

Supplementary material

Table 1 Agreement factors, R_{wp} and Gof (goodness-of-fit) of Rietveld refinement

Samples	R_{wp} (%)	Gof
NiPC(EtOH)	3.27	1.07
NiPC(DMK)	4.92	1.61
NiPC(MeOH)	3.12	1.05
NiPC(MEG)	6.23	1.69

Table 2 Space group and Unit cell obtained with Rietveld refinement

Samples	Delta	$Ni_2P_4O_{12}$	Beta
Space Group	P21/a	C12/c	C12/m
NiPC(EtOH)	a = 5.2044(5) Å b = 9.887(1) Å c = 4.4648(3) Å Be = 97.536(6) (°)	a = 11.564(5) Å b = 8.195(6) Å c = 9.905(7) Å Be = 118.85(4) (°)	
NiPC(DMK)	a = 5.2014(2) Å b = 9.880(4) Å c = 4.461(1) Å Be = 97.55(2) (°)	a = 11.610(1) Å b = 8.229(1) Å c = 9.828(2) Å Be = 118.58(1) (°)	
NiPC(MeOH)	a = 5.2048(4) Å b = 9.8944(8) Å c = 4.4638(4) Å Be = 97.488(4) (°)		
NiPC(MEG)	a = 5.2064(2) Å b = 9.900(1) Å c = 4.4697(6) Å Be = 97.60(1) (°)	a = 11.621(1) Å b = 8.230(1) Å c = 9.845(1) Å Be = 118.58(1) (°)	a = 6.499(4) Å b = 8.372(6) Å c = 4.482(2) Å Be = 103.95(5) (°)

Table 3 Selected Ni-O-O bond angles (°) for the Ni₂P₄O₁₂ crystalline phase

Samples	Ni ₂ P ₄ O ₁₂				
	Ni-O2-O1	Ni-O3-O2	Ni-O4-O3	Ni-O5-O4	Ni-O6-O5
NiP(EtOH)	180	90(3)	180	100(30)	180.000(1)
NiP(DMK)	180	90(3)	180	99(3)	180
NiP(MEG)	180	88(3)	180.0000(1)	103(4)	180.0000(2)

Table 4 Selected P-O-O bond angles (°) for the Ni₂P₄O₁₂ crystalline phase

Samples	Ni ₂ P ₄ O ₁₂			
	P-O1-O2	P-O3-O1	P-O4-O3	P-O5-O4
NiPC(EtOH)	120(6)	110(8)	110(5)	160(6)
NiPC(DMK)	112(4)	109(6)	110(5)	151(5)
NiPC(MEG)	113(5)	110(6)	109(4)	156(5)

Table 5 Selected P-O bond lengths (Å) for the Ni₂P₄O₁₂ crystalline phase

Samples	Ni ₂ P ₄ O ₁₂				
	P-O1	P-O2	P-O3	P-O4	P-O5
NiPC(EtOH)	1.5(2)	1.5(13)	1.6(10)	1.6(1)	3.1(1)
NiPC(DMK)	1.48(8)	1.55(9)	1.51(1)	1.49(1)	3.20(7)
NiPC(MEG)	1.56(1)	1.49(1)	1.54(1)	1.57(1)	3.19(1)

Table 6 Selected Ni-O bond lengths (Å) for the Ni₂P₄O₁₂ crystalline phase

Samples	Ni ₂ P ₄ O ₁₂					
	Ni-O1	Ni-O2	Ni-O3	Ni-O4	Ni-O5	Ni-O6
NiP(EtOH)	2.0(8)	2.0(8)	2.1(6)	2.1(6)	2.2(1)	2.2(1)
NiP(DMK)	2.00(1)	2.00(1)	2.11(6)	2.11(6)	2.18(1)	2.18(1)
NiP(MEG)	2.01(9)	2.01(9)	2.16(1)	2.16(1)	2.13(9)	2.13(9)

Table 7 Selected Ni-O-O bond angles (°) for the δ-Ni₂P₂O₇ crystalline phase

Samples	δ-Ni ₂ P ₂ O ₇				
	Ni-O1-O2	Ni-O4-O1	Ni-O3-O4	Ni-O5-O3	Ni-O6-O5
NiP(EtOH)	81(2)	157(2)	104(2)	77.1(9)	113.1(17)
NiP(DMK)	81(5)	157(5)	106(5)	77(2)	113(4)
NiP(MeOH)	80.5(2)	156.0(2)	106.1(1)	77.1(7)	112.1(1)
NiP(MEG)	79(2)	151.7(2)	107(2)	77.6(7)	112(2)

Table 8 Selected P-O-O bond angles (°) for the δ-Ni₂P₂O₇ crystalline phase

Samples	δ-Ni ₂ P ₂ O ₇				
	P-O3-O1	P-O2-O3	P-O4-O2	P-O6-O4	P-O5-O6
NiP(EtOH)	111(3)	110(3)	103(3)	179.2(18)	98.1(2)
NiP(DMK)	111(8)	112(8)	103(6)	179(5)	98(3)
NiP(MeOH)	110(3)	110(2)	105(2)	178.7(2)	98.4(2)
NiP(MEG)	114(3)	112(3)	105(2)	178(2)	99.5(1)

Table 9 Selected P-O bond angles (Å) for the δ -Ni₂P₂O₇ crystalline phase

Samples	δ -Ni ₂ P ₂ O ₇					
	P-O1	P-O2	P-O3	P-O4	P-O5	P-O6
NiP(EtOH)	1.49(6)	1.53(7)	1.50(3)	1.55(2)	3.08(6)	2.89(7)
NiP(DMK)	1.50(1)	1.54(1)	1.49(9)	1.57(8)	3.11(1)	2.86(1)
NiP(MeOH)	1.50(5)	1.53(5)	1.53(3)	1.54(2)	3.06(5)	2.90(5)
NiP(MEG)	1.54(7)	1.52(6)	1.53(3)	1.56(3)	3.04(7)	2.85(5)

Table 10 Selected Ni-O bond angles (Å) for the δ -Ni₂P₂O₇ crystalline phase

Samples	δ -Ni ₂ P ₂ O ₇					
	Ni-O1	Ni-O2	Ni-O3	Ni-O4	Ni-O5	Ni-O6
NiP(EtOH)	2.03(6)	2.05(6)	2.15(2)	2.12(7)	2.15(4)	2.16(6)
NiP(DMK)	2.02(1)	2.02(1)	2.13(7)	2.10(1)	2.15(1)	2.17(1)
NiP(MeOH)	2.04(5)	2.02(4)	2.14(2)	2.10(5)	2.14(3)	2.18(5)
NiP(MEG)	2.05(7)	2.08(5)	2.17(3)	2.14(5)	1.99(3)	2.18(7)

Table 11 Selected Ni-O bond angles (°) for the β -Ni₂P₂O₇ crystalline phase

Samples	β -Ni ₂ P ₂ O ₇				
	Ni-O2-O1	Ni-O3-O2	Ni-O4-O3	Ni-O5-O4	Ni-O6-O5
NiP(MEG)	80(1)	97(7)	96(9)	114(6)	168(9)
	P-O2-O1	P-O3-O2	P-O4-O3	P-O5-O4	
NiP(MEG)	109(6)	111(1)	110(7)	69(1)	

Table 12 Selected Ni-O bond lengths (Å) for the β -Ni₂P₂O₇ crystalline phase

Samples	β -Ni ₂ P ₂ O ₇					
	Ni-O1	Ni-O2	Ni-O3	Ni-O4	Ni-O5	Ni-O6
NiP(MEG)	1.97(2)	1.97(2)	2.11(1)	2.11(15)	2.24(2)	2.24(2)
	P-O1	P-O2	P-O3	P-O4	P-O5	
NiP(MEG)	1.53(8)	1.53(2)	1.53(2)	1.6(3)	2.7(3)	