

Contents

	Page
บทคัดย่อ	(3)
Abstract	(4)
Acknowledgement	(5)
The relevancy of the research work to Thailand	(6)
Contents	(7)
List of tables	(9)
List of figures	(11)
List of abbreviations	(14)
1 Introduction	1
Introduction	1
Literature reviews	3
Objectives	10
2 Methods of study	11
Chemicals	11
Instruments	12
Preparation of oxalato complexes	13
Products characterizations	14
3 Results	17
Preparation of oxalato complexes	17
Products characterizations	18
Titration technique	18
Elemental analysis	18

Contents (continued)

	Page
3 Results (continued)	
X-ray single crystal data of $K_{18}\{K[Al_{0.97}Cr_{0.03}(C_2O_4)_3]_6\}Cl.18H_2O$ (RedRhomb)	20
UV-Visible absorption spectroscopy	33
Infrared spectrometry	35
X-ray powder diffraction	40
Scanning electron microscope with energy dispersive x-ray spectrometer	44
Thermogravimetric analysis	46
Differential scanning calorimeter	49
4 Discussion	53
Preparation of oxalato complexes	53
Products characterizations	53
Titration technique	53
Elemental analysis	55
X-ray single crystal data of $K_{18}\{K[Al_{0.97}Cr_{0.03}(C_2O_4)_3]_6\}Cl.18H_2O$ (RedRhomb)	56
UV-Visible absorption spectroscopy	57
Infrared spectrometry	67
Thermogravimetric analysis and differential scanning calorimeter	72
5 Conclusion	78
References	79
Appendix	86
Vitae	92

List of tables

Table		Page
1	Isomorphous oxalate complexes, $K_3[M(C_2O_4)_3] \cdot 3H_2O$	4
2	The solubility of RedCubic, RedRhombic, and Blue crystals	17
3	The content of oxalate group by titration technique	18
4	The percentage of element (K, Na, Cr, Al) in oxalato complexes	19
5	The percentage of element (C, H, O) in oxalato complexes	19
6	Crystal data and structure refinement of $K_{18}\{K[Al_{0.97}Cr_{0.03}(C_2O_4)_3]_6\}$ Cl.18H ₂ O (RedRhombic)	21
7	The selected bond distances (Å) of $K_{18}\{K[Al_{0.97}Cr_{0.03}(C_2O_4)_3]_6\}$ Cl.18H ₂ O (RedRhombic)	23
8	Bond Angles (°) of $K_{18}\{K[Al_{0.97}Cr_{0.03}(C_2O_4)_3]_6\}$ Cl.18H ₂ O	25
9	Absorption band of RedCubic, RedRhombic, Blue, and $K_3[Cr(C_2O_4)_3] \cdot 3H_2O$	35
10	IR data of $K_3[Cr(C_2O_4)_3] \cdot 3H_2O$, $K_3[Al(C_2O_4)_3] \cdot 3H_2O$, RedCubic, RedRhombic, and Blue complexes	39
11	The elements in oxalato complexes as found by SEM/EDX	46
12	Summary of the TGA results obtained by heating from 30° to 1200°C at 10 °C/min in air	49
13	Summary of the DSC results obtained by heating from 30° to 500°C at 10 °C/min in air	52
14	Calculated spectral transitions for $[Cr(C_2O_4)_3]^{3-}$ complex	60
15	Observed frequencies of various vibrational modes of $K_3[Cr(C_2O_4)_3] \cdot 3H_2O$ and $K_3[Al(C_2O_4)_3] \cdot 3H_2O$ complexes (cm ⁻¹)	72
16	$K_3[Al(C_2O_4)_3] \cdot 3H_2O$ x-ray diffraction analysis of residues	73

List of tables (continued)

Table		Page
17	Non-hydrogen atom coordinate and site occupation parameters of $K_{18}\{K[Al_{0.97}Cr_{0.03}(C_2O_4)_3]_6\}Cl.18H_2O$ (RedRhombo)	87
18	Hydrogen atom coordinates and isotropic displacement parameters of $K_{18}\{K[Al_{0.97}Cr_{0.03}(C_2O_4)_3]_6\}Cl.18H_2O$ (RedRhombo)	89
19	Non-hydrogen atomic displacement parameters of $K_{18}\{K[Al_{0.97}Cr_{0.03}(C_2O_4)_3]_6\}Cl.18H_2O$ (RedRhombo)	90

List of figures

Figure		Page
1	The 50% thermal ellipsoid of core structure of $K_{18}\{K[Al_{0.97}Cr_{0.03}(C_2O_4)_3]_6\}Cl.18H_2O$ (RedRhomb) complex molecule plot	29
2	Two 180° views of $K_{18}\{K[Al_{0.97}Cr_{0.03}(C_2O_4)_3]_6\}Cl.18H_2O$ (RedRhomb) complex molecule	30
3	Two views of unit cell plotted down c axis	31
4	Unit cell plotted down b axis	32
5	UV-Visible absorption spectrum of oxalic acid in aqueous solution	33
6	UV-Visible absorption spectrum of RedCubic, RedRhomb, Blue, and $K_3[Cr(C_2O_4)_3].3H_2O$ in aqueous solution	34
7	UV-Visible absorption spectrum of RedCubic, RedRhomb, Blue, and $K_3[Cr(C_2O_4)_3].3H_2O$ in solid state	34
8	IR spectrum of polystyrene	36
9	IR spectrum of oxalic acid dihydrate	36
10	IR spectrum of $K_3[Cr(C_2O_4)_3].3H_2O$	37
11	IR spectrum of $K_3[Al(C_2O_4)_3].3H_2O$	37
12	IR spectrum of RedCubic complex	38
13	IR spectrum of RedRhomb complex	38
14	IR spectrum of Blue complex	39
15	X- ray diffraction pattern of $K_3[Cr(C_2O_4)_3].3H_2O$ complex from powder technique	41
16	X- ray diffraction pattern of $K_3[Al(C_2O_4)_3].3H_2O$ complex from powder technique	41
17	X- ray diffraction pattern of RedCubic complex from powder technique	42

List of figures (continued)

Figure		Page
18	X- ray diffraction pattern of RedCubic complex from single crystal technique	42
19	X- ray diffraction pattern of RedRhombic complex from single crystal technique	43
20	X- ray diffraction pattern of Blue complex from powder technique	43
21	SEM/EDX spectrum of RedCubic complex	44
22	SEM/EDX spectrum of RedRhombic complex	45
23	SEM/EDX spectrum of Blue complex	45
24	TGA spectrum of $K_3[Al(C_2O_4)_3].3H_2O$ complex	46
25	TGA spectrum of $K_3[Cr(C_2O_4)_3].3H_2O$ complex	47
26	TGA spectrum of RedCubic complex	47
27	TGA spectrum of RedRhombic complex	48
28	TGA spectrum of Blue complex	48
29	DSC spectrum of $K_3[Al(C_2O_4)_3].3H_2O$ complex	50
30	DSC spectrum of $K_3[Cr(C_2O_4)_3].3H_2O$ complex	50
31	DSC spectrum of RedCubic complex	51
32	DSC spectrum of RedRhombic complex	51
33	DSC spectrum of Blue complex	52
34	Orbital energy level diagrams of Cr^{3+} (d^3) configuration in octahedral	58
35	Partial and full energy level diagram of a Cr^{3+} (d^3)	59
36	Tanabe-Sugano diagram for d^3 in an octahedral field	59
37	Absorption spectrum of ruby, (Cr^{3+} impurities in corundum, Al_2O_3)	62

List of figures (continued)

Figure		Page
38	The color-change of RedCubic	62
39	Partial energy level diagram of ruby laser	64
40	Part of the absorption and emission spectra of ruby (Al_2O_3 with Cr^{3+} impurities)	66
41	The main linkage types of oxalate ligand with metal	67
42	Atomic displacements in the totally symmetric normal modes of the planar and 90° twisted oxalate anion	68
43	Normal modes of vibration octahedral XY_6 molecules	69

Lists of abbreviations

°	=	degree
Å	=	angstrom unit (1 Å = 10 ⁻¹⁰ meter)
A.R.	=	Analytical Reagent
cm ⁻¹	=	wavenumber
λ	=	wavelength
ε	=	molar extinction coefficient
D _c	=	calculated density
D _m	=	measured density
IR	=	Infrared
UV	=	Ultraviolet
L	=	ligand
g	=	gram
g/cm ³	=	gram per cubic centimeter
mg	=	milligram
mmol	=	millimole
mL	=	milliliter
nm	=	nanometer
h	=	hour
keV	=	kilo electron volt