

List of Figures

Fig.		Page
1	Polarization-electric field hysteresis for ferroelectric materials	5
2	Poling process	6
3	Perovskite cubic structure ABO_3	11
4	Phases structure of barium titanate	16
5	Block flow-sheet diagram of formation of barium titanate through solid state method according to Simon-Seveyrate et al, 2007 and Stojanvic et al, 2005	20
6	Block flow sheet diagram for the production of $BaTiO_3$ or $PbTiO_3$ nanopowders by Co-precipitation method	22
7	Flow sheet diagram for the production of $BaTiO_3$ or $PbTiO_3$ nanopowders by sol-gel method	26
8	Flow sheet diagram for the production of $BaTiO_3$ or $PbTiO_3$ nanopowders by microemulsion method	29
9	Flow sheet diagram for the production of $BaTiO_3$ or $PbTiO_3$ nanopowders by organic carboxylic acid precursor method	35
10	Hydrothermal dissolution / precipitation and / or in – situ transformation process	37
11	Flow sheet diagram of hydrothermal method	44
12	Relation between dielectric constant and temperature in barium titanate	51
13	Structure of multi-layer capacitor	52
14	Autoclave for preparation of barium titanate and lead titanate by hydrothermal method	60

15	XRD patterns of produced barium titanate powders with different types of organic carboxylic acid[(a): using oxalic acid, (b): citric acid, (c): tartaric acid, (d): acetic acid, and (e): benzoic acid]	72
16	XRD patterns of produced barium titanate with different organic acid types[(a): using oxalic acid, (b): citric acid, (c): tartaric acid]	72
17	XRD patterns of produced powders at different experiments statistical design conditions	76
18	Contour plots for the effects of calcination temperature and time on conversion % of BaTiO ₃ phase at different oxalic acid mole ratios [(a): 0.5, (b): 1 and (c): 1.5]	79
19	Effect of calcination temperature on the conversion of BT at different time and oxalic acid mole ratios [(a) 0.5, (b) 1 and (c) 1.5]	81
20	Effect of time on conversion of BT at different temperature and oxalic acid mole ratio	83
21	XRD analysis of the product barium titanate using oxalic acid at calcination temperature 800 °C and calcination time 2.25 h [(a) mole ratio 0.5, (b) mole ratio 1, (c) mole ratio 1.5]	86
22	3-D Plot Graph for all experimental data for conversion of BT	86
23	Contour plots for the effects of calcination temperature and time on crystallite size of conversion BaTiO ₃ at different oxalic acid mole ratios [(a): 0.5, (b): 1 and (c): 1.5]	88
24	3-D plot graph for all experimental data for average crystallite size of conversion BT	89
25	SEM micrographs of barium titanate nanopowders obtained at calcination temperature 800 °C, calcination time 2.25 h and different oxalic acid mole ratios [(a) 0.5, (b) 1, (c) 1.5]	91

26	IR spectra of precursor at different oxalic acid mole ratios [(a) oxalic acid mole ratio 0.5, (b) oxalic acid mole ratio 1 and (c) oxalic acid mole ratio 1.5]	93
27	IR spectra of spectra after calcination at different oxalic acid mole ratios [(a) oxalic acid mole ratio 0.5, (b) oxalic acid mole ratio 1 and (c) oxalic acid mole ratio 1.5]	93
28	Temperature dependence of dielectric constant of BT with different frequency at calcination time 2h, oxalic acid mole ratio 0.5, calcination temperature [(a): 800°C, (b): 1000°C and (c)1200°C]	95
29	Effect of Ti/ Zr mole ratio on phase formation, where; [(a) without added zirconium ions, (b) Ti / Zr mole ratio 0.9/0.1, (c) Ti / Zr mole ratio 0.7/0.3, (d) Ti / Zr mole ratio 0.5/0.5]	98
30	SEM micrographs of produced samples using different Ti/ Zr mole ratios, where; [(a) without added zirconium ions, (b) Ti/ Zr mole ratio 0.9/0.1, (c) Ti/ Zr mole ratio 0.7/0.3 and (d) Ti/ Zr mole ratio 0.5/0.5]	100
31	TEM images of produced samples using different of Ti/ Zr mole ratios [(a) without added zirconium ions, (b) Ti/ Zr mole ratio 0.9/0.1, (c) Ti/ Zr mole ratio 0.7/0.3 and (d) Ti/ Zr mole ratio 0.5/0.5]	101
32	EDX analysis of produced samples using different of Ti/ Zr mole ratios, where; [(a) without added zirconium ions, (b) Ti/ Zr mole ratio 0.9/0.1, (c) Ti/ Zr mole ratio 0.7/0.3 and (d) Ti/ Zr mole ratio 0.5/0.5]	103
33	Dielectric properties of produced samples using different of Ti/ Zr mole ratios, where[(a) Ti/ Zr mole ratio 0.9/0.1, (b) Ti/ Zr mole ratio 0.7/0.3, and (c) Ti/ Zr mole ratio 0.5/0.5].	104
34	XRD analysis of the produced BT samples using citric acid [(a) mole ratio 0.5, (b) mole ratio 1, (c) mole ratio 1.2 and (d) mole ratio 1.5]	107

35	XRD analysis of the produced samples using tartaric acid [(a) mole ratio 0.5, (b) mole ratio 1, (c) mole ratio 1.2 and (d) mole ratio 1.5]	107
36	XRD patterns of the produced samples using citric acid mole ratio 1.2, calcination time 2h and different calcination temperature [(a) 800°C, (b) 900°C, (c) 1000°C]	110
37	XRD patterns of the produced samples using tartaric acid mole ratio 1.2, calcination time 2h and different calcination temperature [(a) 800°C, (b) 900°C, (c) 1000°C]	110
38	XRD patterns of produced samples using citric acid mole ratio 1.2, calcination temperature 1000°C at different calcination time [(a) 1 h, (b) 1.5h and(c) 2 h]	113
39	XRD patterns of produced samples using tartaric acid mole ratio 1.2, calcination temperature 900°C at different calcination time [(a) 1 h, (b) 1.5h and(c) 2 h]	113
40	SEM micrographs of barium titanate nanopowders obtained at calcination temperature 1000°C, calcination time 2h using [(a) using citric acid mole ratio 1.2 and (b) tartaric acid mole ratio 1.2]	114
41	FT-IR spectra for (Ba, Ti) precursor using [(a) citric acid mole ratio 1.2 and (b) tartaric acid mole ratio 1.2]	116
42	FT-IR spectra of barium titanate powders at calcination temperature 1000°C, calcination time 2 h at [(a) citric acid mole ratio 1.2 and (b) tartaric acid mole ratio 1.2]	117
43	Dielectric properties of barium titanate nanopowders at calcination temperature 1000°C, calcination time 2h using [(a) citric acid mole ratio 1.2 and (b) tartaric acid 1.2]	119
44	XRD patterns of produced BT powders samples by experimental statistical design at different hydrothermal conditions.	123

45	Contour plots for the effects of hydrothermal temperature and pH on conversion of BT [(a): hydrothermal time =12 h; (b): 18 h; (c): 24 h]	126
46	Contour plots for the effects of hydrothermal temperature and hydrothermal time on conversion of BT [(a): pH =12; (b): 13; (c): 14]	127
47	Effect of hydrothermal temperature on the conversion of BT at different pH and different hydrothermal time [(a) 12, (b)18 and (c) 24 h]	129
48	Effect of time on conversion of BT at different hydrothermal temperature with different pH value [(a): 12 and (b):13]	131
49	Effect of pH value on phase formation at different hydrothermal temperature with different hydrothermal time [(a): 12h, (b): 18h and (c): 24 h]	133
50	3-D plot graph for all experimental data for conversion of BT	134
51	Contour plots for the effects of hydrothermal temperature and pH value on crystallite size of conversion BaTiO ₃ at different hydrothermal time [(a): 12, (b): 18 and (c): 24h]	136
52	3-D plot graph for all experimental data for average crystallite size of conversion BT by hydrothermal method	137
53	SEM micrographs of barium titanate nanopowders by hydrothermal method obtained at hydrothermal temperature 200°C and pH value 13 [(a) hydrothermal time 24 h, (b) hydrothermal time 12 h]	139
54	FT-IR spectra of barium titanate powders using hydrothermal method at hydrothermal temperature 170°C, hydrothermal time 24 h and pH value 13	140
55	Dielectric constant of BT using hydrothermal temperature at hydrothermal temperature 170°C, pH value 13 and hydrothermal time [(a): 12 h , (b): 24h]	142

56	XRD patterns of lead titanate powders at different calcination temperature 500-1000 °C, calcination time 2h using [(a) oxalic acid, (b) citric acid and (c) tartaric acid]	146
57	XRD patterns of lead titanate at different calcination time 1-2h a calcination temperature 600 °C using [(a) oxalic acid, (b) citric acid and (c) tartaric acid]	148
58	SEM micrographs of lead titanate powders at calcination temperature (700&1000 °C), calcination time 2 h using [(a) oxalic acid, (b) citric acid and (c) tartaric acid]	150
59	TEM micrographs of lead titanate powders at calcination temperature 600 °C, Calcination time 2 h using [(a) oxalic acid, (b) citric acid and (c) tartaric acid]	151
60	IR spectra of precursors using [(a) oxalic acid, (b) citric acid and (c) tartaric acid]	154
61	FT-IR spectra of spectra after calcination of the precursor at calcination temperature 600 °C and calcination time 2 h using [(a) oxalic acid, (b) citric acid and (c) tartaric acid]	155
62	Dielectric properties of lead titanate powders at calcination temperature 600 °C, calcination time 2 h using [(a) oxalic acid, (b) citric acid and (c) tartaric acid]	157
63	Effect of Ti/ Zr mole ratio on phase formation of lead titanate, where; [(a)without Zr ⁴⁺ ions, (b)Ti / Zr mole ratio 0.9/0.1, (c) Ti / Zr mole ratio 0.7/0.3, (d) Ti / Zr mole ratio 0.5/0.5]	159
64	TEM images of produced samples using different of Zr ⁴⁺ ions mole ratios [(a) Zr ⁴⁺ ions mole ratio 0.1, (b) Zr ⁴⁺ ions mole ratio 0.3 and (c) Zr ⁴⁺ ions mole ratio 0.5]	161
65	EDX analysis of produced samples using different of Ti/Zr mole ratios, where; [(a)without added zirconium ions, (b) Zr ⁴⁺ ions mole 0.1, (c) Zr ⁴⁺ ions mole 0.3 and (d) Zr ⁴⁺ ions mole 0.5]	163

66	Dielectric properties of produced samples using different of Zr^{4+} ions mole ratios, [(a) Zr^{4+} ions mole ratio 0.1, (b) Zr^{4+} ions mole ratio 0.3 and (c) Zr^{4+} ions mole ratio 0.5].	164
67	XRD patterns of lead titanate synthesized by hydrothermal method at different hydrothermal temperature , hydrothermal time 24h and pH 13.5 [(a) $150^{\circ}C$, (b) $180^{\circ}C$, (c) $200^{\circ}C$, (d) $230^{\circ}C$]	167
68	XRD patterns of lead titanate samples at different hydrothermal time at constant hydrothermal temperature $200^{\circ}C$ & pH 13 [(a) 24h, (b) 30 h and (c) 36 h]	169
69	XRD patterns of lead titanate samples at different pH at constant hydrothermal temperature $200^{\circ}C$ & hydrothermal time 24 h [(a) pH 13, (b) 13.5 and (c) 14]	171
70	TEM micrographs of lead titanate at hydrothermal temperature $200^{\circ}C$, hydrothermal time 30h and pH 13	173
71	TEM micrographs of lead titanate at hydrothermal temperature $200^{\circ}C$, hydrothermal time 30h and pH ≥ 14	173
72	IR spectra of lead titanate nanopowders at hydrothermal temperature $200^{\circ}C$, hydrothermal time 30h and pH 13	174
73	Dielectric constant of lead titanate nanopowders at hydrothermal temperature $200^{\circ}C$, hydrothermal time 30 h and pH 13	175

List of Tables

Table		Page
1	Curie Temperature and spontaneous polarization of some ferroelectric crystals	8
2	Electromechanical coupling factors of some piezoelectric materials	14
3	General comparison of the synthesis of BT & PT powders by different routes	45
4	Dielectric constant and dissipation factor of some piezoelectric materials	47
5	Ferroelectric properties of BT & (1-x) PZT ceramics	48
6	Relation between thickness PTC heaters and voltage	54
7	Weight of organic acids used in the synthesis of barium titanate by organic acid precursor	58
8	Weight of organic acids used in the synthesis of lead titanate nanopowders by organic acid precursor	58
9	Box-Behnken design with 3 levels and 3 variables	65
10	The levels of variables chosen for the Box-Behnken optimization experiments by organic carboxylic acid precursor	66
11	The levels of variables chosen for the Box-Behnken optimization experiments by hydrothermal method	66
12	Conversion and crystallite size of BaTiO ₃ according to experimental statistical Design conditions	75
13	Barium titanate phase conversion and crystallite size according to experimental statistical Design conditions	122
14	Crystallite size of lead titanate PT using different of organic acid at calcination time 2h	145