APPENDIX E

X-Ray Diffraction

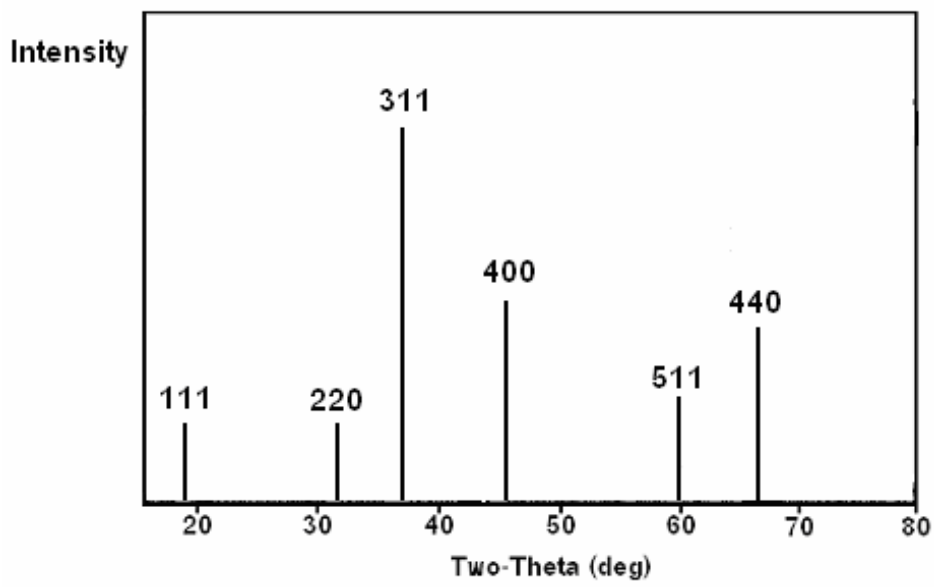


Figure E1 Major peak and *hkl* reflection of reference $NiAl_2O_4$ spinel (JCPDS file No. 10-0339).

Note: Two-theta (deg), interplanar spacing (d), intensity (%I) and *hkl* reflection data of reference $NiAl_2O_4$ spinel are listed in the following information (JCPDS file No. 10-0339):

Two-theta (deg)	Interplanar spacing (nm)	Intensity (%I)	*hkl* reflection
19.037	0.46579	20.0	(111)
31.361	0.28501	20.0	(220)
37.062	0.24237	100.0	(311)
45.080	0.20094	65.0	(400)
59.719	0.15471	30.0	(511)
65.660	0.14208	60.0	(440)

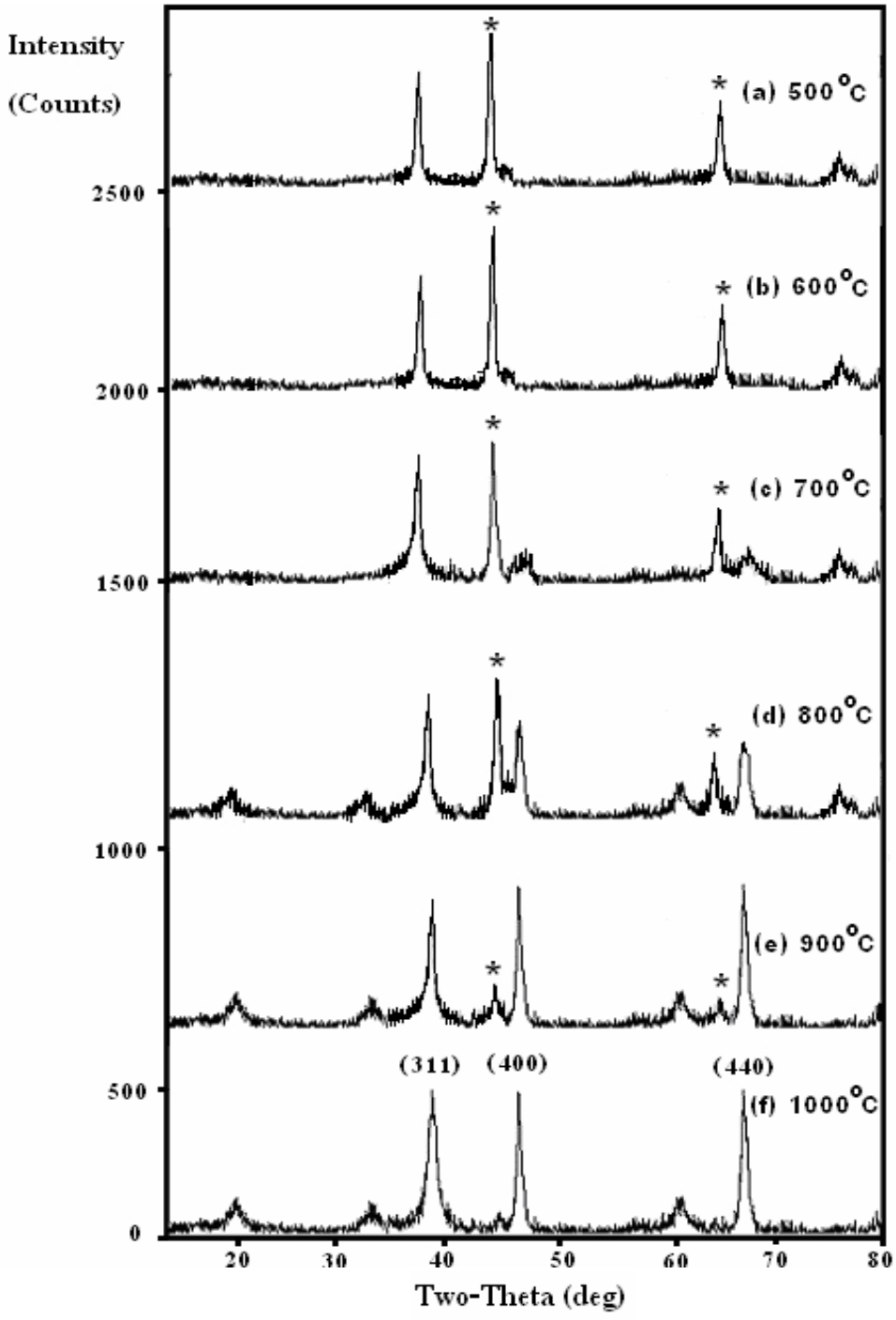


Figure E2 XRD patterns of spinel precursor (SPNO) at increasing calcination temperature (a) 500°C, (b) 600°C, (c) 700°C, (d) 800°C, (e) 900°C (f) 1000°C

*: NiO

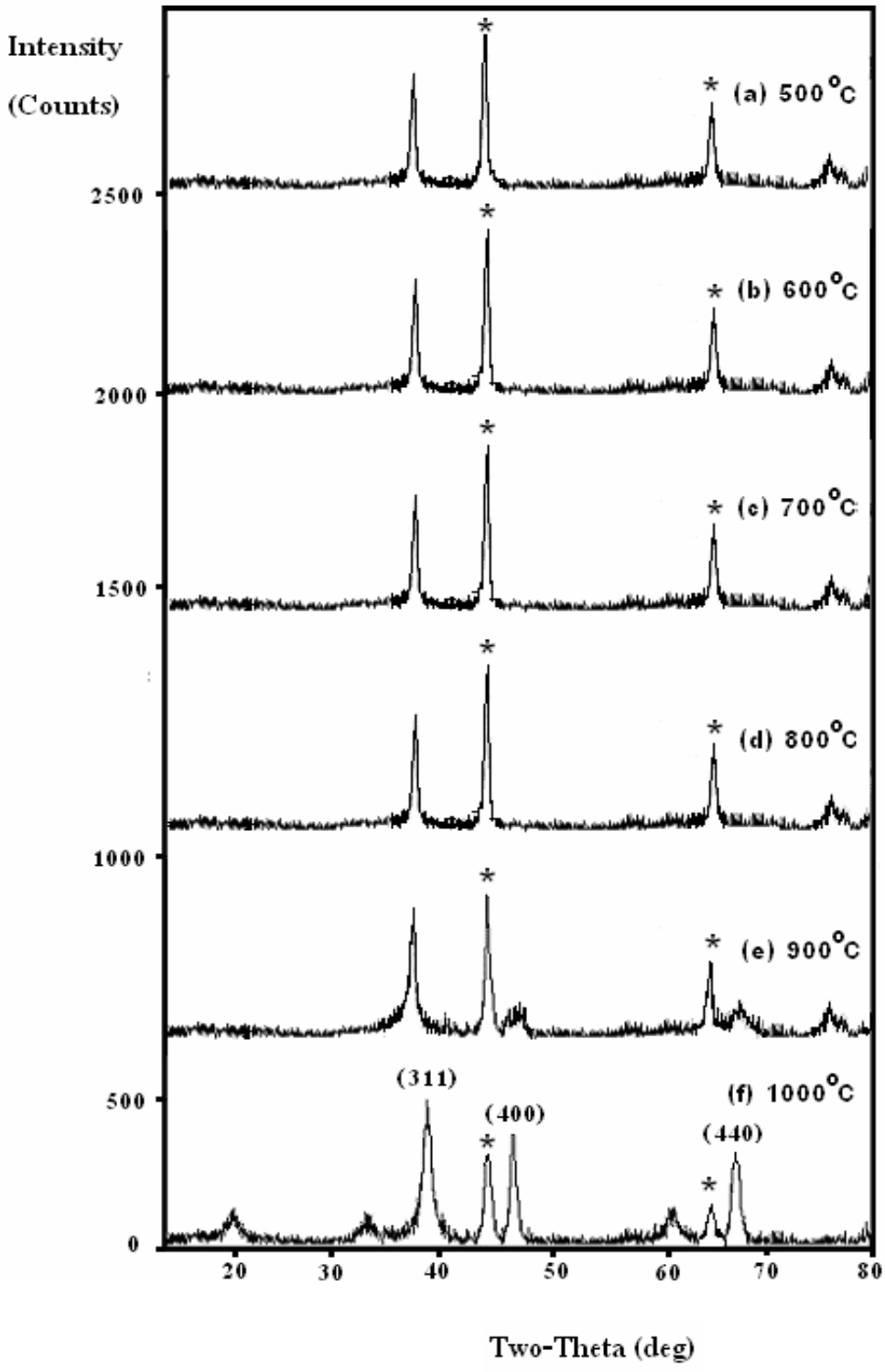


Figure E3 XRD patterns of spinel precursor (SPCl) at increasing calcination temperature (a) 500°C, (b) 600°C, (c) 700°C, (d) 800°C, (e) 900°C (f) 1000°C *: NiO

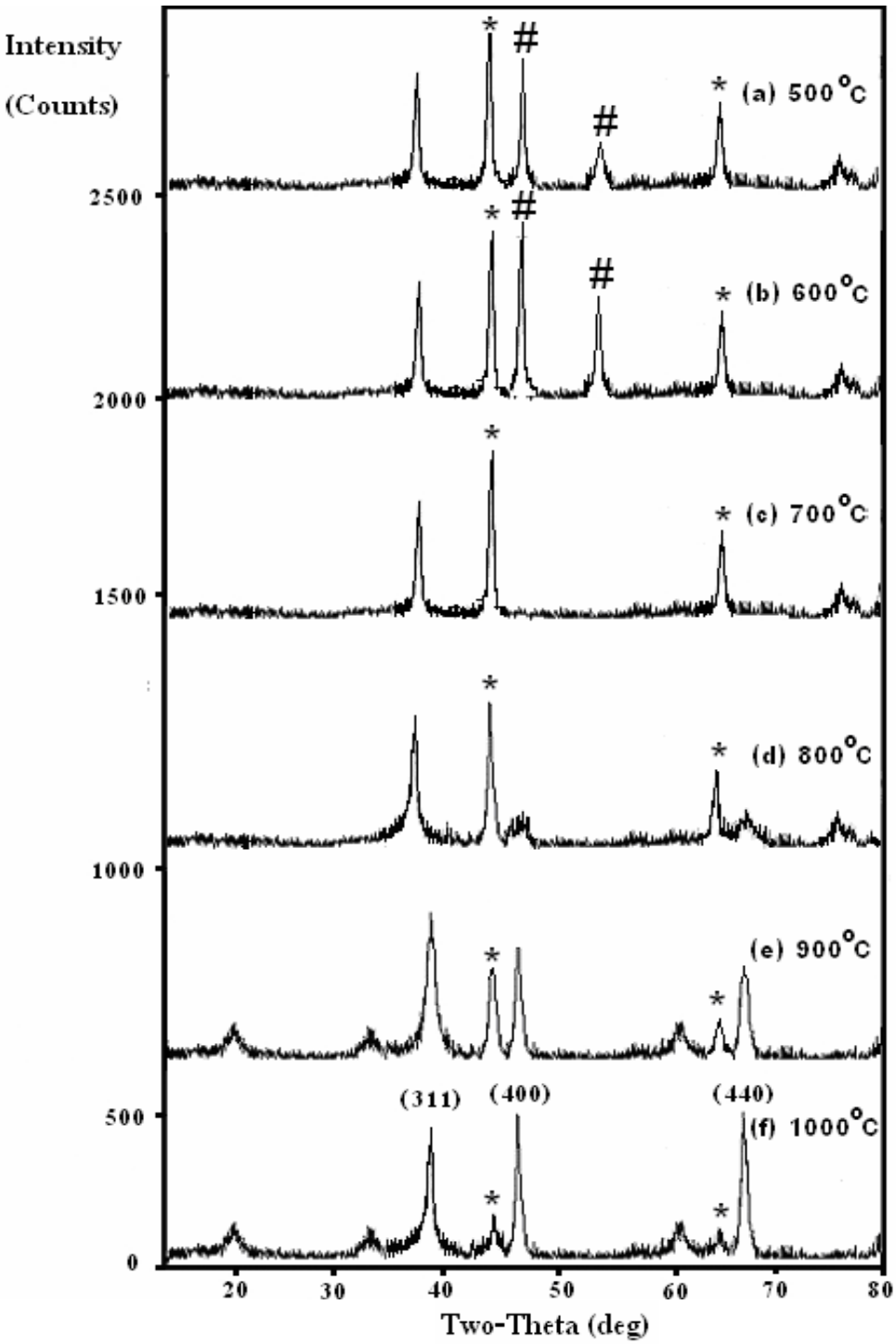


Figure E4 XRD patterns of spinel precursor (SPOH) at increasing calcination temperature (a) 500°C, (b) 600°C, (c) 700°C, (d) 800°C, (e) 900°C (f) 1000°C *: NiO, #: Ni.

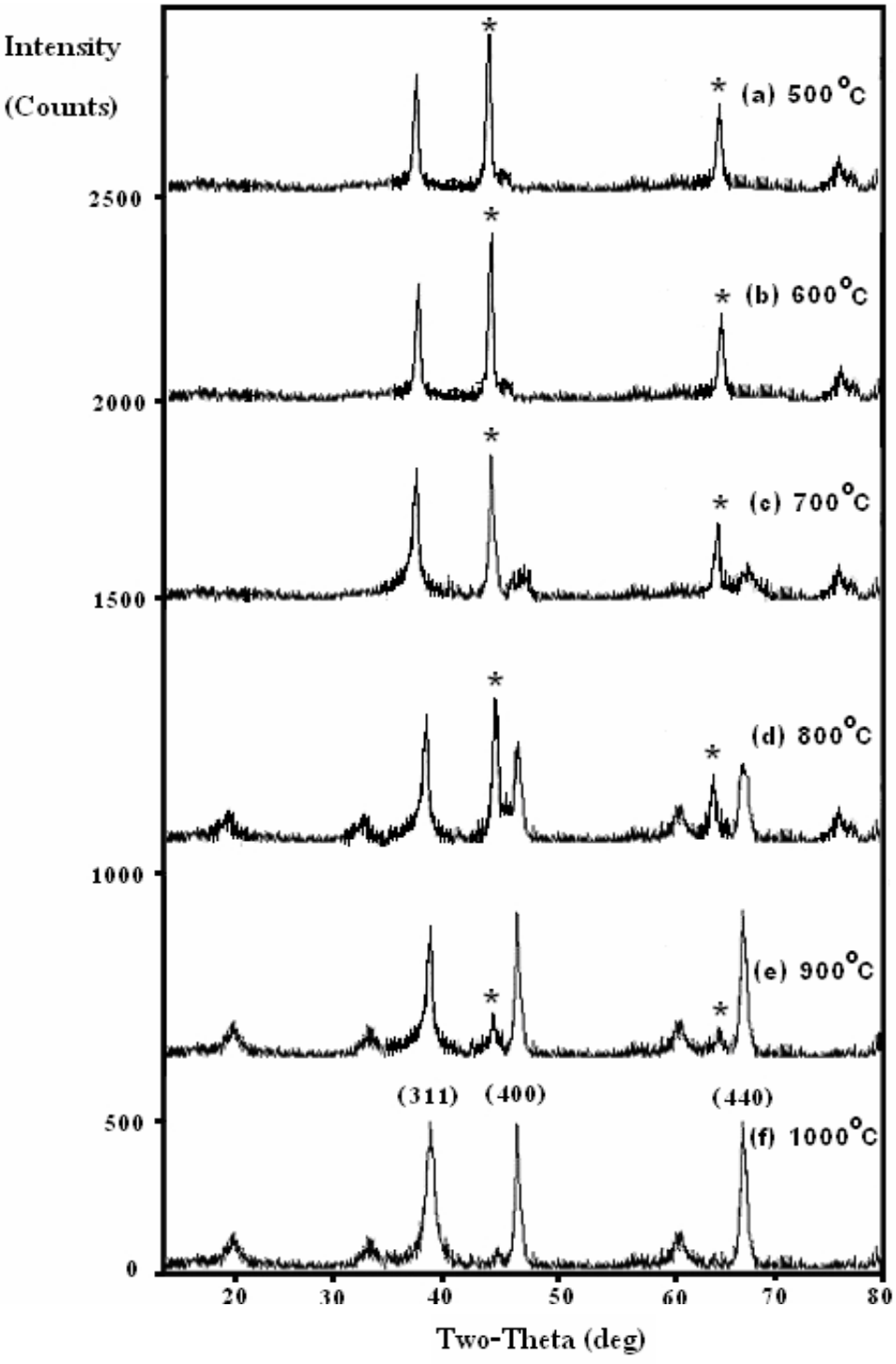


Figure E5 XRD patterns of spinel precursor (SPAc) at increasing calcination temperature (a) 500°C, (b) 600°C, (c) 700°C, (d) 800°C, (e) 900°C (f) 1000°C *: NiO

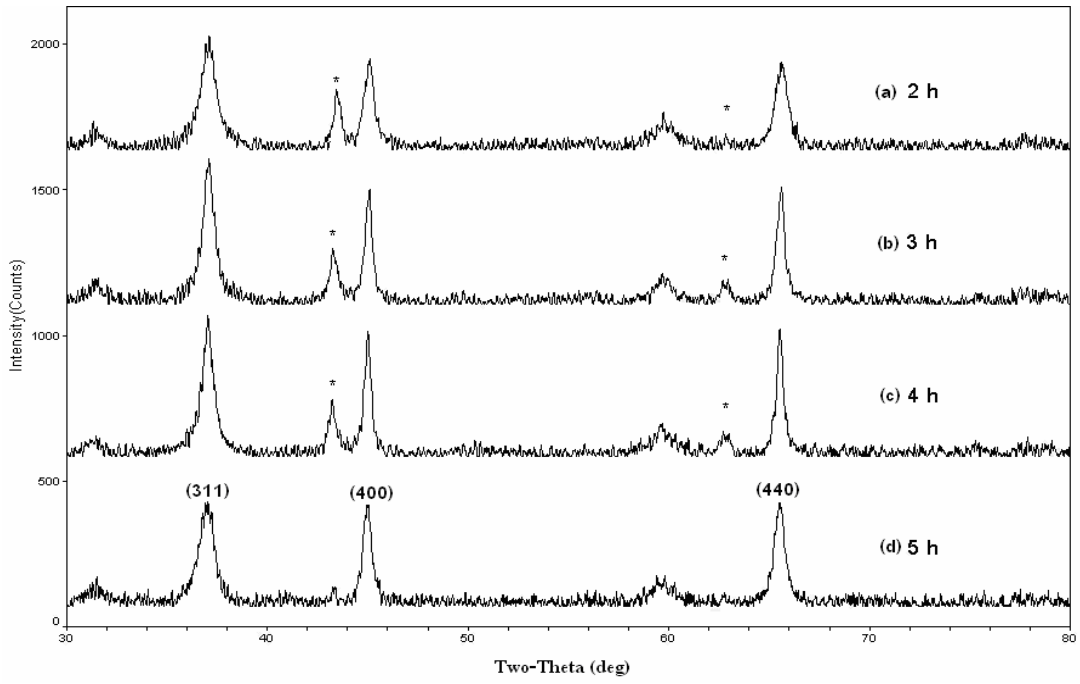


Figure E6 XRD spectra of products calcined (SPNO) at different time

(a) 2 h, (b) 3 h, (c) 4 h, (d) 5 h *: NiO

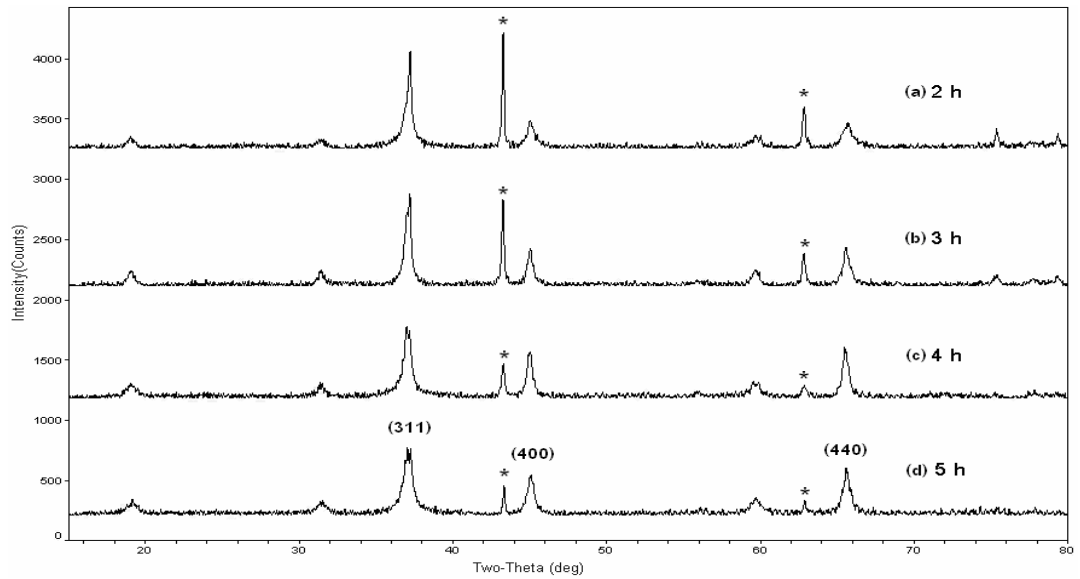


Figure E7 XRD spectra of products calcined (SPCl) at different time

(a) 2 h, (b) 3 h, (c) 4 h, (d) 5 h *: NiO

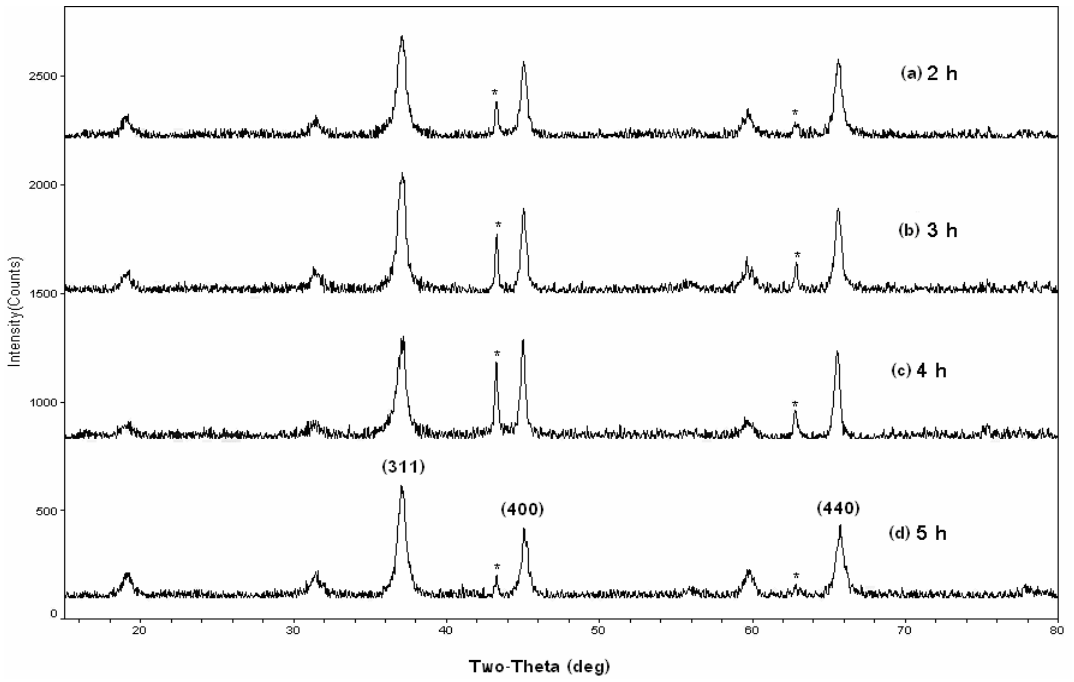


Figure E8 XRD spectra of products calcined (SPOH) at different time

(a) 2 h, (b) 3 h, (c) 4 h, (d) 5 h *: NiO

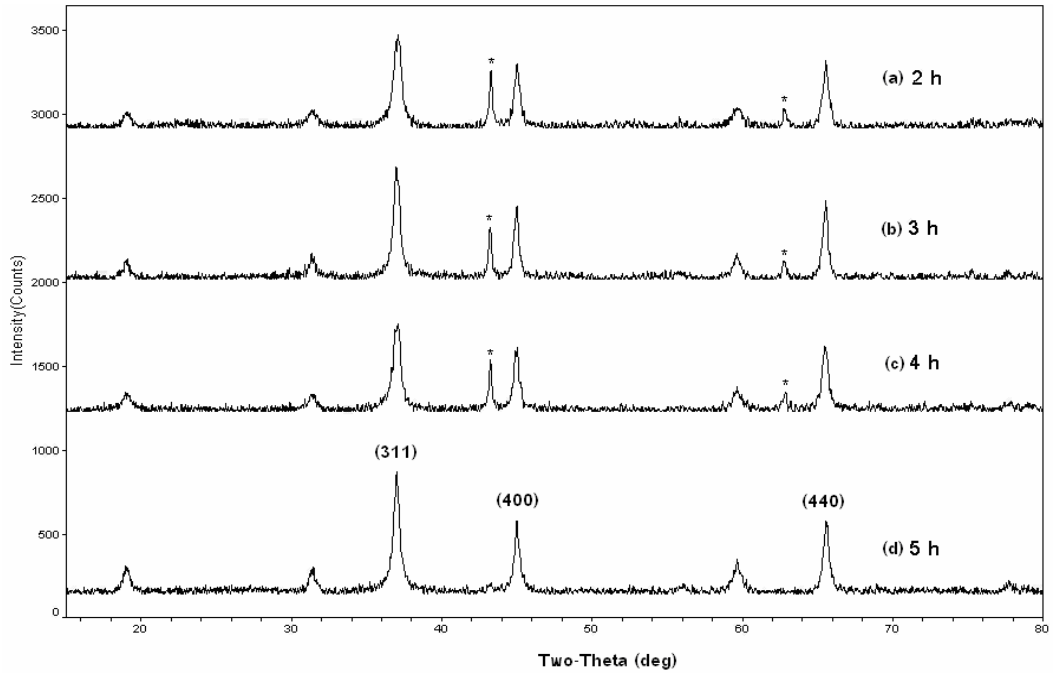


Figure E9 XRD spectra of products calcined (SPAc) at different time

(a) 2 h, (b) 3 h, (c) 4 h, (d) 5 h *: NiO

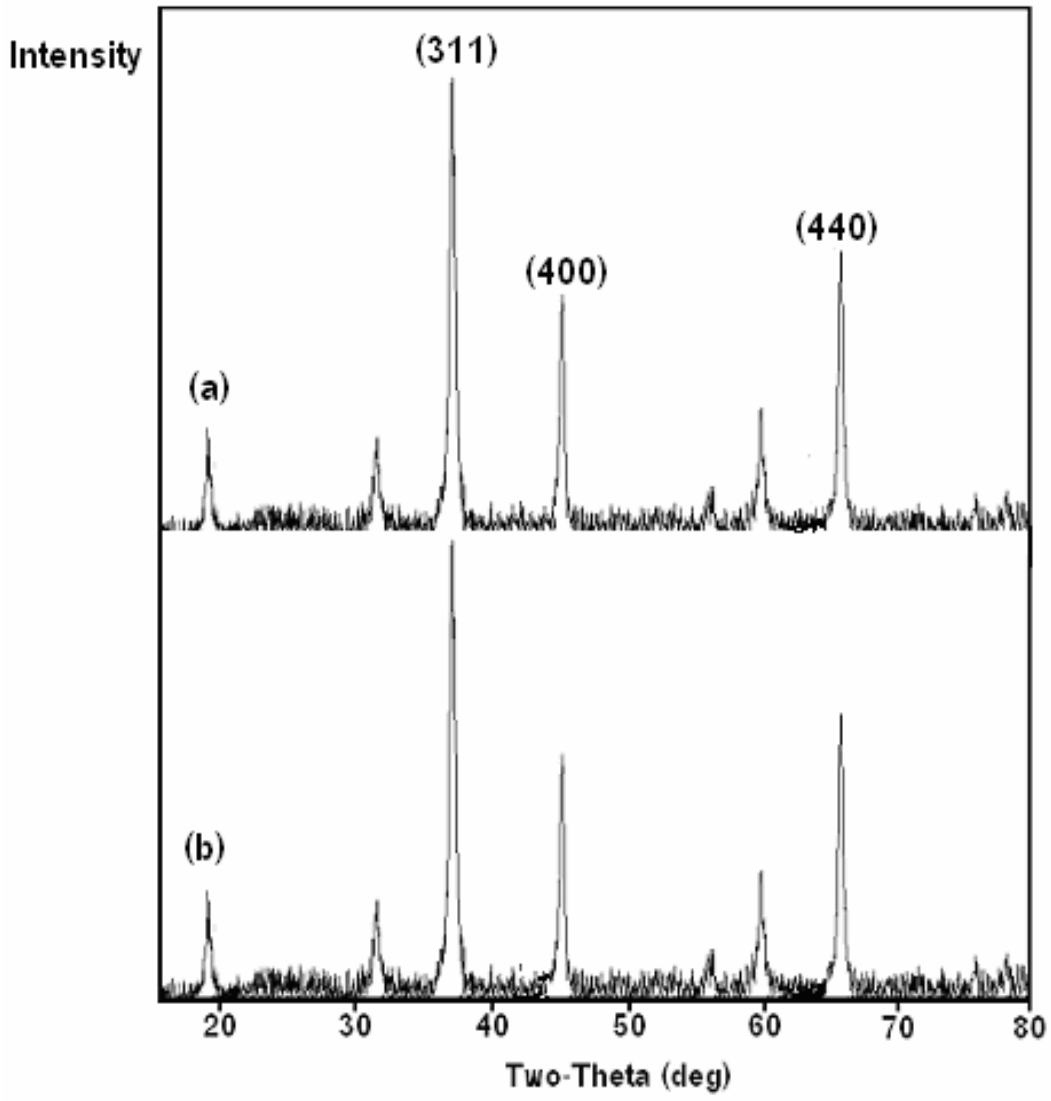


Figure E10 (a) Powder diffraction patterns of the SPNO pyrolyzed at 1000°C for 5 h.
(b) Powder diffraction patterns of the SPNO pyrolyzed at 500°C for 5 h
and held at 1000°C for 5 h.

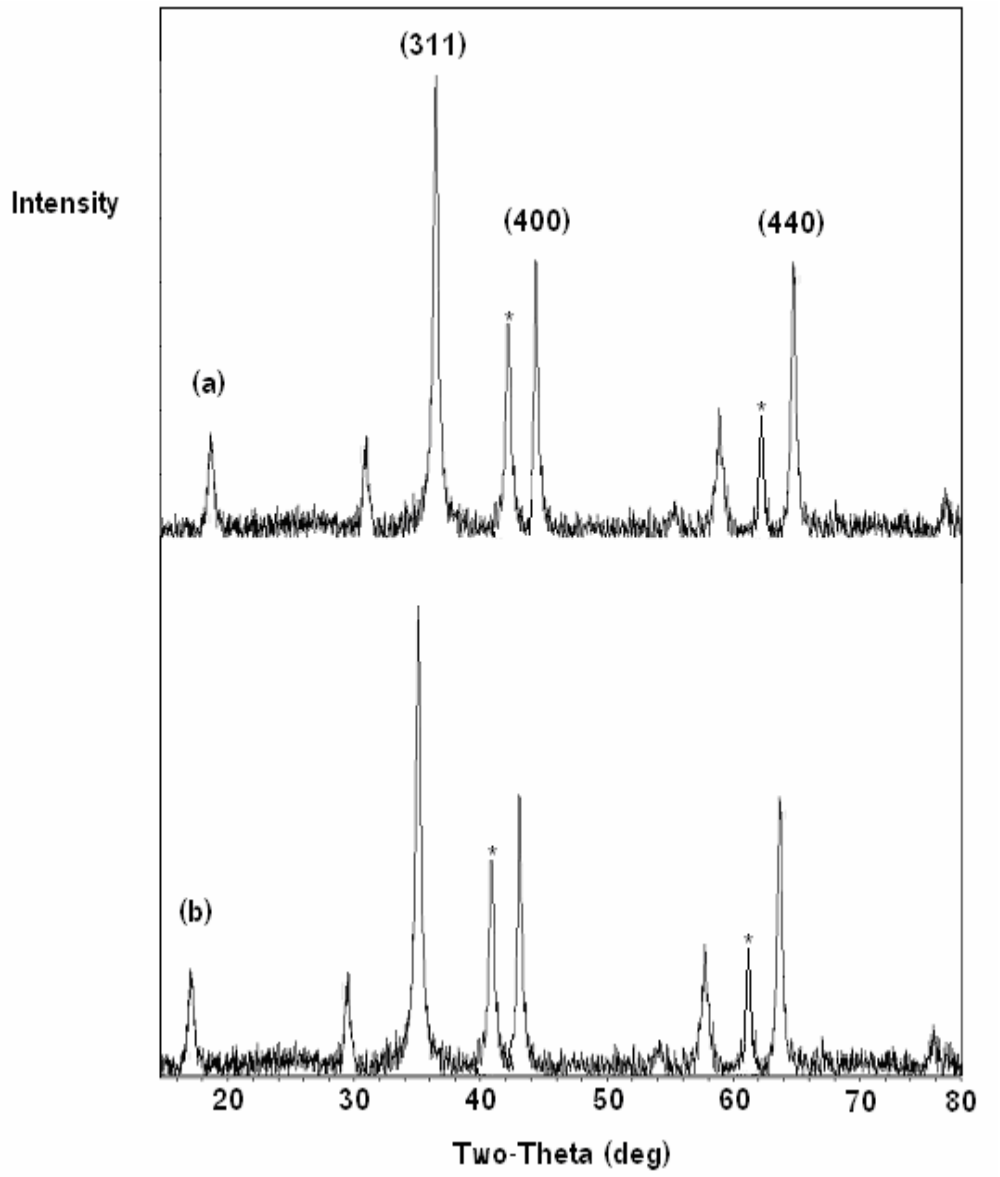


Figure E11 (a) Powder diffraction patterns of the SPCl pyrolyzed at 1000°C for 5 h.
(b) Powder diffraction patterns of the SPCl pyrolyzed at 500°C for 5 h
and held at 1000°C for 5 h *: NiO.

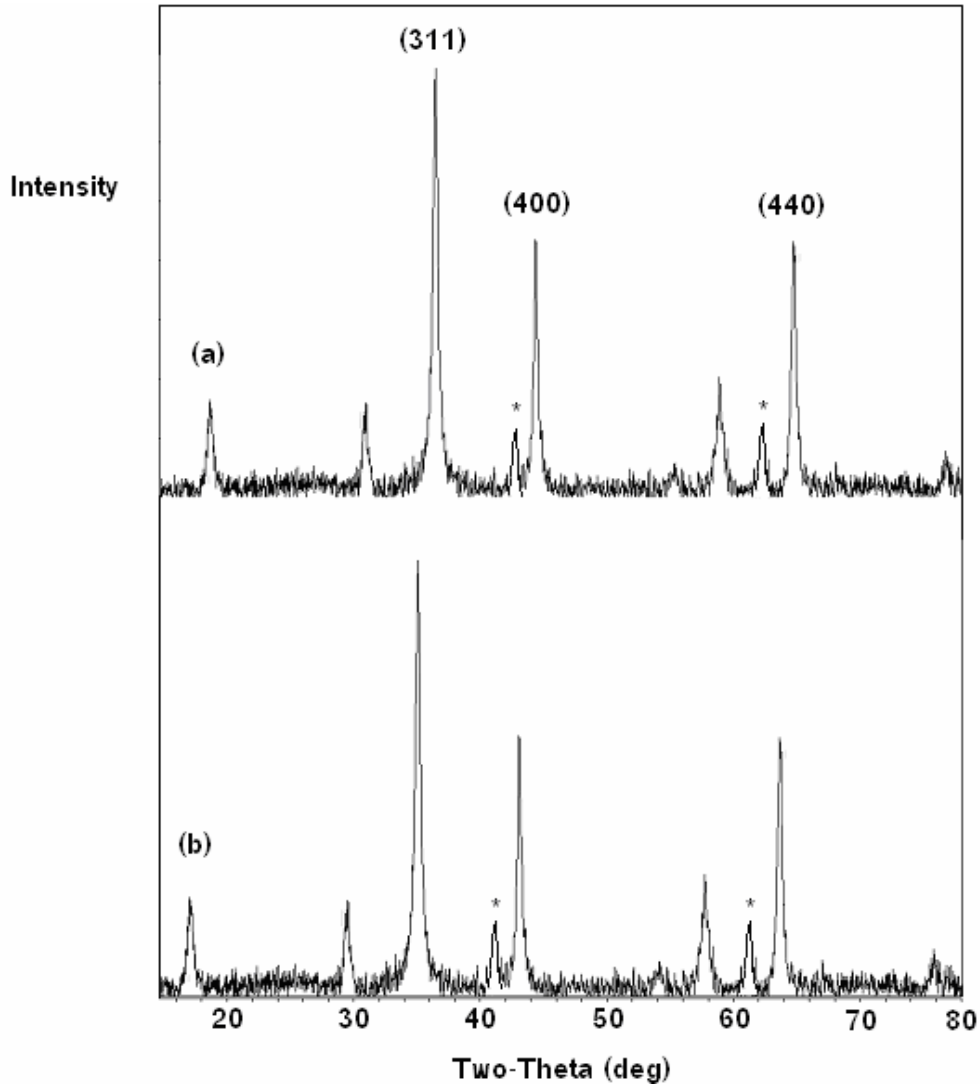


Figure E12 (a) Powder diffraction patterns of the SPOH pyrolyzed at 1000°C for 5 h. (b) Powder diffraction patterns of the SPOH pyrolyzed at 500°C for 5 h and held at 1000°C for 5 h *: NiO.

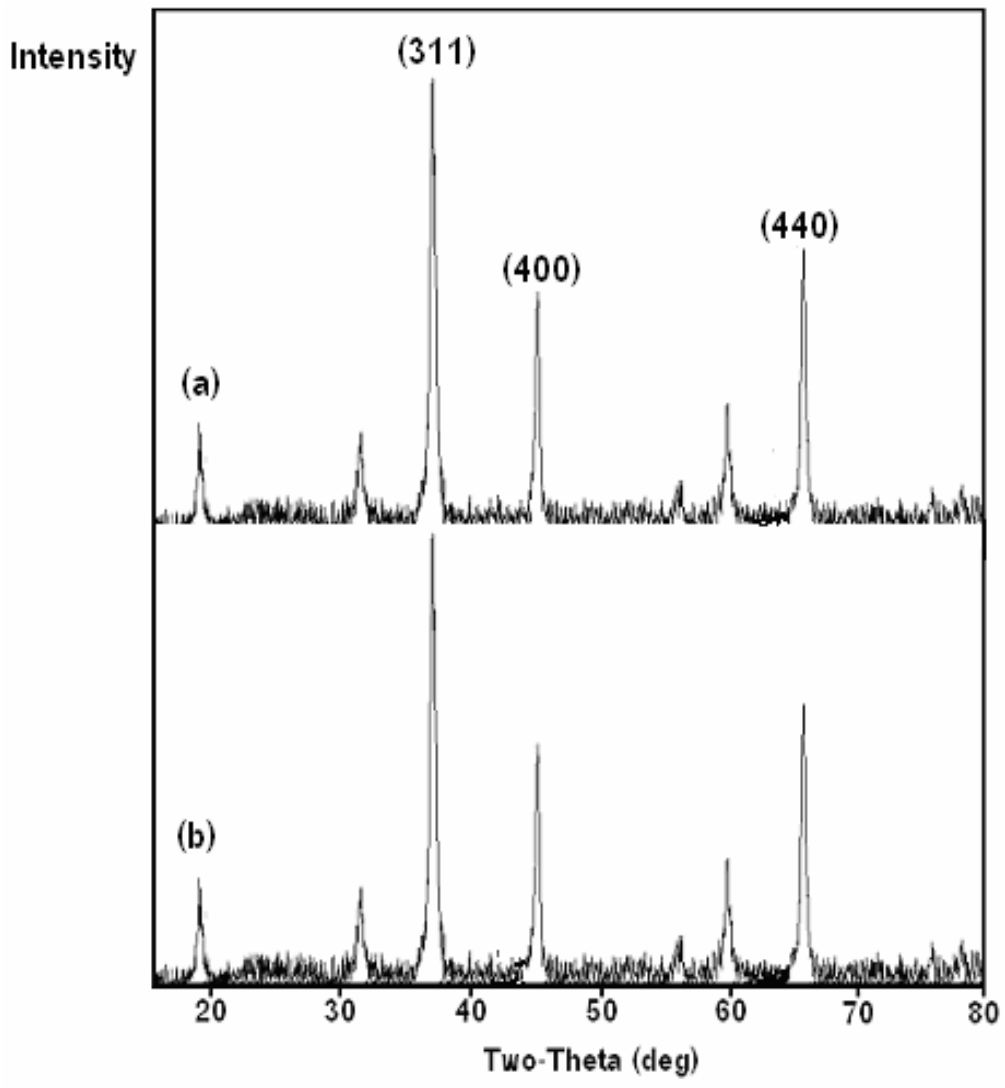


Figure E13 (a) Powder diffraction patterns of the SPAc pyrolyzed at 1000°C for 5 h.
(b) Powder diffraction patterns of the SPAc pyrolyzed at 500°C for 5 h
and held at 1000°C for 5 h.

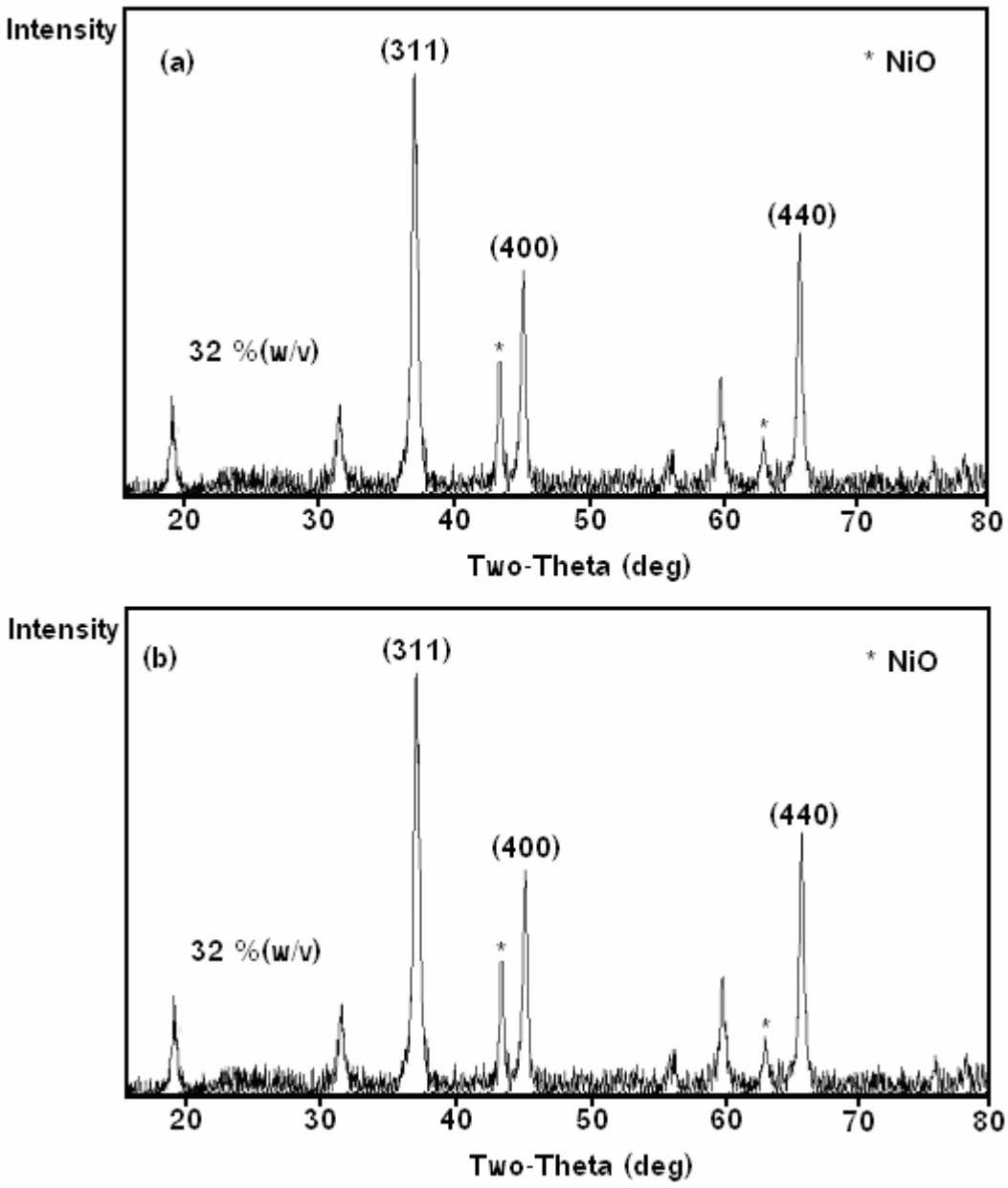


Figure E14 X-ray diffraction pattern of the powder obtained from pyrolysis of gel occurring at room temperature in the ethanolic solution of SPNO precursor, pH 6.0 with various calcination conditions: (a) heated at 1000 °C for 5 h and (b) heated at 500 °C for 5 and held at 1000 °C for 5 h.

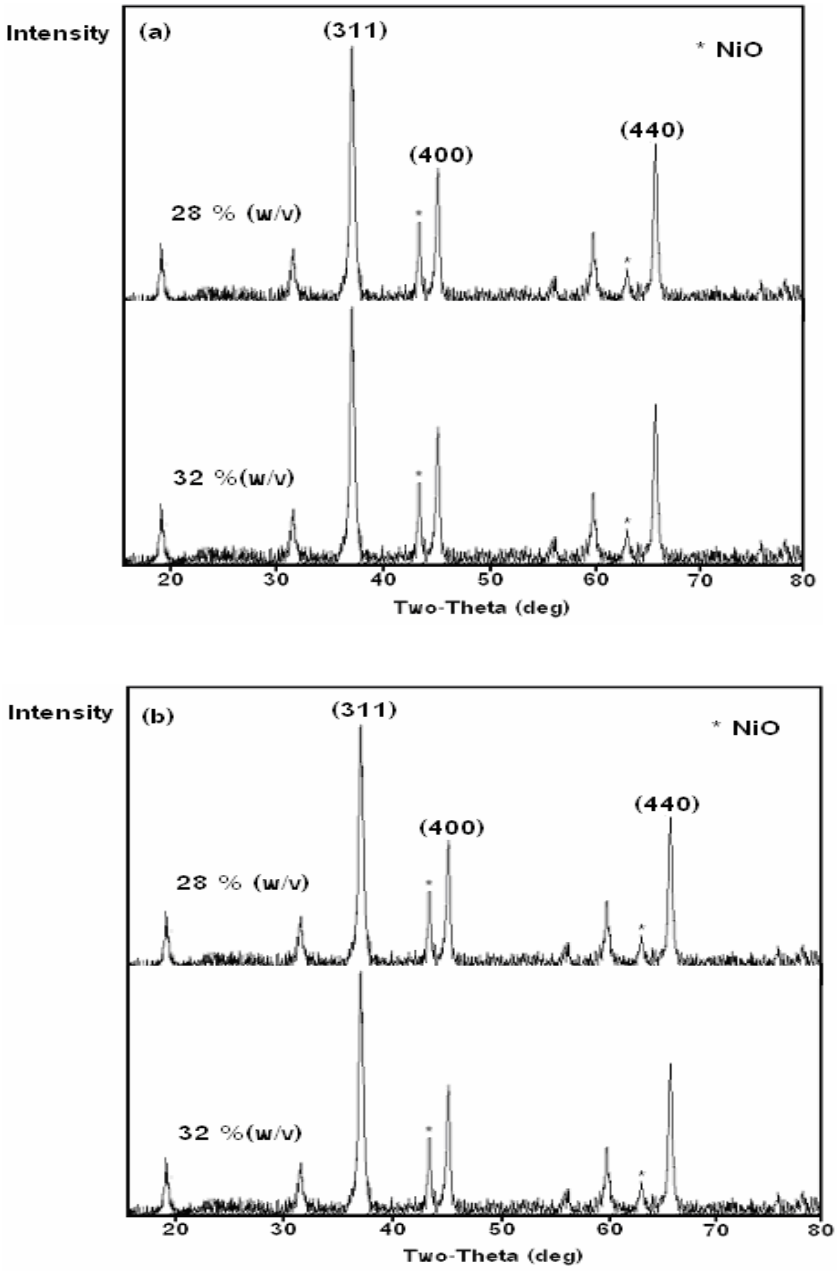


Figure E15 X-ray diffraction pattern of the powder obtained from pyrolysis of gel occurring at room temperature in the ethanolic solution of SPNO precursor, pH 7.0 with various calcination conditions: (a) heated at 1000 °C for 5 h and (b) heated at 500 °C for 5 and held at 1000 °C for 5 h.

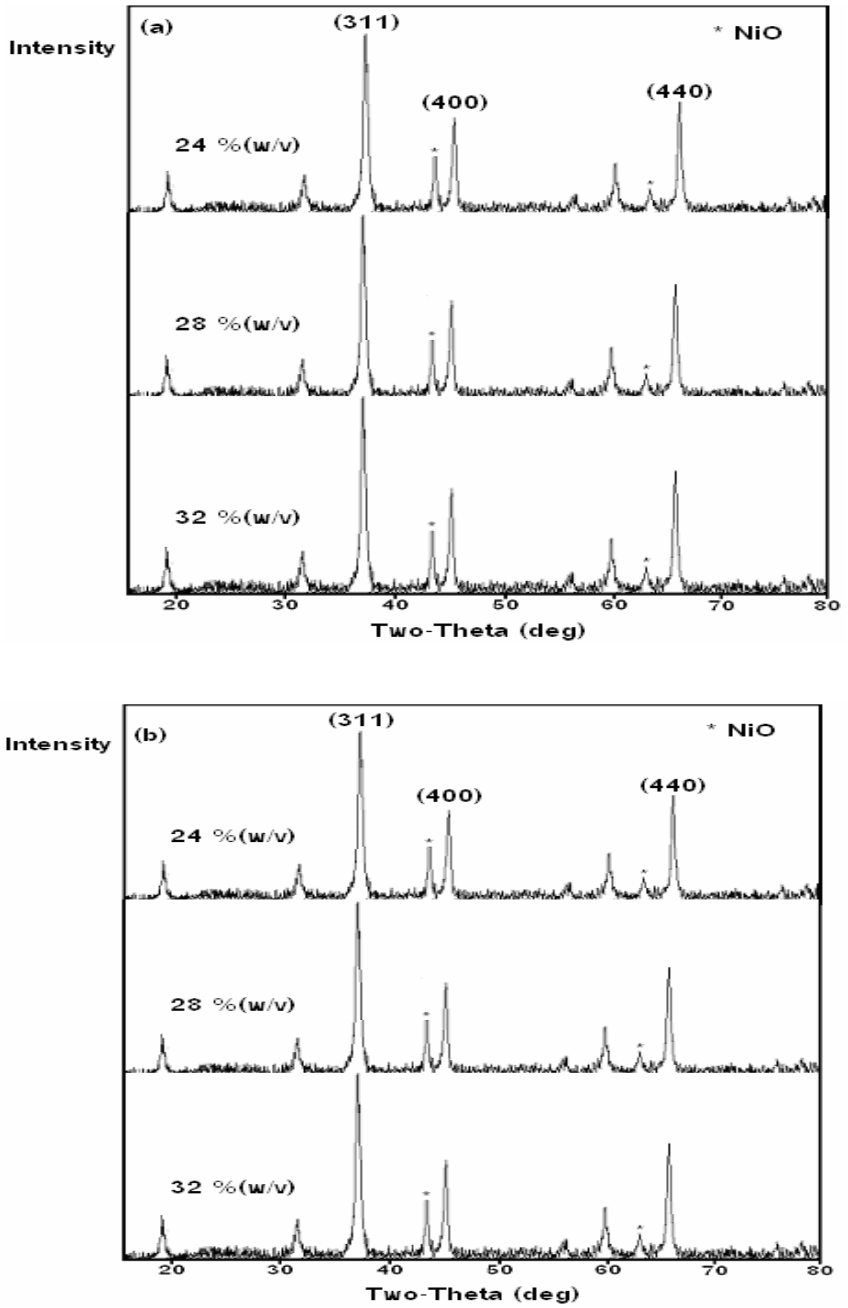


Figure E16 X-ray diffraction pattern of the powder obtained from pyrolysis of gel occurring at room temperature in the ethanolic solution of SPNO precursor, pH 8.0 with various calcination conditions: (a) heated at 1000 °C for 5 h and (b) heated at 500 °C for 5 and held at 1000 °C for 5 h.

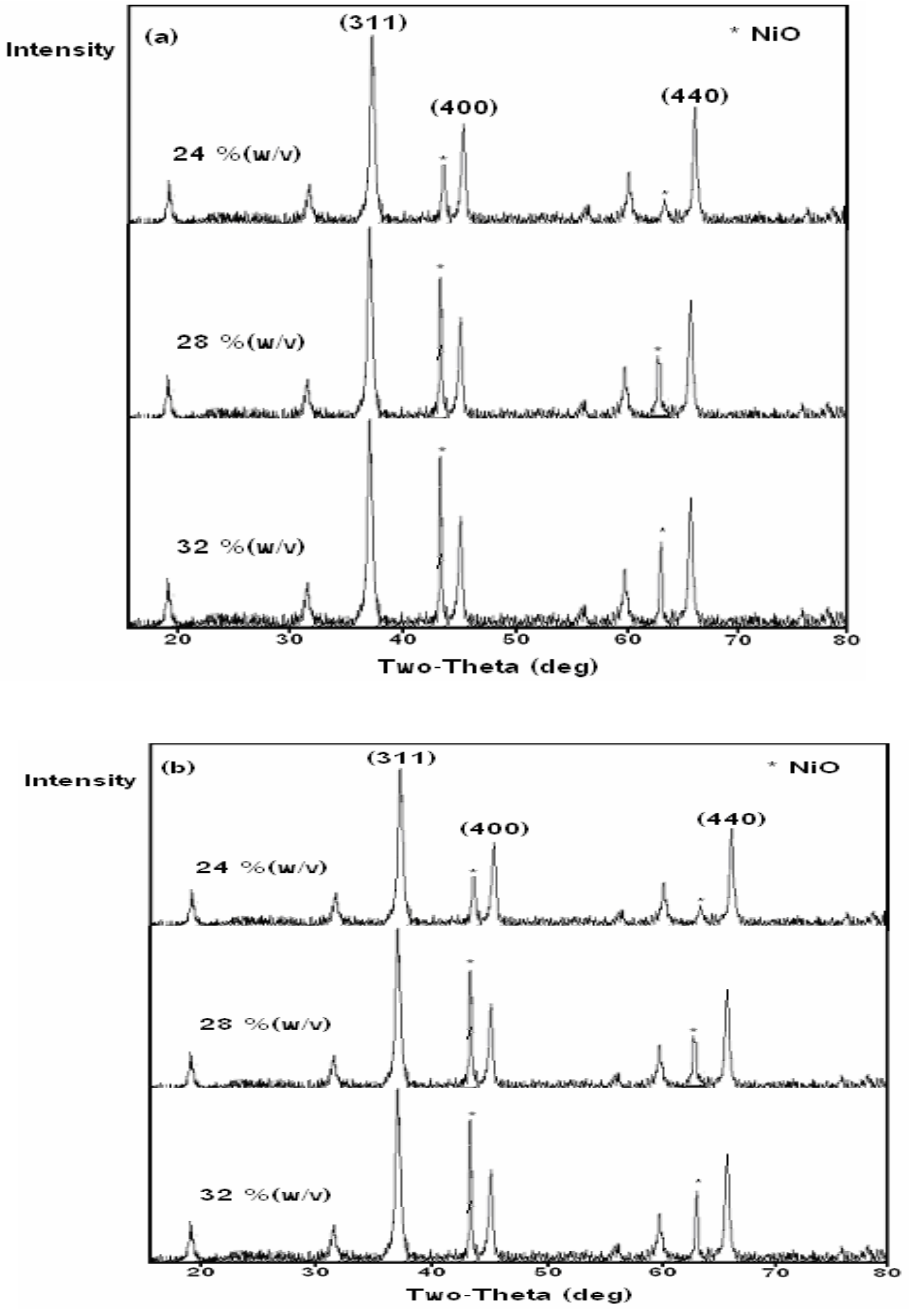


Figure E17 X-ray diffraction pattern of the powder obtained from pyrolysis of gel occurring at room temperature in the ethanolic solution of SPNO precursor, pH 9.0 with various calcination conditions: (a) heated at 1000 °C for 5 h and (b) heated at 500 °C for 5 and held at 1000 °C for 5 h.

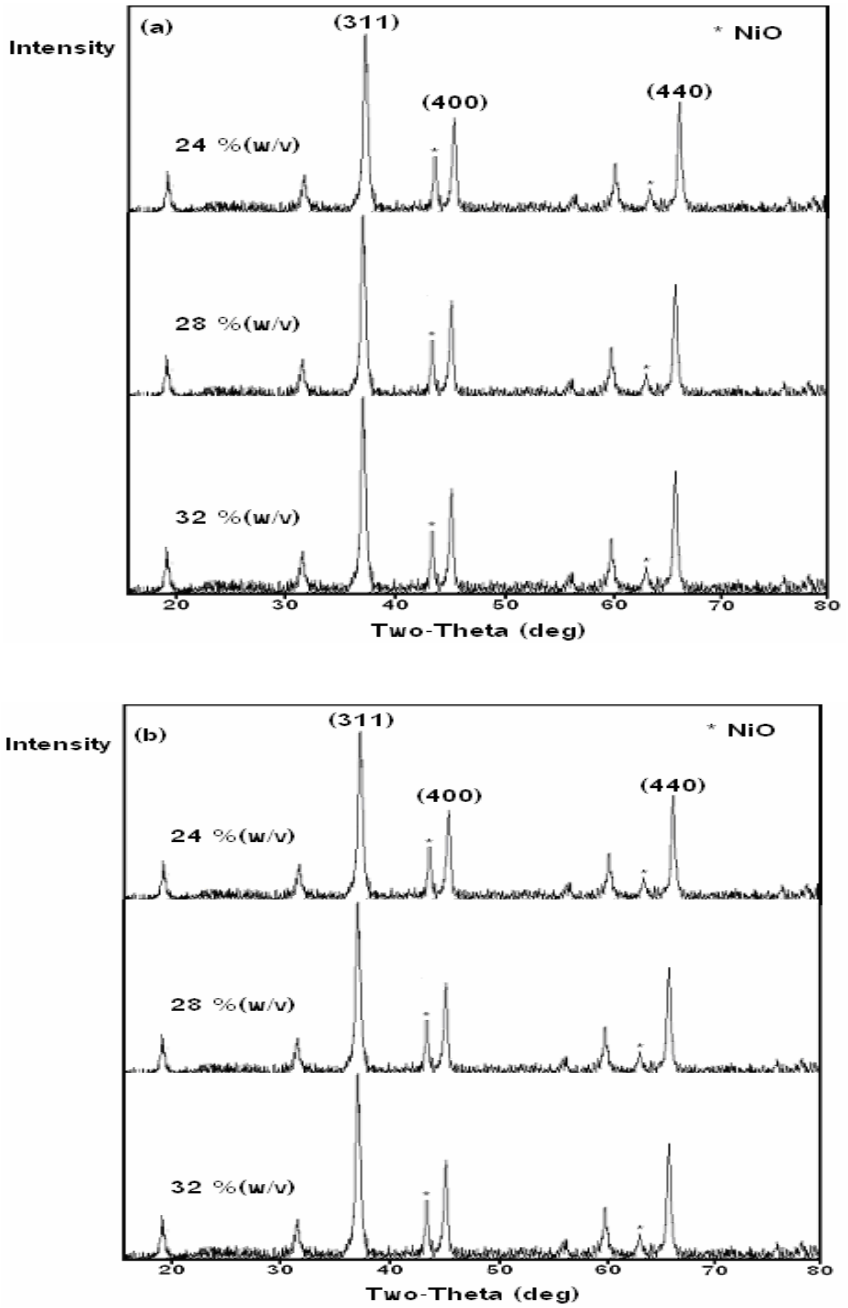
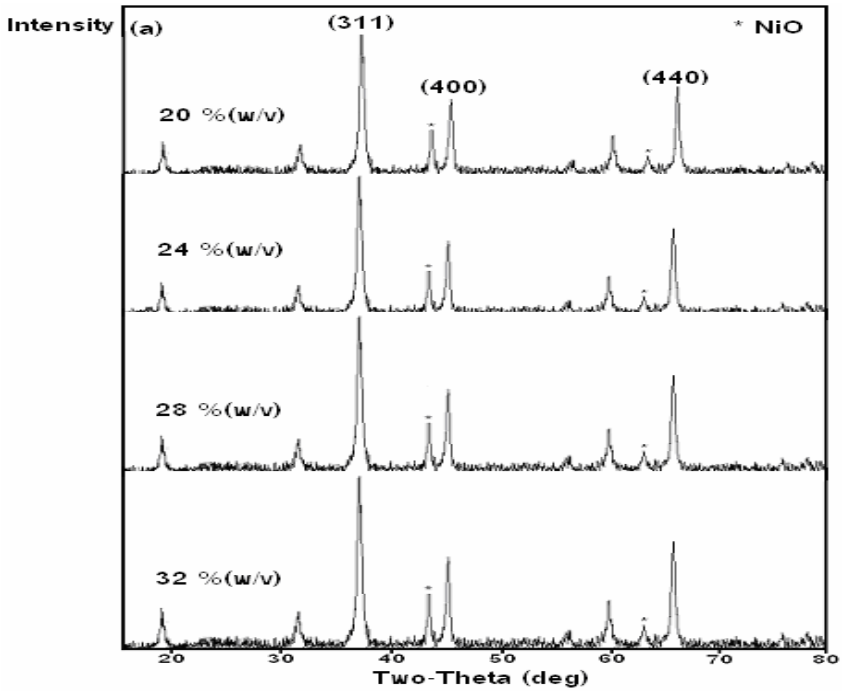


Figure E18 X-ray diffraction pattern of the powder obtained from pyrolysis of gel occurring at 60 °C in the ethanolic solution of SPNO precursor, pH 9.0 with various calcination conditions: (a) heated at 1000 °C for 5 h and (b) heated at 500 °C for 5 and held at 1000 °C for 5 h.



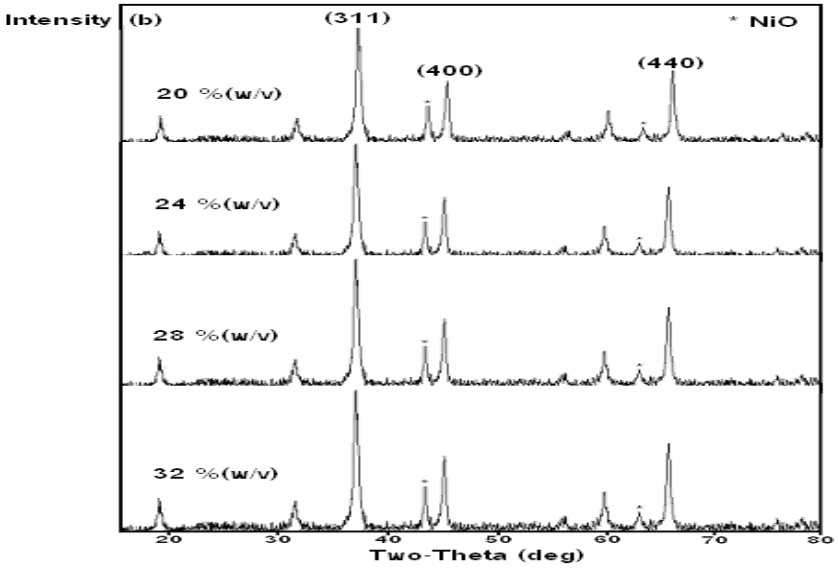


Figure E19 X-ray diffraction pattern of the powder obtained from pyrolysis of gel occurring at room temperature in the ethanolic solution of SPNO precursor, pH 10.0 with various calcination conditions: (a) heated at 1000 °C for 5 h and (b) heated at 500 °C for 5 and held at 1000 °C for 5 h.

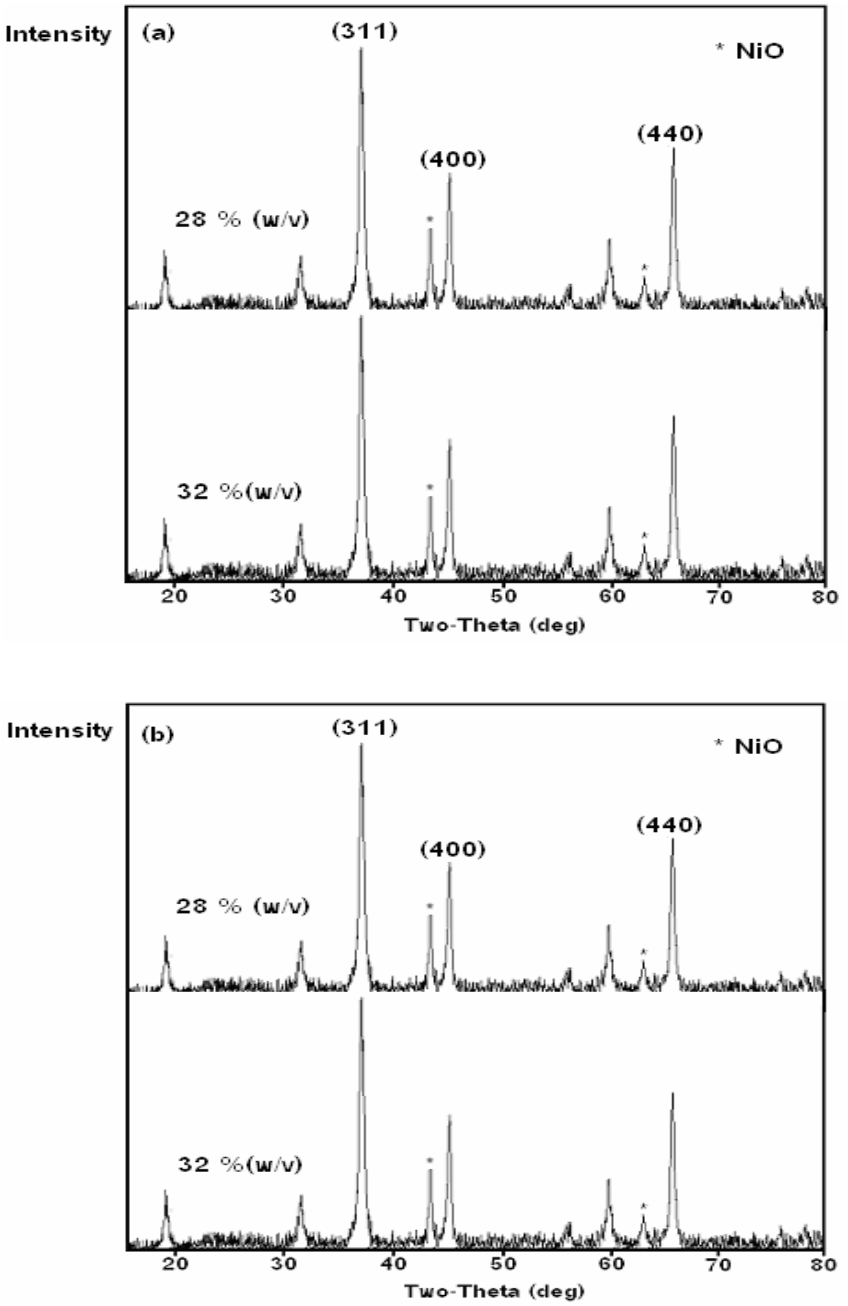


Figure E20 X-ray diffraction pattern of the powder obtained from pyrolysis of gel occurring at room temperature in the n-propanolic solution of SPNO precursor, pH 9.0 with various calcination conditions: (a) heated at 1000 °C for 5 h and (b) heated at 500 °C for 5 and held at 1000 °C for 5 h.

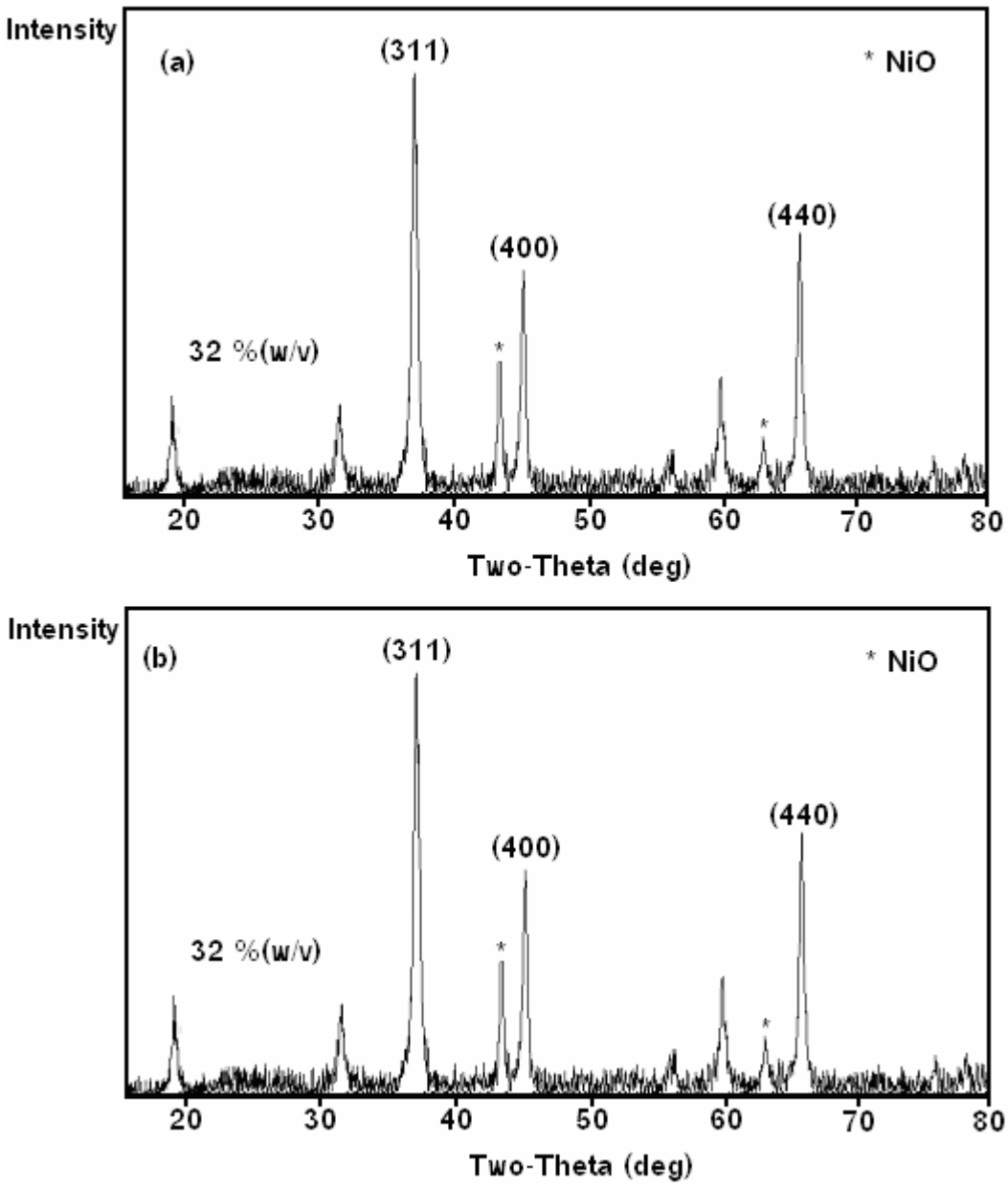


Figure E21 X-ray diffraction pattern of the powder obtained from pyrolysis of gel occurring at 60 °C in the n-propanolic solution of SPNO precursor, pH 9.0 with various calcination conditions: (a) heated at 1000 °C for 5 h and (b) heated at 500 °C for 5 and held at 1000 °C for 5 h.

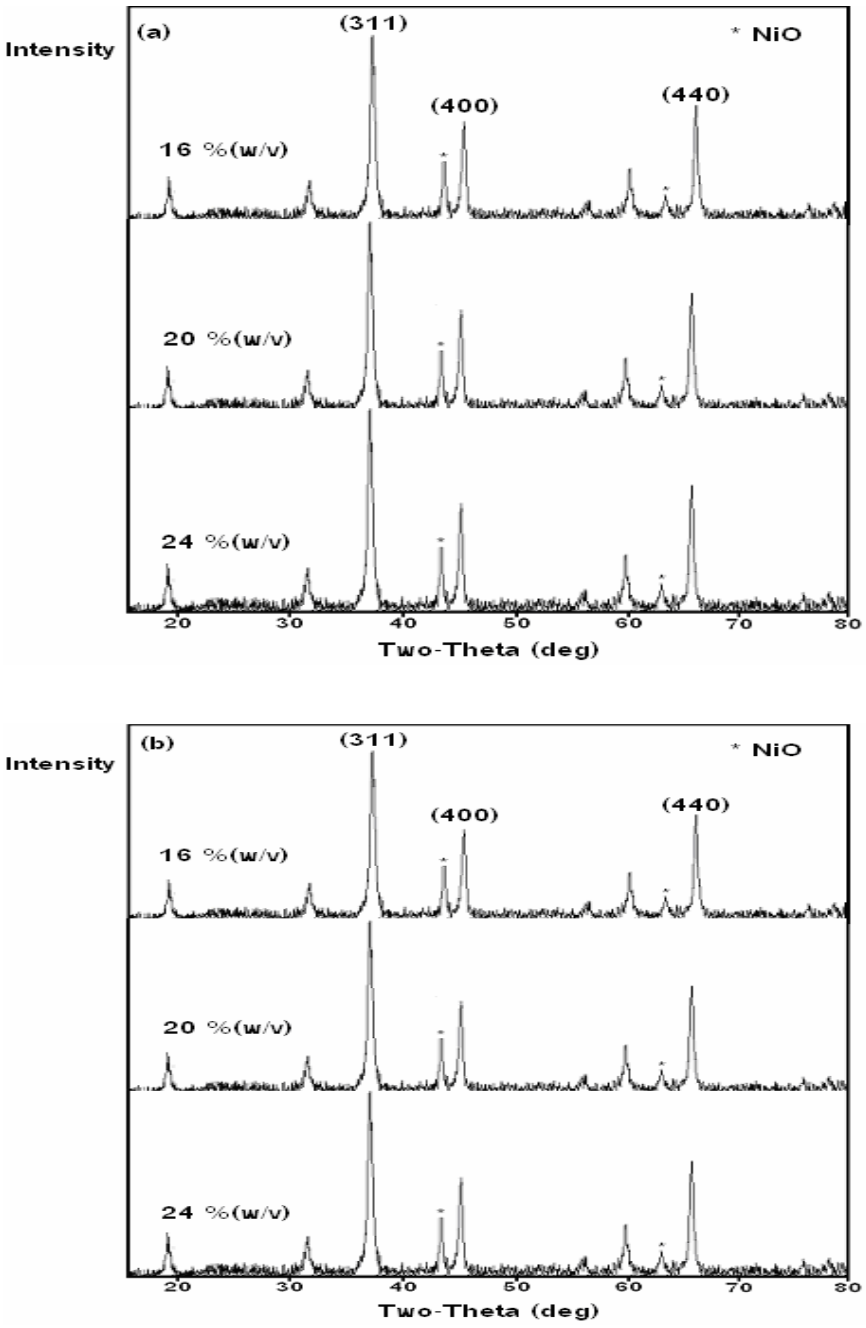


Figure E22 X-ray diffraction pattern of the powder obtained from pyrolysis of gel occurring at 60 °C in the ethanolic solution of SPAc precursor, pH 7.0 with various calcination conditions: (a) heated at 1000 °C for 5 h and (b) heated at 500 °C for 5 and held at 1000 °C for 5 h.

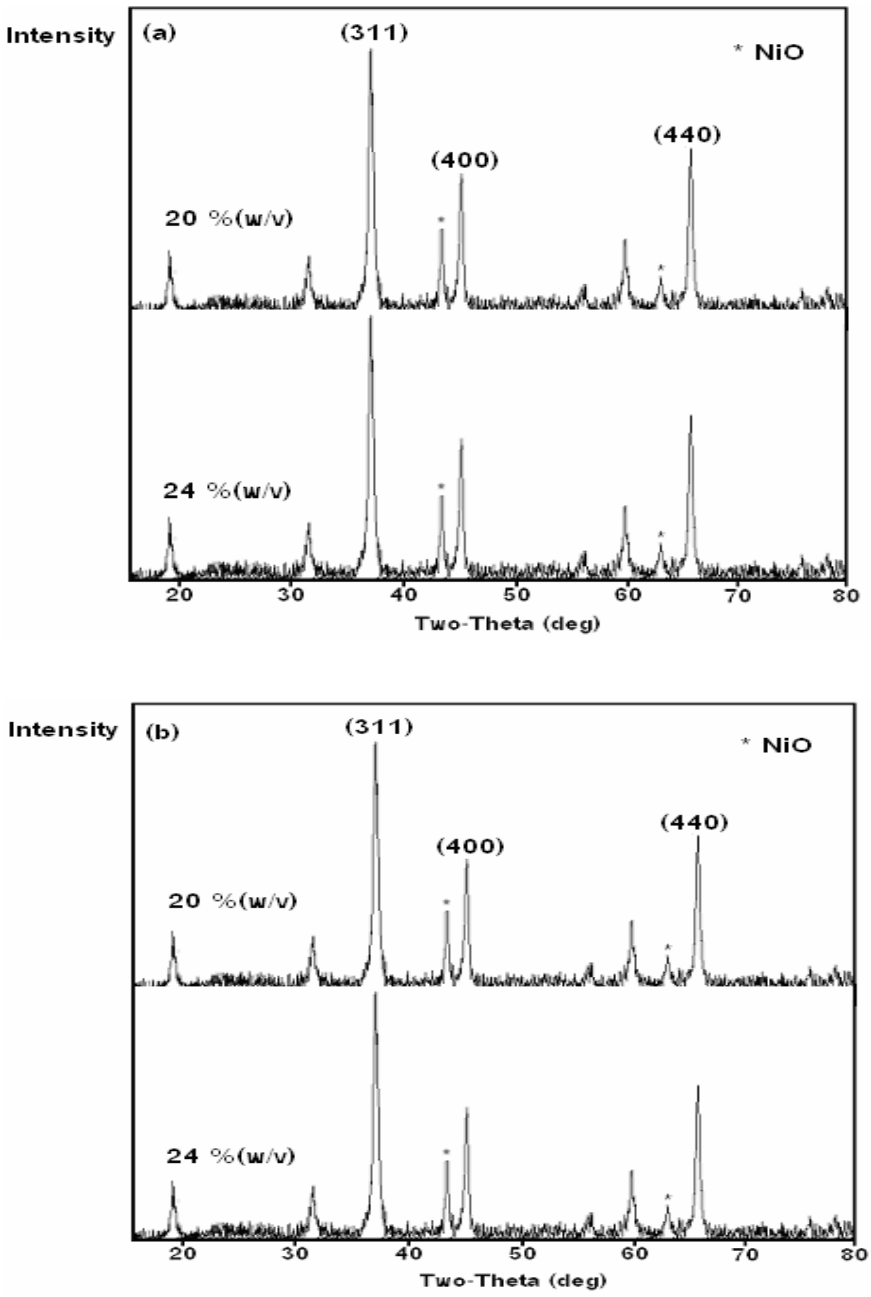


Figure E23 X-ray diffraction pattern of the powder obtained from pyrolysis of gel occurring at room temperature in the n-propanolic solution of SPAc precursor, pH 7.0 with various calcination conditions: (a) heated at 1000 °C for 5 h and (b) heated at 500 °C for 5 and held at 1000 °C for 5 h.

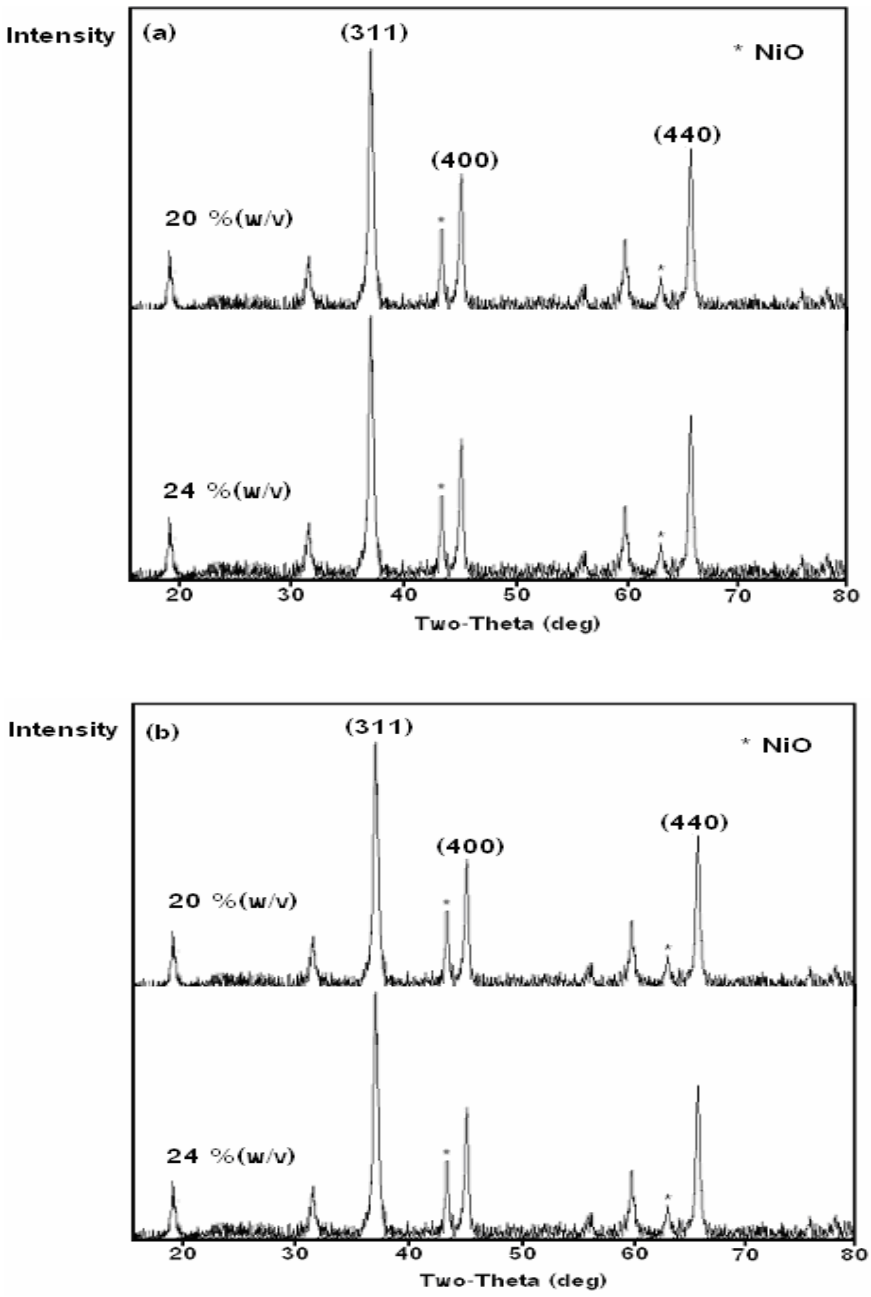
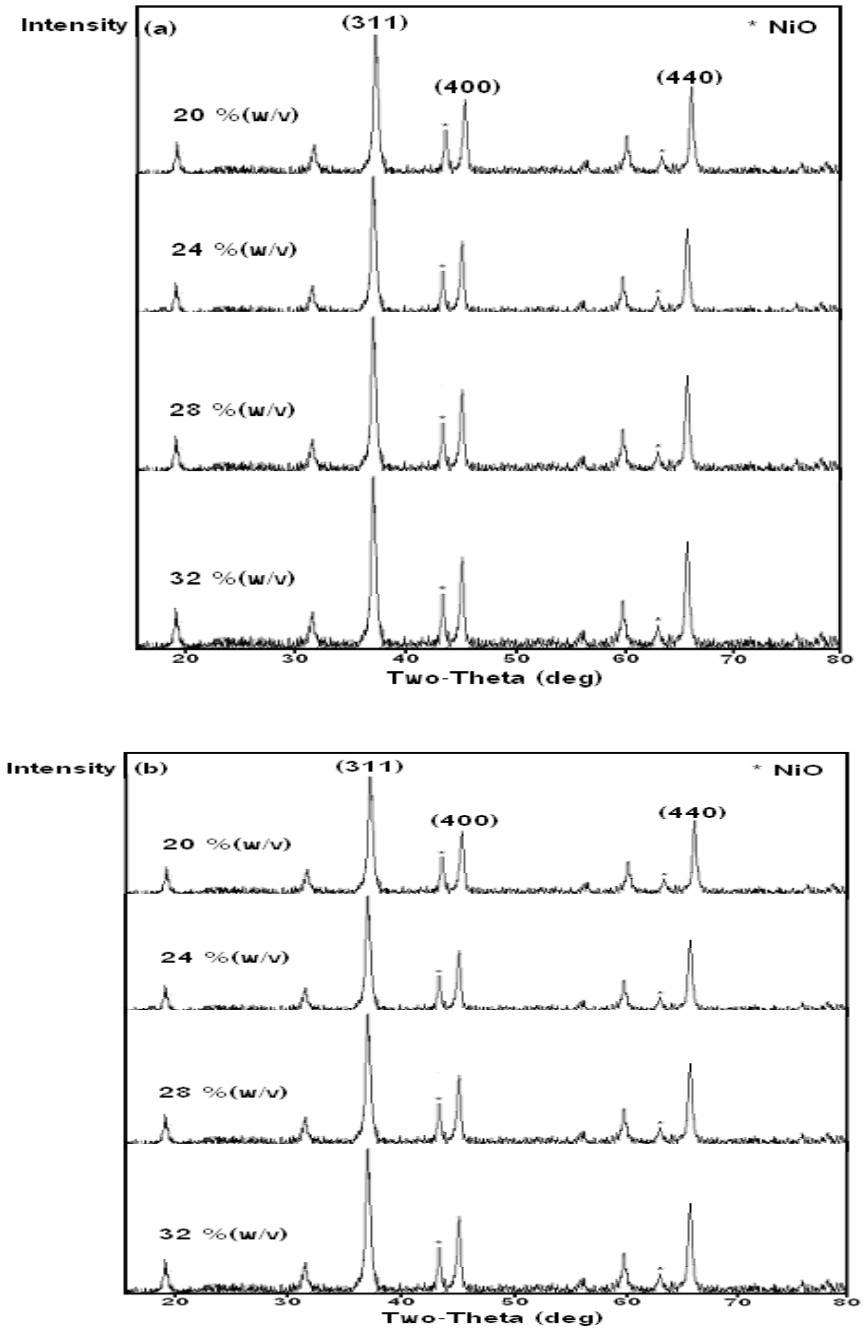
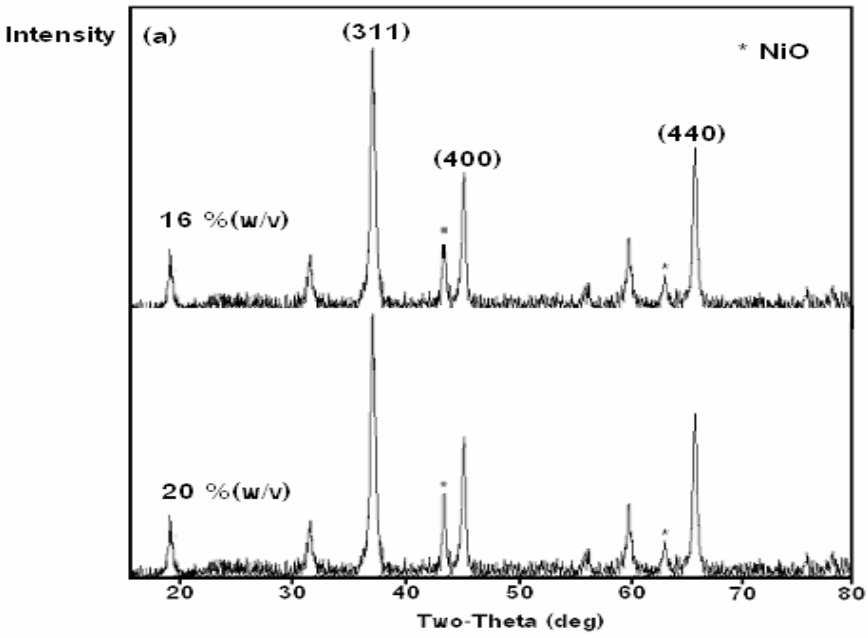
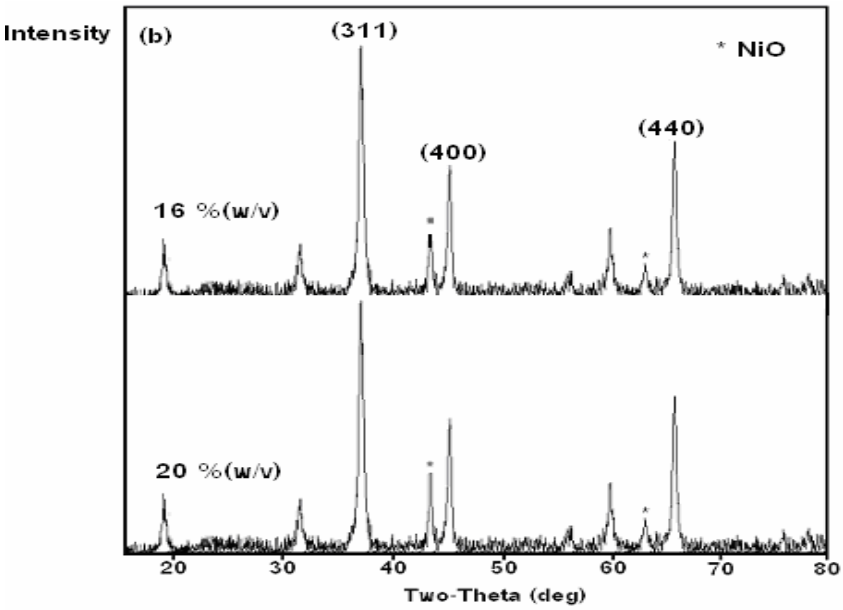


Figure E24 X-ray diffraction pattern of the powder obtained from pyrolysis of gel occurring at 60 °C in the n-propanolic solution of SPAc precursor, pH 7.0 with various calcination conditions: (a) heated at 1000 °C for 5 h and (b) heated at 500 °C for 5 and held at 1000 °C for 5 h.



. Figure E25 X-ray diffraction pattern of the powder obtained from pyrolysis of gel occurring at room temperature in the i-propanolic solution of SPAc precursor, pH 7.0 with various calcination conditions:
(a) heated at 1000 °C for 5 h and (b) heated at 500 °C for 5 and held at 1000 °C for 5 h.





. Figure E26 X-ray diffraction pattern of the powder obtained from pyrolysis of gel occurring at room temperature in the i-propanolic solution of SPAc precursor, pH 8.0 with various calcination conditions:
(a) heated at 1000 °C for 5 h and (b) heated at 500 °C for 5 and held at 1000 °C for 5 h.

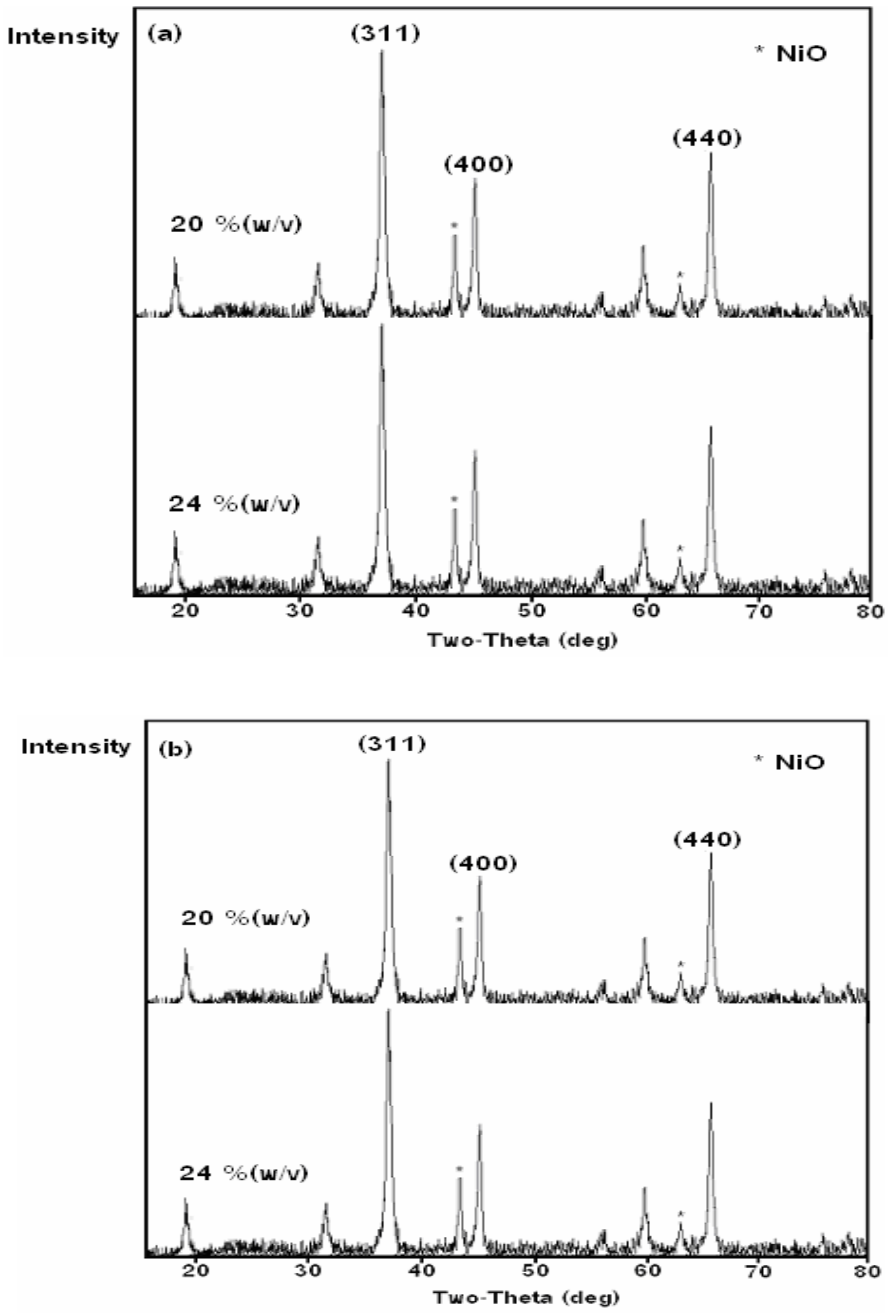


Figure E27 X-ray diffraction pattern of the powder obtained from pyrolysis of gel occurring at 60 °C in the i-propanolic solution of SPAc precursor, pH 8.0 with various calcination conditions: (a) heated at 1000 °C for 5 h and (b) heated at 500 °C for 5 and held at 1000 °C for 5 h.