{-1}
Crystalline System; Space Group	Monoclinic, C2/c
Unit Cell Dimensions	a = 14.8688 (8), b = 5.7591 (3), c = 13.5957 (7) β = 147.2406 (12)
Volume; Z	V = 629.97 (6) $Å^3$; 4
Data Collection	
Diffractometer	Bruker D8 ADVANCE
Wavelength	$\lambda_{Cu\ K\alpha}$ = 1.54056 Å
Temperature	298 (2) K
Angle Range	4.91°–69.91°
Step Scan Increment (°2θ)	0.02°
Counting Time	2

3. RESULTS

In this work, we isolated a novel sodium diphosphate-diarsenate member, Na2CuP1.5As0.5O7, in the polycrystalline form by exploring the Na2MP1.5As0.5O7 and Na2MPAsO7. However, the other transition metals (Zn, Ni, and Mn) demonstrate that in order to get pure crystalline samples, the synthesis technique utilizing the aforementioned regents must be modified. The same synthesis technique, the solid-state reaction, can also be used to alter the precursors. The results obtained for the new Na2CuP1.5As0.5O7 material demonstrate the effectiveness of the synthesis procedure used to produce a pure and well-crystallized powder. The prepared compound might be controlled thanks to the various characterisation approaches. The acquired sample's good crystallinity is revealed by the XRD pattern's intense diffracted peaks. Our material's purity is demonstrated by the indexing of all the diffractogram's peaks. As of right now, our substance is the second sodium metal diphosphate-66 diarsenate. Actually, only Na2CoP2-xAsxO7 systems are examined in the sodium metal P/As materials; the explored systems Na2MP2-xAsxO7 are essentially virgin. We observe right away that it is not the same structure as the sodium cobalt diphosphate-diarsenate Na2CoP1.5As0.5O7 that was recently studied by Marzouki and colleagues (2019).[3] Indeed, the cobalt material crystallizes in the tetragonal system of the P42/mnm space group with the unit cell parameters a=7.764(3) Å, c=10.385(3) Å. While, the studied material Na2CuP1.5As0.5O7, crystallizes in the monoclinic system of the C2/c space group with the unit cell parameters a= 14.798(2) Å; b= 5.729(3) Å; c= 8.075(2) Å; β= 5.00(3)°. Figures illustrates the remarkable difference between our studied structure and that of the Na2CoP1.5As0.5O7 material.

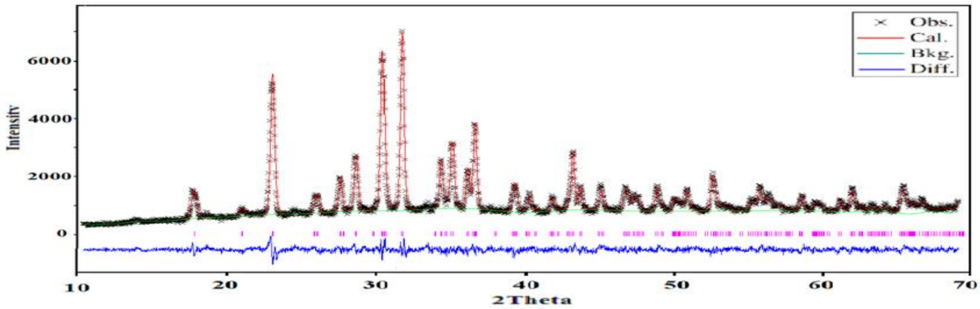

Figure 2: Results of Rietveld refinement of the powder of Na2CuP1.5As0.5O7 based on XRD data. Blue and red curves and violet line: experimental data and calculated profile with their difference, respectively. Tick marks: positions of the Bragg peaks.

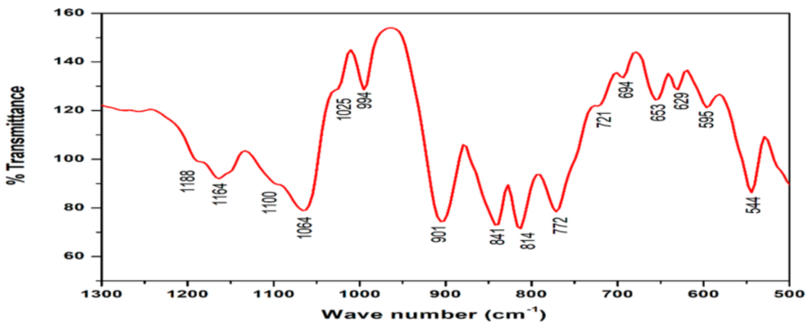

Figure 11: FT-IR spectrum (1300–500 cm^{-1}) of Na2CuP1.5As0.5O7

ACKNOWLEDGEMENTS

We are grateful to University of White Nile, University of Imam Mahdi staff.

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