

# Hydrothermal Synthesis and Crystal Structure of 3D Hydrogen Bonded Framework Obtained by Linear Chain: $[\text{Ni}(\text{pyridine 2,6 dicarboxylic acid})_2(\text{K})_2]_7(\text{H}_2\text{O})_n$ , polymer

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## 1. Introduction

The transition metal based MOFs have been the subject of intensive research in the last decades but the alkali and alkaline earth metals which offer higher coordination number and more flexible coordination geometry and may produce structures with charming topologies different from transition metals has not been given due importance. The metal organic frameworks built by carboxylate group containing linkers are most commonly reported. Beside carboxylate other popular linking units are imidazole and pyridine based linkers. Dicarboxylate ligands are widely used to assemble supramolecular network structures organized by coordination bonds, hydrogen bonds and  $\pi$ - $\pi$  stacking interactions. To synthesize novel MOFs having unique structure and properties, much research is being focused on choosing the preferred geometry and function of metal ions and organic ligands. The members of aromatic polycarboxylic acid, pyridine dicarboxylic acid, which can offer possibilities to form MOFs through both carboxylate and N atom of pyridine ring is the preferred choice. A series of extended coordination polymers from pyridine-2, 6 dicarboxylic acid and with transition metal centres have been constructed. These system display distinct architecture varying from 1D helix, 2D brick wall and herringbone to 3D interpenetrated structures [1].

The research on metal organic frameworks has generally based on assembly of organic ligands with d-block metal ions. The research on alkali and alkaline earth metal ions for the synthesis of MOFs has been undeveloped. Transition metal coordination polymers constructed by multi-carboxylate ligands have been extensively studied, as these linkers give rise to materials which can have potential applications like zeolite materials. These materials find application in molecular selection, catalysis ion exchange, non-linear optics and microelectronics, besides giving variety of interesting architectures and topologies. Recently, the extended structure of supra-molecular compounds by the self-assembly of metal-organic units via hydrogen bonds and pi-pi interaction has attracted much interest [2].

The MOFs built by aromatic carboxylate and transition metal are the most famous among this family of new materials and have been the subject of investigation of many regroups [3]. To the best of our knowledge, MOFs of dicarboxylate with mixed metal ions such as alkali metal ions and transition metals are very less reported [4].

Here we report a new 3D MOFs constructed by alkali metals and transition metal together with 2, 6-pydc.  $\text{Ni}^{2+}$  is transition metal and  $\text{K}^+$  alkali metal while organic linker is pyridine 2, 6-dicarboxylic acid. The network is built by  $[\text{NiK}_2(2,6\text{-pydc})_2(\text{H}_2\text{O})_7]$ , in which water molecules also act as ligand. This coordination network is characterized by IR spectroscopy, thermal analysis and single crystal X-ray analysis. The nickel atom has a slightly elongated axially distorted octahedral coordination. In this structure, most interesting aspect is that  $\text{K}^+$  ion shows two different coordination numbers, 6 and 7.  $\text{K}_2$  is showing 7 while  $\text{K}_1$  shows 6 coordination numbers. The carboxylate group of pyridine 2, 6-dicarboxylate ligand adopts  $\mu$ -2 bridging mode, oxygen atom of carboxylic group coordinates with both two metals  $\text{Ni}^{2+}$  and  $\text{K}^+$  ions. The presence of water molecules and carboxylic groups makes extensive intermolecular Hydrogen bonding.

## 2 Materials and Methods

### 2.1 Materials and Physical Measurement of Compound

Nickel (II) acetate tetra- hydrate (Sigma Aldrich), Potassium chloride (Aldrich) and Pyridine 2,6-dicarboxylic acid were purchased and used without further purification. IR spectra were recorded in KBr pellets with a Nicolet 170 SXFT-IR spectrophotometer in the 4000–400  $\text{cm}^{-1}$  region. In order to reveal the thermal stability of compound TGA measurements has been carried out on Perkin-Elmer Diamond TGA/DTA instrument with flow of dry nitrogen at a heating rate of 5<sup>o</sup>C/min from room temperature to 1000<sup>o</sup>C. Variable-temperature magnetic susceptibility data were collected using a Quantum Design MPMS SQUID magnetometer. Magnetic susceptibility of complex was corrected for diamagnetism of the constituent atoms, which was calculated using Pascal's constant.

## 2.2 Synthesis of Compound

A mixture of Nickel acetate tetrahydrate (0.246; 1.0 mmol), KCl (0.410 g; 3.0 mmol), pyridine-2,6-dicarboxylic acid (0.312 g, 3.0 mmol) and H<sub>2</sub>O (10 ml), sealed in a 20ml Teflon-lined reactor and was heated in an oven at 170 °C for 48h followed by cooling to room temperature. The resultant green colour solution was obtained. After filtration the final clear solution left undisturbed at room temperature for slow evaporation. After two week, star shaped green crystals were collected and dried in vacuum over silica gel and immersed in silicon oil.

## 2.3 Crystallographic details of compound

The crystal structure was solved by direct methods using the program SHELXS of the SHELX-97 program package [5]. Single crystal X-ray data were collected at 100 K on a Bruker SMART APEX diffractometer using graphite monochromatic MoK $\alpha$  radiation ( $\lambda = 0.71069\text{\AA}$ ). The linear absorption coefficients, scattering factors for the atoms, and the anomalous dispersion corrections were taken from International Tables for X-Ray Crystallography [6]. The data integration and reduction were processed with SAINT software.

An empirical absorption correction was applied to the collected reflections with SADABS using XPREP. The structure was refined on F<sup>2</sup> by full-matrix least-squares technique using non-hydrogen atoms were refined anisotropically. The hydrogen atoms were geometrically fixed and treated as riding atoms using SHELXL default parameters. Crystal data and structure refinement parameters are listed in Table 6.1. The final  $R = 0.0736$ ,  $wR = 0.1422$   $w = 1/[\sigma^2(F_o^2) + (0.0731P)^2 + 2.3926P]$  where  $P = (F_o^2 + 2F_c^2)/3$ ,  $S = 1.092$ ,  $(\Delta\rho)_{\max} = 0.736 \text{ e/\AA}^3$ ,  $(\Delta\rho)_{\min} = -0.719 \text{ e/\AA}^3$  and  $(\Delta/\sigma)_{\max} = 0.001$ .

## 2.4 Geometrical special details

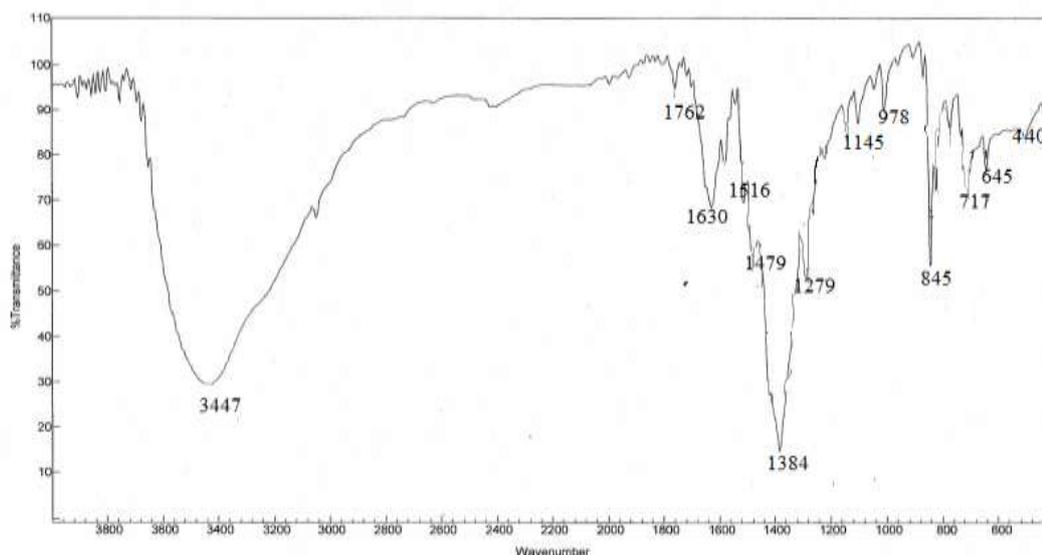
All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds involving l.s. planes.

## 2.5 Refinement special details

Refinement of F<sup>2</sup> against all reflections. The weighted R-factor wR and goodness of fit S are based on F<sup>2</sup>, conventional R-factors R are based on F, with F set to zero for negative F<sup>2</sup>. The threshold expression of F<sup>2</sup> > 2 $\sigma$ (F<sup>2</sup>) is used only for calculating R-factors (gt) etc. and is not relevant to the choice of reflections for refinement. R-factors based on F<sup>2</sup> are statistically about twice as large as those based on F, and R-factors based on all data will be even larger.

## 3 Results and Discussion

### 3.1 IR Spectral Analysis of Compound



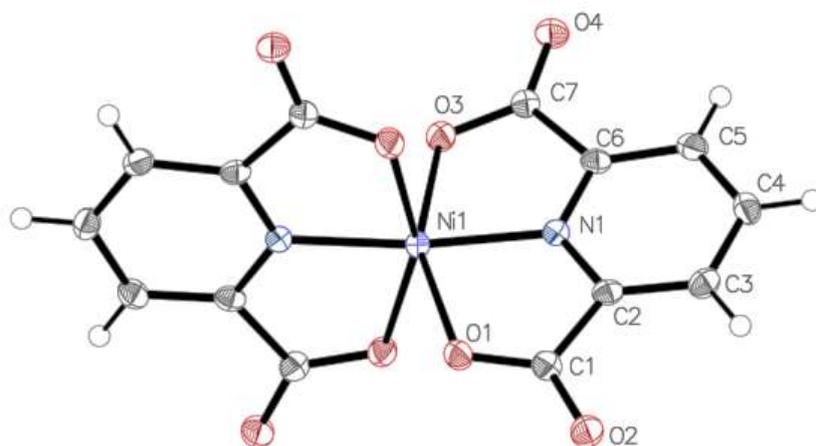
**Figure 1: IR spectrum of [NiK<sub>2</sub>(2,6-pvdc)<sub>2</sub>(H<sub>2</sub>O)<sub>7</sub>]<sub>n</sub> polymer**

The IR spectrum shows peak at  $3447\text{ cm}^{-1}$  that confirm the presence of the water molecule hydrogen bonded to the structure. The typical antisymmetric ( $1630\text{ cm}^{-1}$ ) and symmetric ( $1516$  and  $1479\text{ cm}^{-1}$ ) stretching bands of carboxylate ions are also observed in the IR spectrum. Other skeletal ring vibrations observed at  $1279\text{ cm}^{-1}$ ,  $1145\text{ cm}^{-1}$ ,  $978\text{ cm}^{-1}$ ,  $645\text{ cm}^{-1}$  [7]. (Figure 6.1)

### 3.2 Single Crystal Structure

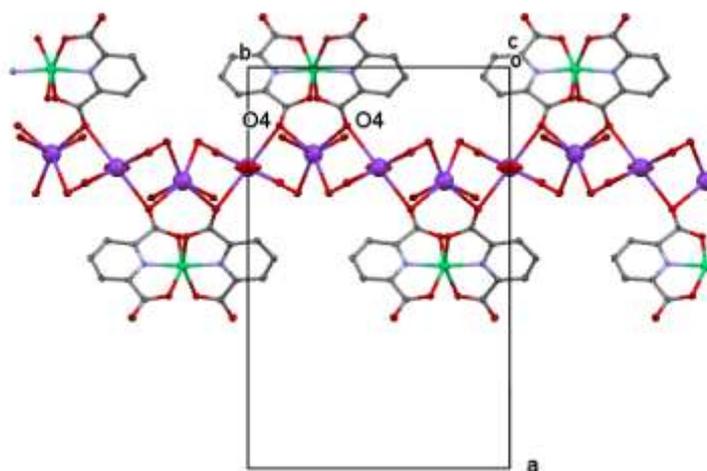
The single crystal X-ray crystallographic studies revealed the compound  $[\text{NiK}_2(2,6\text{-pydc})_2(\text{H}_2\text{O})_7]$  crystallizes as a coordination polymer in the orthorhombic space group Pnna.

One unit cell consists of two similar nickel atoms Ni1, two identical potassium atoms with six coordination number K2, one potassium atom with seven coordination number K1, two pyridine 2, 6 dicarboxylic acid ligand, and eight water molecules. Crystallographic data is summarized in Table 6.1.

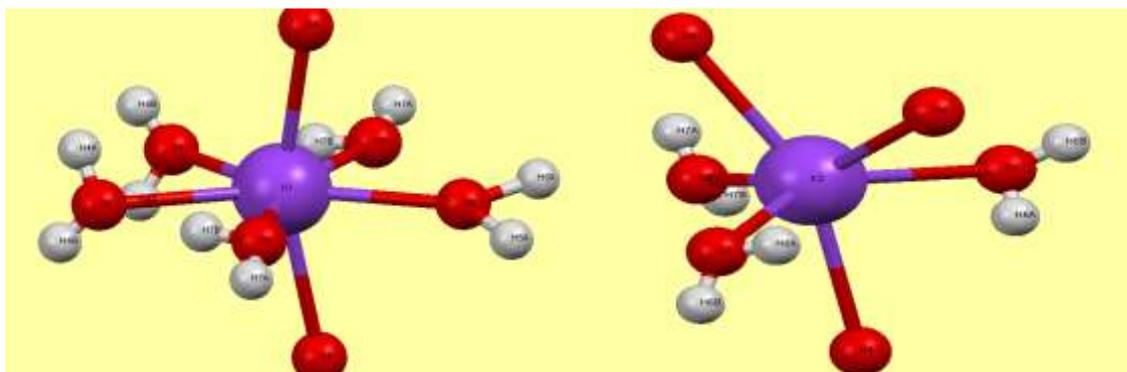


**Figure 2: ORTEP view of the complex anion  $\text{Ni}(\text{pydc } 2,6)_2$  with the atom labelling. Thermal ellipsoids are drawn at 50% probability level.**

The octahedrally coordinated nickel (II) ion lies on crystallographic 2-fold axis. The Nickel coordination consists of one N atom, two O atoms from the carboxylate group from each of the two pyridine dicarboxylic acid rings. The nickel atom has a slight axially distorted octahedral coordination; four carboxylate group O atoms are equatorial with bond distances Ni1-O1 and Ni1-O3 are  $2.142\text{ \AA}$  and  $2.107\text{ \AA}$  respectively and the two pyridine axial N atoms are at bond distances of (Ni-N1)  $1.967\text{ \AA}$  (Figure 6.2). The coordination geometry is in good agreement with Ni complexes reported in the literature [8].



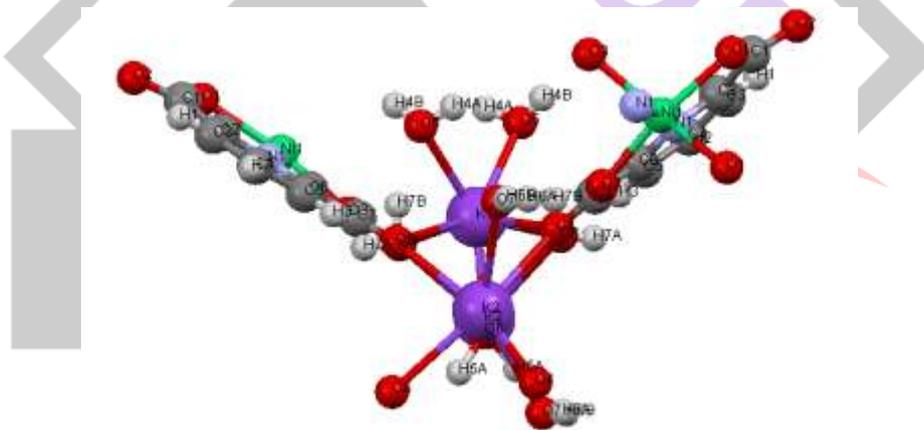
**Figure 3: One dimensional coordination polymer along b-axis.**



**Figure 4: showing diverse coordination of Potassium ions, K1 and K2**

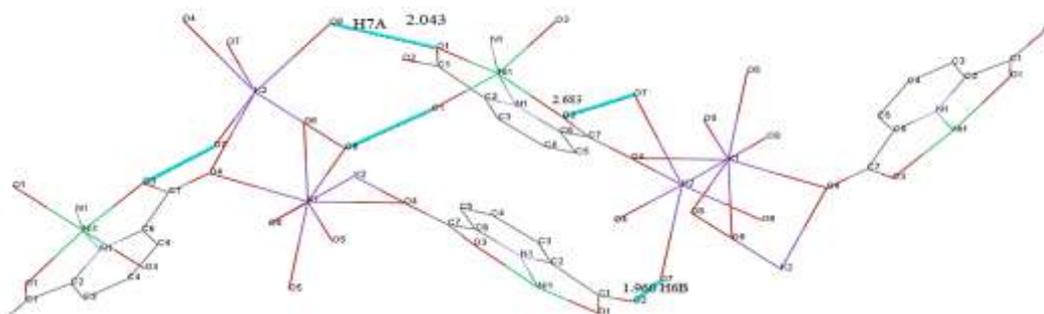
Crystal structure contains infinite one dimensional coordination polymers that run along b-axis (Figure 3). In this polymer, molecules of  $[\text{Ni}(\text{pydc})_2]^{2-}$  anions from either side coordinate via oxygen atoms (O4) to Potassium ions  $\text{K1}^+$  and  $\text{K2}^+$ .

The Potassium ions adopt diverse coordination numbers in the polymer; K1 showing 7 and K2 showing 6 coordination number (Figure 6. 4). In the polymer, K1 is coordinated by three water oxygen (bond distance 2.725(3) Å), two oxygen atoms (at bond distance 2.902(2) Å) from bridged water molecules and two oxygen [at bond distance 2.720(2) Å] from carboxylate group showing pentagonal bipyramid geometry. K2 is coordinated by two oxygen (O5) from non-bridged waters (bond distance of 2.752(3) Å), two bridged oxygen (O4) from carboxylates (K1-O4) is 2.72(2) Å and two oxygen (O8) from bridged water molecules (K1-O8 is 2.902(2) Å) having distorted octahedral geometry. The ions K1 and K2 are doubly bridged to each other by two oxygen atoms O4 and O8 from the carboxylate group of the ligand and water molecule, respectively. The bridging angles K1-O4- K2 and K1-O8-K2 are 93.34(7) and 91.35(7), respectively.



**Figure 5: showing two nickel atoms and three potassium atoms make “V” shape structure.**

The potassium polyhedra are linked to a dimer via bridge to dimer via O4 and O4' as a common edge. The dianionic edge  $[\text{Ni}(\text{pydc})_2]^{2-}$  act as a building blocks to coordinate three Potassium atoms. As a result each group of two nickel atoms and three potassium atoms make “V” shape structure (Figure 6.5).

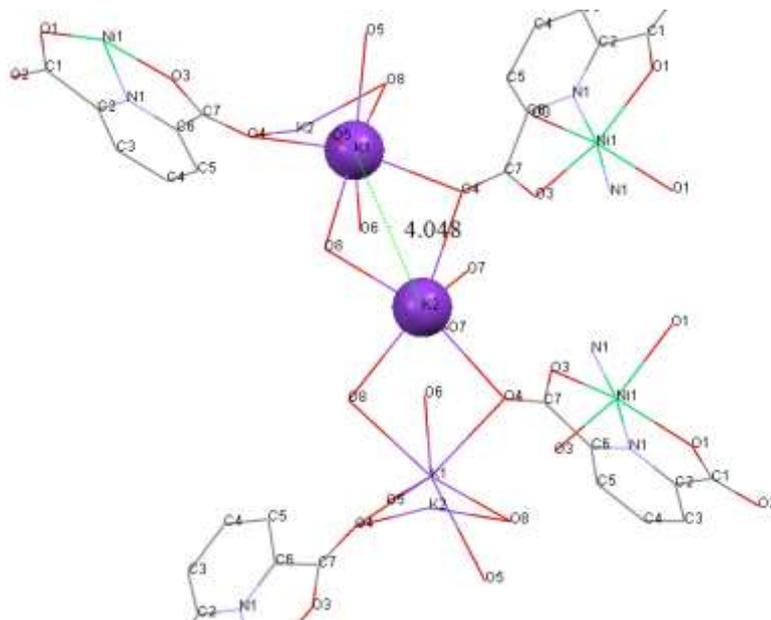


**Figure 6: Hydrogen bonding between Oxygen atom from carboxylic group and Hydrogen atom from water molecules forming these interlayer  $O2\cdots H6B-O7$  and  $O1\cdots H7A-O8$**

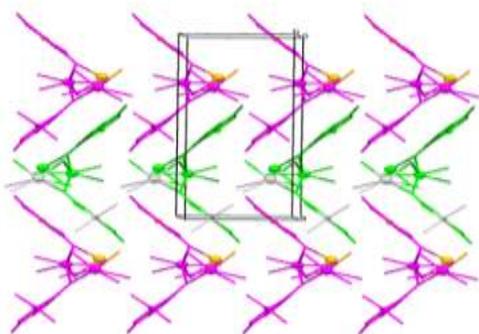
Table 1: Crystal data and structure refinement parameters

CCDC 900514	
Empirical formula	'C <sub>14</sub> H <sub>20</sub> K <sub>2</sub> N <sub>2</sub> Ni O <sub>15</sub> '
Formula weight	593.23
Temperature	123(2) K
Wavelength	0.71073 Å
Crystal system	Orthorhombic
Space group	p <sub>2</sub> 1n <sub>1</sub>
Unit cell dimensions	a = 20.594(4) b = 13.319(3) c = 8.1267(15)
Alpha	90.00
Beta	90.00
Gamma	90.00
Volume	2229.1(7) Å <sup>3</sup>
Z	4
Density (calculated)	1.768 Mg/m <sup>3</sup>
Absorption coefficient	1.323 mm <sup>-1</sup>
F(000)	1216
Theta range for data collection	2.50 to 28.33°
Index ranges	-26 ≤ h ≤ 27, -17 ≤ k ≤ 13, -9 ≤ l ≤ 10
Reflections collected	2770
Completeness to theta = 25.00°	99.2 %
Absorption correction	Semi-empirical from equivalents
Max. and min. transmission	1.00000 and 0.68978
Refinement method	Full-matrix least-squares on F <sup>2</sup>
Data / restraints / parameters	4752 / 3 / 132
Goodness-of-fit on F <sup>2</sup>	1.092
Final R indices [I > 2σ(I)]	R <sub>1</sub> = 0.0736, wR <sub>2</sub> = 0.1422
R indices (all data)	R <sub>1</sub> = 0.0469, wR <sub>2</sub> = 0.1139

The presence of water molecules and carboxylic groups makes extensive intermolecular hydrogen bonding between oxygen atoms O1, O2 from the carboxylic acid group with hydrogen atoms of the coordinated water molecules O7 and O8. Hydrogen bonding parameters are given in Table 2. The  $O2\cdots H6B-O7$  and  $O1\cdots H7A-O8$  hydrogen bond are formed with bond distance 1.960 Å and 2.043 Å. The intramolecular hydrogen bond is also formed between O3 of the carboxylate oxygen and H6A Hydrogen atom of coordinated water molecule ( $O3\cdots H6A-O7=1.921$  Å). The atoms O8' and O4' bridge K2 of one unit to K1 of another unit, the bridging angles K1-O4'-K2 and K1-O8'-K2 are 93.34(7) and 91.35(7) respectively and the distance between K1 and K2 is 4.048 Å. Selected bond length and bond angles are listed in Table 3 and 4.

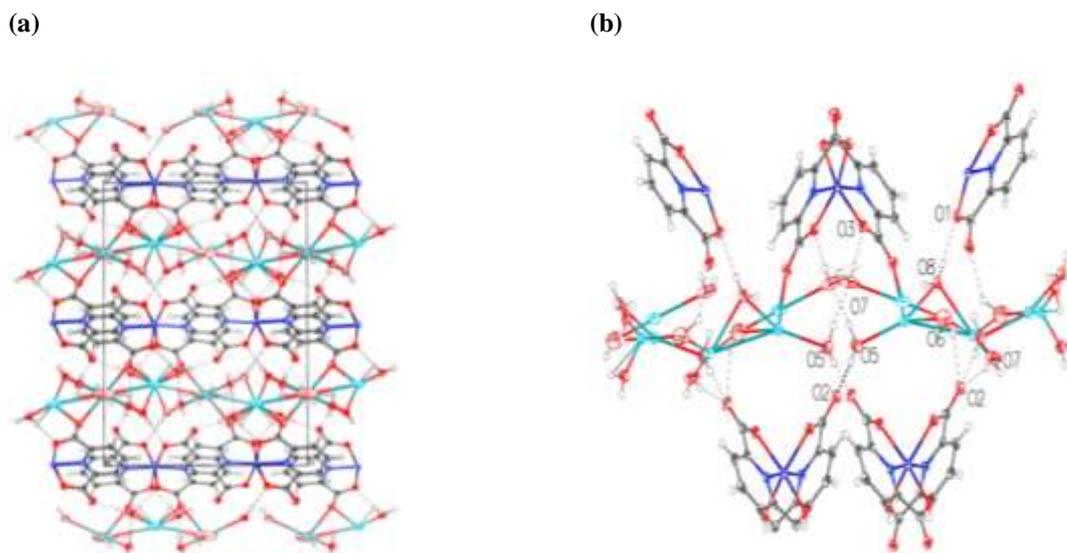


**Figure 7: showing distance between K1 and K2 (K1 and K2 doubly bridged belongs from the adjacent upside molecule)**



**Figure 8: Crystal Packing Diagram of Polymer along the b axis**

The additional hydrogen bonds formed between hydrogen atom of coordinated water molecules and oxygen atom of carboxylate group from pyridine 2,6 dicarboxylate ligand (O1--H7A-O8) with bond distance 2.043Å, Oxygen atom of carboxylate group from pyridine-2,6 dicarboxylic acid molecules makes bifurcated hydrogen bond (H4B- -O2- -H6B) with hydrogen atoms of coordinated water molecules bond distance (H4B-O2=2.055Å)(H6B-O2=1.960Å) and Oxygen atom of Water molecule (O7- -H4A-O5=1.914Å) bonded with Hydrogen atom of water molecule from adjacent unit. Hydrogen bonding distance and angles are summarized in Table 2.



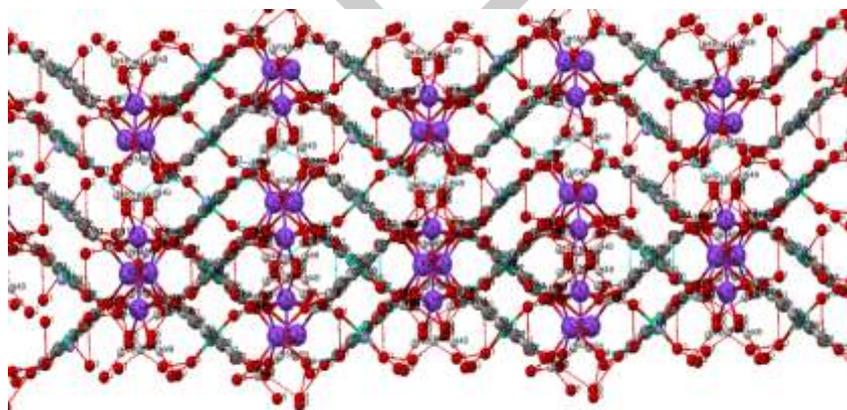
**Figure 9: showing oxygen atom of carboxylate group from pyridine 2,6 dicarboxylic acid molecules makes bifurcated hydrogen bond from hydrogen atoms of coordinated water molecules**

The crystal lattice shows extensive O-H...O hydrogen bonding network (Figure 9), a zoom in view is shown in the figure upper right(b). All the four water molecules O5, O6, O7 and O8 take part in the hydrogen bonding with the carbonyl oxygen O1, O2 and O3 (O4 is coordinated to the Potassium ion). The bridging atoms O8' and O4' bridge K2 of one unit to K1 of another unit, The bridging angles K1-O4- K2 and K1-O8-K2 are 93.34(7) and 91.35(7) respectively. The bond distance between K1 and K2 is 4.048Å, it gives overall 2D polymeric chain (Figure 10).

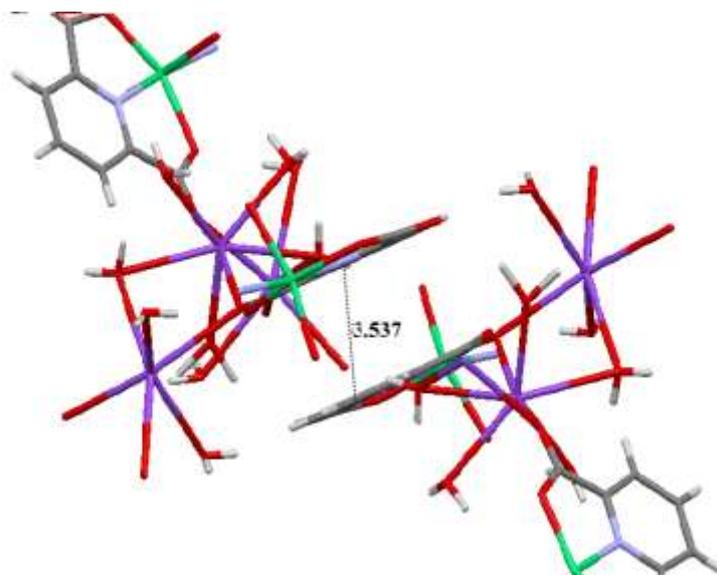
Table 2: Hydrogen bonding distances (Å) and angles (°) for polymer

Donor -H...Acceptor	D - H	H...A	D...A	∠D - H...A
O(5)--H(4A)..O(7)	0.87(4)	1.91(4)	2.770(4)	168(4)
O(5)--H(4B)..O(2)	0.79(5)	2.06(5)	2.831(3)	168(4)
O(6)--H(5A)..O(2)	1.13(12)	2.13(13)	3.056(3)	138(9)
O(7)--H(6A)..O(3)	0.76(5)	1.92(5)	2.683(3)	176(5)
O(7)-H(6B)..O(2)	0.85(6)	1.96(5)	2.778(4)	162(6)
O(8)-H(7A)..O(1)	0.79(5)	2.04(4)	2.828(3)	178(5)
O(8)-H(7B)..O(5)	0.83(4)	2.03(4)	2.786(4)	153(4)
C(5)-H(3)..O(8)	0.93	2.56	3.442(4)	158

Symmetry code (i)  $x, 1/2-y, 3/2-z$ , (ii)  $-1/2+x, y, 1-z$ ,  
 (iii)  $2-x, 1/2+y, -1/2+z$  (iv)  $1/2+x, 1/2-y, 1/2+z$  (v)  $-x, -1/2+y, -1/2+z$  (vi)  $-1/2+x, y, 2-z$

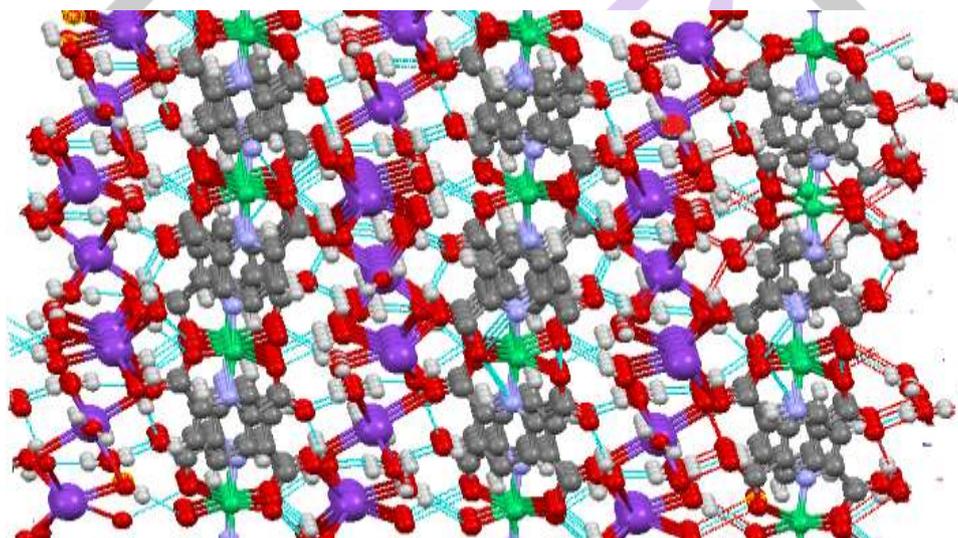


**Figure 10: 2D Polymeric chain showing regular zigzag channels along the b-axis**



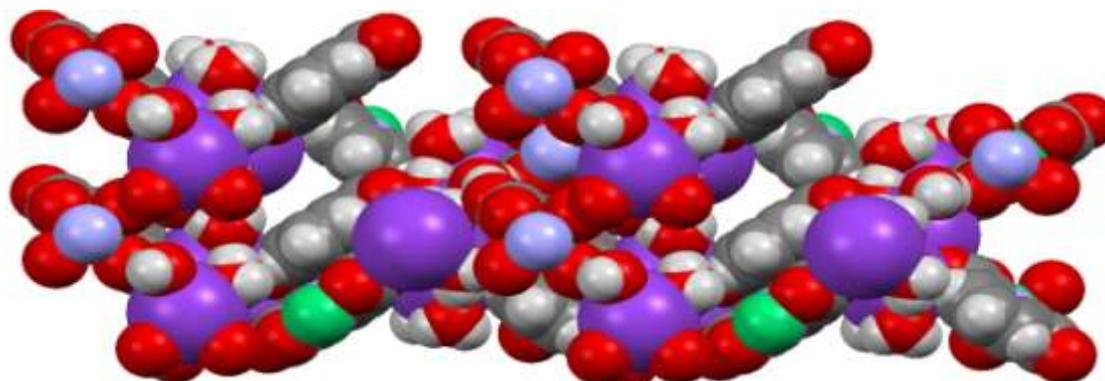
**Figure 11: Stacking of two pyridine rings of the neighbouring ligands through  $\pi$ - $\pi$  interactions running along the c axis for polymer**

The 3D polymeric structure is further stabilized by  $\pi$ - $\pi$  intermolecular stacking interactions established between the pyridine rings of the pyridine-2, 6 dicarboxylate ligands belonging to neighbouring chains ranging from 3.537 Å, (Figure 6.11) which is in good agreement with the reported values in the literature [9]. It gives additional strength to 3D structure. The van-der Waals interaction,  $\pi$ - $\pi$  stacking and hydrogen bonding gives further stability to 3D polymeric structure.



**Figure 12: Perspective View of 3D Polymeric chain along the b-axis**

The 3D polymeric chain formed with the aid of K2-O4-K1 and K2-O8-K1 interlayer. The front K2 atom of one molecule doubly bridged with K1 atom of adjacent molecule by O4 and O8 atom from carboxylic acid and water molecules respectively. The distance between K2-K1 is 4.048 Å (Figure 12). The space fill diagram along the b-axis showing large rectangular cavity (Figure 13).



**Figure 13: Space fill Diagram of Polymer Showing Large Rectangular Pores**

**Table 3: Bond angles for coordination polymer**

Atom1	Atom2	Atom3	Angle
O5	K1	O6	139.1(1)
O5	K1	O4	89.68(8)
O5	K1	O8	140.72(8)
O5	K1	O5	81.80(8)
O5	K1	O4	104.44(8)
O5	K1	O8	58.97(7)
O6	K1	O4	80.7(1)
O6	K1	O8	80.1(1)
O6	K1	O5	139.1(1)
O6	K1	O4	80.7(1)
O6	K1	O8	80.1(1)
O4	K1	O8	97.71(7)
O4	K1	O5	104.44(8)
O4	K1	O4	161.43(8)
O4	K1	O8	79.08(7)
O8	K1	O5	58.97(7)
O8	K1	O4	79.08(7)
O8	K1	O8	160.30(8)
O5	K1	O4	89.68(8)
O5	K1	O8	140.72(8)
O4	K1	O8	97.71(7)
O2	C1	O1	125.5(2)
O2	C1	C2	118.5(2)
O1	C1	C2	116.0(2)
O7	K2	O4	71.43(8)
O7	K2	O8	82.60(8)
O7	K2	O7	146.72(9)
O7	K2	O4	91.60(8)
O7	K2	O8	123.28(9)
O4	K2	O8	79.55(7)
O4	K2	O7	91.60(8)
O4	K2	O4	119.05(7)
O4	K2	O8	157.70(8)
O8	K2	O7	123.28(9)
O8	K2	O4	157.70(8)
O8	K2	O8	85.68(8)
O7	K2	O4	71.43(8)
O7	K2	O8	82.60(8)
O4	K2	O8	79.55(7)
K1	O5	H4A	97(3)
K1	O5	H4B	126(3)
H4A	O5	H4B	114(4)

Atom1	Atom2	Atom3	Angle
K2	O7	H6A	84(4)
K2	O7	H6B	129(4)
H6A	O7	H6B	105(5)
K1	O6	H5A	141(7)
K1	O6	H5A	141(7)
H5A	O6	H5A	78(9)
O1	Ni1	N1	77.57(9)
O1	Ni1	O3	155.42(8)
O1	Ni1	O1	87.12(7)
O1	Ni1	N1	107.84(9)
O1	Ni1	O3	96.78(8)
N1	Ni1	O3	78.14(9)
N1	Ni1	O1	107.84(9)
N1	Ni1	N1	172.8(1)
N1	Ni1	O3	96.69(9)
O3	Ni1	O1	96.78(8)
O3	Ni1	N1	96.69(9)
O3	Ni1	O3	89.72(8)
O1	Ni1	N1	77.57(9)
O1	Ni1	O3	155.42(8)
N1	Ni1	O3	78.14(9)
K1	O4	K2	93.34(7)
K1	O4	C7	134.2(2)
K2	O4	C7	125.4(2)
C1	O1	Ni1	114.1(2)
Ni1	N1	C6	118.4(2)
Ni1	N1	C2	119.9(2)
C6	N1	C2	121.6(2)
Ni1	O3	C7	114.8(2)
O4	C7	O3	126.2(3)
O4	C7	C6	119.6(2)
O3	C7	C6	114.1(2)
N1	C6	C7	113.1(2)
N1	C6	C5	120.9(2)
C7	C6	C5	126.0(2)
C1	C2	N1	111.9(2)
C1	C2	C3	127.0(2)
N1	C2	C3	121.0(2)
C6	C5	H3	120.9(3)
C6	C5	C4	118.2(2)
H3	C5	C4	120.9(3)
C5	C4	H2	119.9(3)
C5	C4	C3	120.1(3)
H2	C4	C3	120.0(3)
C2	C3	C4	118.2(3)
C2	C3	H1	120.9(3)
C4	C3	H1	120.9(3)
K1	O8	H7A	150(3)
K1	O8	H7B	77(3)
K1	O8	K2	91.35(7)
H7A	O8	H7B	101(4)
H7A	O8	K2	113(3)
H7B	O8	K2	123(3)

Table 4: Bond length for coordination polymer

Atom1	Atom2	Length
K1	O5	2.752(3)
K1	O6	2.578(5)
K1	O4	2.720(2)
K1	O8	2.902(2)
K1	O5	2.752(3)
K1	O4	2.720(2)
K1	O8	2.902(2)
C1	O2	1.256(3)
C1	O1	1.266(3)
C1	C2	1.518(4)
K2	O7	2.956(4)
K2	O4	2.844(2)
K2	O8	2.755(2)
K2	O7	2.956(4)
K2	O4	2.844(2)
K2	O8	2.755(2)
O5	H4A	0.87(4)
O5	H4B	0.79(4)
O7	H6A	0.76(5)
O7	H6B	0.85(6)
O6	H5A	1.1(1)
O6	H5A	1.1(1)
Ni1	O1	2.142(2)
Ni1	N1	1.967(3)
Ni1	O3	2.107(2)
Ni1	O1	2.142(2)
Ni1	N1	1.967(3)
Ni1	O3	2.107(2)
O4	C7	1.226(3)
N1	C6	1.338(4)
N1	C2	1.336(4)
O3	C7	1.285(3)
C7	C6	1.517(4)
C6	C5	1.383(4)
C2	C3	1.375(4)
C5	H3	0.931(3)
C5	C4	1.388(4)
C4	H2	0.930(3)
C4	C3	1.401(4)
C3	H1	0.930(3)
O8	H7A	0.78(5)
O8	H7B	0.83(4)
O8	K2	2.755(2)
C1	O2	1.256(3)
C1	O1	1.266(3)
C1	C2	1.518(4)
O5	H4A	0.87(4)
O5	H4B	0.79(4)
O4	C7	1.226(3)
O4	K2	2.844(2)
O1	Ni1	2.142(2)
N1	C6	1.338(4)
N1	C2	1.336(4)
N1	Ni1	1.967(3)
O3	C7	1.285(3)
O3	Ni1	2.107(2)
C7	C6	1.517(4)
C6	C5	1.383(4)
C2	C3	1.375(4)

Atom1	Atom2	Length
C5	H3	0.931(3)
C5	C4	1.388(4)
C4	H2	0.930(3)
C4	C3	1.401(4)
C3	H1	0.930(3)
O8	H7A	0.78(5)
O8	H7B	0.83(4)
O7	H6A	0.76(5)
O7	H6B	0.85(6)

## Conclusions

In this work, a new 3D MOFs synthesized hydrothermally by reacting alkali metal salt (KCl) and transition metal salt  $[\text{Ni}(\text{CH}_3\text{COOH})_2] \cdot 4\text{H}_2\text{O}$  together with 2,6-pydc in water for 2 days at  $170^\circ\text{C}$ . This coordination network is characterized by IR spectroscopy, thermal analysis and single crystal X-ray analysis. The 3D structure is constructed by  $[\text{NiK}_2(2,6\text{-pydc})_2(\text{H}_2\text{O})_7]_n$  unit in which water molecules also act as ligand. This coordination network is characterized by IR spectroscopy, thermal analysis and single crystal X-ray analysis. The nickel atom has a slightly elongated axially distorted octahedral coordination. In this structure, most interesting aspect is that  $\text{K}^+$  ion shows two different coordination numbers, 6 and 7. K2 is showing 7 while K1 shows 6 coordination numbers. The carboxylate group of pyridine 2, 6-dicarboxylate ligand adopts  $\mu$ -2 bridging mode, oxygen atom of carboxylic group coordinates with both two metals  $\text{Ni}^{2+}$  and  $\text{K}^+$  ions. The presence of water molecules and carboxylic groups makes extensive intermolecular Hydrogen bonding.

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