Fabrication and Application of (1-x)NaCl+xKCl Solid Solution

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***Abstract*— (1-x)NaCl+ xKCl solid solutions are prepared by the starting materials NaCl (0.9, 0.95)and KCl (0.1, 0.05)in equal molar ratio. The solid solutions are heat-treated at various temperature and XRD analyses are carried out for the solid solutions to examine the crystalline phase, crystallographic orientation and lattice parameters. The electrical properties of the solutions are determined by using the conductometer. The solid solutions are utilized as crystal oscillator and outcoming frequencies, capacitances and dielectric constants are also investigated.**

***Keywords*— NaCl, KCl, XRD analyses, crystal oscillator, conductometer, electrical properties.**

I. Introduction

A great deal has been known for many years about materials that are useful in every day life and industry. The deeper understanding of materials which we now have has come largely on the way that atoms are arranged when they are close together. Sodium Chloride (NaCl) and Potassium Chloride (KCl) are two common ionic crystals and have the characteristics such as strong infrared absorption, low electrical conductivity and good resistivity at low temperature, low electrical conductivity and low resistivity at high temperature. This type of solutions are called mixed crystals and sufficiently simple in structure so it is possible to study the qualitative change in lattice parameter of the solvent caused by the present of solute. In this research the characteristics of these materials are carried out by studying X-ray diffraction analyses and Crystal oscillator and Conductometer measurements.[1-3]

II. Experimental Procedures

Sample preparation procedure, XRD analyses, construction of conductometer and crystal oscillator are described and measurements of electrical properties of solid solution by these instruments are also presented.

A. Sample Preparation

Sample preparation includes the following steps: weighing of raw materials (NaCL and KCl), grinding, shaping by mould pressing or roll-pressing and calcining. Firstly analar grade 99.9% purified raw materials NaCl and KCl are weighed according to desire compositions (0.9 NaCl + 0.1 KCl and 0.95NaCl + .05 KCl for this research). Then sample materials are mixed and grounded to obtain homogeneous and uniform grain size by agate motor. The grinded solution is shaped by sample making machine to get 2.511cm diameter and 0.385cm thickness circular shaped pallet samples. These pallet samples are heat treated 30minutes under temperature 100, 150, 200, 250, 300, 350, 400, 450 and 500°C respectively. Before and after measurements the samples are stored in dry cabinet (with temperature 30°C).

B. XRD analyes

The X-ray diffractometry is mainly used for the indentification and qualification of compounds by their diffraction pattern and variation of lattice parameter and intensity of (1-x)NaCl+ xKCl solid solutions according to various temperature are also investigated. The samples are scanned through intensity as a function of an angle 2θ from 20° to 60°. The diffracted wave actually arises from true lattice planes and lattice parameters are derived from the equation: $1/d^2=(h^2+k^2+l^2)/a^2$. Each diffracted ray was recorded as a peak. The peak heights are roughly proportional to the rays' intensity.

C. Design and Construction of Conductometer and Measurement of Electrical Properties

The circuit diagram of conductometer is shown in Fig.1. Before measuring the conductivity and resistivity of solid solution, the conductometer circuit is constructed with two 353ICs (4 op-amp / 4 stages)[4]. The first stage is an astable multivibrator or square wave generator configuration. The square wave output from this stage is the input of second stage of voltage follower configuration with high input and low output impedance. It serves to isolate the multivibrator stage from loading effects due to third and fourth stages. The third stage is an inverting amplifier configuration. The output of this stage is related to the reading of conductance of the tested sample (cell). The second and third op-amps are connected with 1% tolerance resistors (10kΩ, 100kΩ, 30kΩ, 300kΩ) and applied as READ and CALIBRATION of test cell. The fourth stage is used to convert AC voltage to DC current in rectifier bridge feedback network. Before measuring the conductance of sample pallet, it needs to check the calibration (10mA scale meter reading for standard calibration). After calibration, set first switch to READ position and read the scale value in mA. Conductance can be calculated from multiplying meter reading with 0.01 for 0.02mS scale. The conductivity can determined by using the formula σ = GL/A. where σ, G, L and A are conductivity, conductance, thickness and crosssectional area of the sample. The resistivity of the sample can also calculated by the formula $\rho = 1/\sigma$.

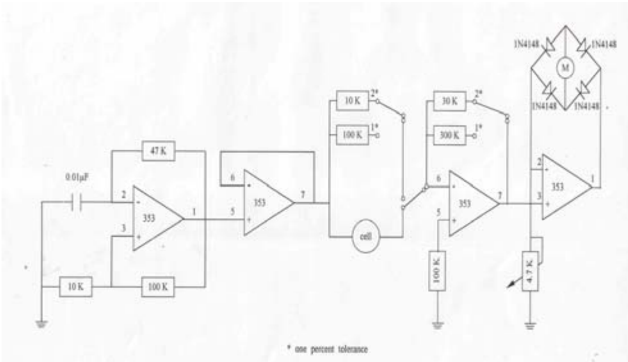

Fig. 1 Conductometer Circuit

D. Design and Construction of Crystal Oscillator and Measurement of Frequency, Capacitance and dielectric constant

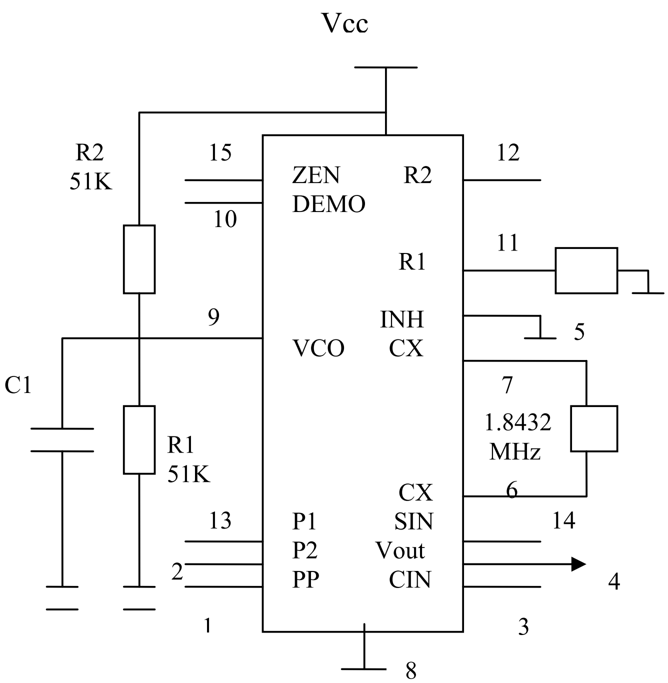

Fig.2 Crystal Oscillator Circuit

In Fig.2. the crystal oscillator circuit used for this research is described. There are many kinds of oscillators such as Hartley oscillator, Wien bridge oscillator, Crystal oscillator and Phase shift oscillator. Among these oscillators, output frequency of a crystal oscillator is more stabilized than other type of oscillators. Construction of crystal oscillator circuit in this research depends mainly in CD 4046B IC[5]. The crystal is placed between pin 6 and 7 of IC as used as resonant tank circuit. 20kΩ resistor in this circuit is used as standard resistor to determine the frequency of the crystal. It needs standard calibration before measurement and quartz crystal of 1.8432MHz is used for standard calibration. The sample pallet is placed in place of quartz crystal and starts the measurement of frequency. For CD 4046B IC, the constant K = 1/3 and the relation f = k/RC turns to f = 1/3RC. Capacitance of the sample can be calculated from this formula. Permittivity of solid solution is calculated by equation $C = \varepsilon A/d$ and dielectric constant can also be obtained from the equation $\varepsilon_r = \varepsilon / \varepsilon_o$ (where $\varepsilon_o = 8.85\text{x}\ 10^{-12}\ \text{F m}^{-1}$).

III. Results and Discussion

The results for XRD analyses of (1-x)NaCl+ xKCl solid solutions are shown in Table I,II, and also in Fig. 3 to 10. From X-ray diffraction patterns it was investigated that both NaCl and KCl crystal structure in all patterns coexist in 0.9NaCl+0.1KCl ratio and means that molar ratio 0.1mol of KCL is not soluble in this solid solution. In 0.95NaCl+0.05KCl ratio, pure NaCl-type crystal structure is found in all XRD patterns. In both cases, lattice parameter increase with increasing temperature. It means that volume expansion of unit cell is assumed to be uniform in solid solution. The relative intensity ratio was decreased with increasing temperature in 2θ values of (200) reflection plane increased[6,7].

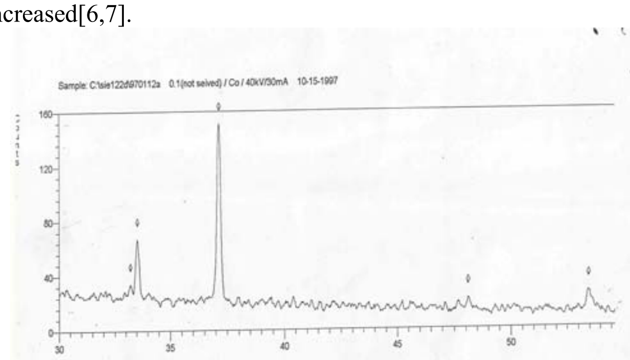

Fig.3 XRD Patterm of 0.9NaCl+0.1KCl solid solution heat treated at temperature 100°C

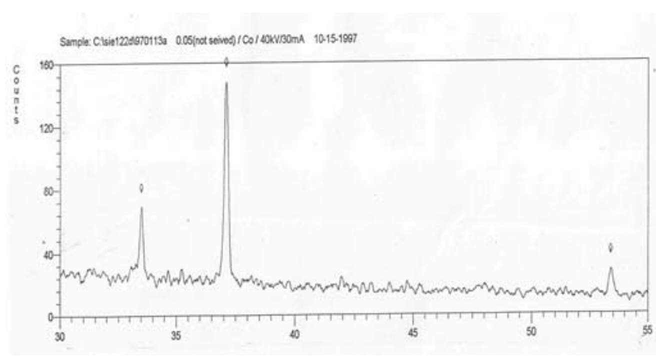

Fig.4 XRD Patterm of 0.95NaCl+0.05KCl solid solution heat treated at temperature 100°C

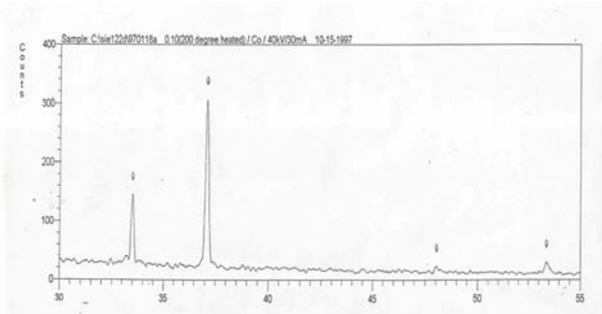

Fig.5 XRD Patterm of 0.9NaCl+0.1KCl solid solution heat treated at temperature 200°C.

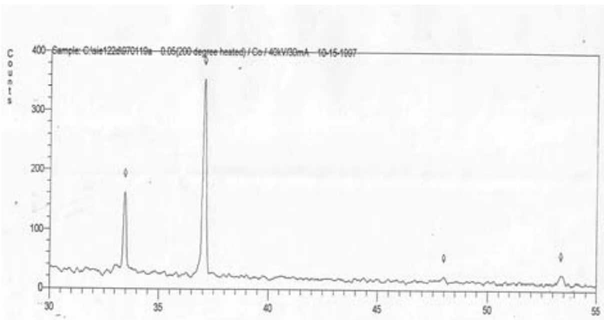

Fig.6 XRD Patterm of 0.95NaCl+0.05KCl solid solution heat treated at temperature 200°C.

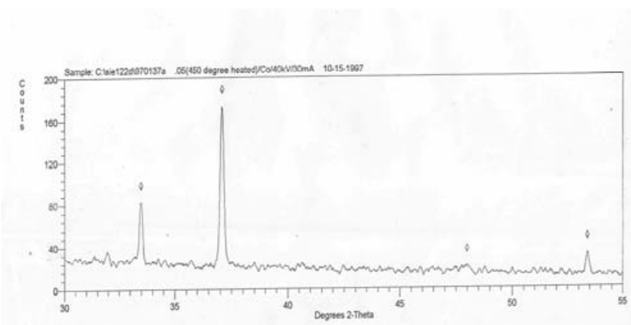

Fig.7 XRD Patterm of 0.9NaCl+0.1KCl solid solution heat treated at temperature 450°C.

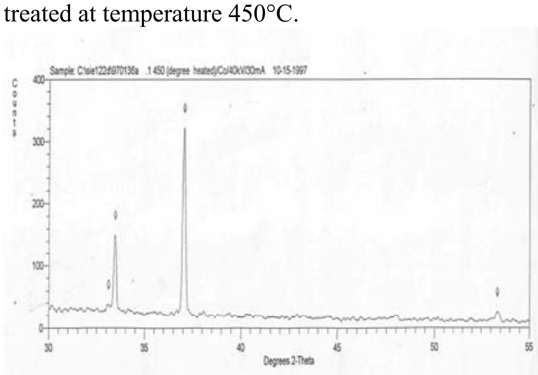

Fig.8 XRD Patterm of 0.95NaCl+0.05KCl solid solution heat treated at temperature 450°C.

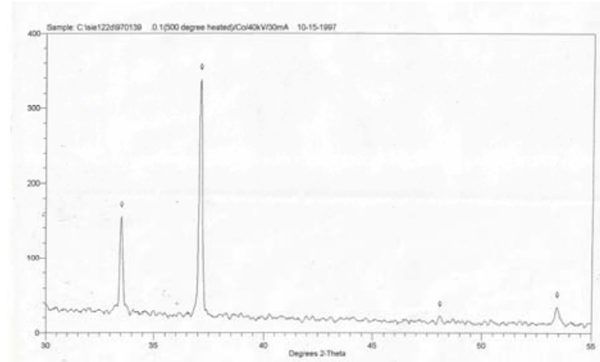

Fig.9 XRD Patterm of 0.9NaCl+0.1KCl solid solution heat treated at temperature 500°C.

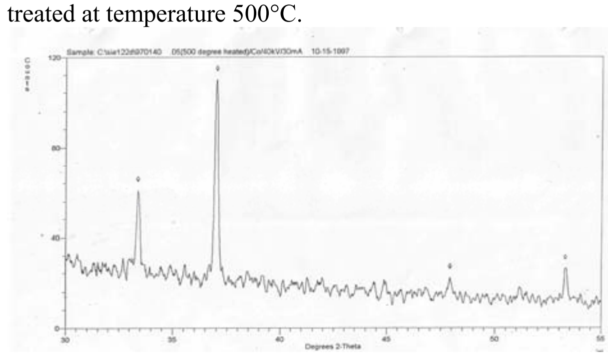

Fig.10 XRD Patterm of 0.95NaCl+0.05KCl solid solution heat treated at temperature 500°C.

TABLE I
TEMPERATURE DEPENDENCE OF LATTICE PARAMETER

Temperature(°C)	Lattice Parameter (°A)		
	0.9 NaCl	0.1KCl	0.95NaCl+ 0.05KCl
100	5.618	6.258	5.624
150	5.620	6.262	5.626
200	5.624	6.272	5.629
250	5.626	6.272	5.630
300	5.629	6.280	5.631
350	5.631	6.288	5.632
400	5.632	6.292	5.634
450	5.637	6.294	5.638
500	5.640	6.298	5.643

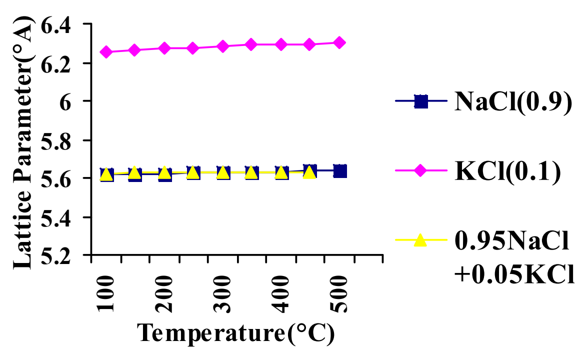

Fig. 11 Temperature Dependance of Lattice Parameter of solid solutions

TABLE II

TEMPERATURE DEPENDENCE OF RELATIVE INTENSITY RATIO AND 2Θ VALUE

Tempera -ture(°C)	0.9NaCl+0.1KCl		0.95NaCl+0.05KCl	
	Relative intensity ratio	2θ	Relative intensity ratio	2θ
100	0.9346	36.98	0.9259	37
150	0.9259	37	0.9174	37.04
200	0.9252	37.04	0.8928	37.06
250	0.9197	37.08	0.8772	37,07
300	0.9090	37.10	0.8695	37.08
350	0.9090	37.12	0.8974	.3709
400	0.9009	37.14	0.8974	37.10
450	0.8621	37.15	0.8403	37.12
500	0.8541	37.16	0.8064	37,14

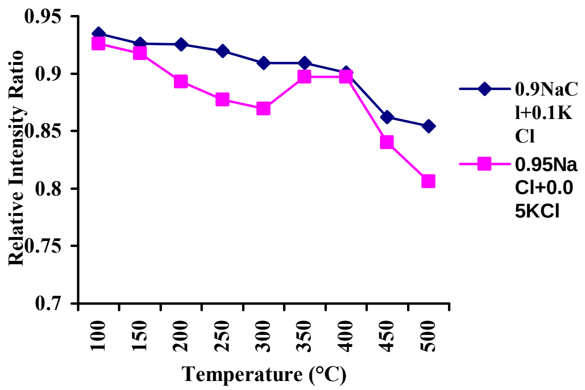

Fig. 12 Temperature Dependence of Relative intensity ratio of solid solutions.

The measurement results of (1-x)NaCl+ xKCl solid solutions by using conductometer circuit and crystal oscillator circuits are shown in Table III,IV, and also in Fig. 11 to 14. Measurements results by conductometer circuit shows that conductivity of the solution is decrease and resistivity of the solution increase when temperature is raised. In perfect crystals conductivity can occur only if ions exchange places by diffusion. In this case, the number of existing vacancies or interstitial ions decrease when the temperature is raised.

Temperature dependence of frequency, capacitance and dielectric constant from crystal oscillator measurement show that frequency decreases with increasing temperature while capacitance and dielectric constants increase. The frequency of measured sample solution is in MHz range and the capacitance is in pF range for both molar ratios. The dielectric constant is 9.95 at 100°C and 11.83 at 500°C for 0.95NaCl+ 0.05KCl ratio and 10.47 at 100°C and 11.31 at 500°C for 0.9NaCl+ 0.1KCl ratio.

TABLE III

TEMPERATURE DEPENDENCE OF CONDUCTIVITY , RESISTIVITY, FREQUENCY, CAPACITANCE AND DIELECTRIC CONSTANT 0.9 NACL+ 0.1 KCL

Temper-ature (°C)	σ ($x10^{-5}$ Sm^{-1})	ρ ($x10^3$ Ωm)	F (MHz)	C (pF)	ε_r
100	7.541	13.261	1.398	11.92	10.47
150	7.308	13.684	1.384	12.04	10.58
200	7.075	14.134	1.351	12.34	10.84
250	6.842	14.616	1.342	12.42	10.91
300	6.608	15.133	1.337	12.47	10.95
350	6.375	15.686	1.326	12.57	11.04
400	6.142	16.281	1.312	12.70	11.14
450	5.909	16.923	1.304	12.78	11.23
500	5.675	17.621	1.295	12.87	11.31

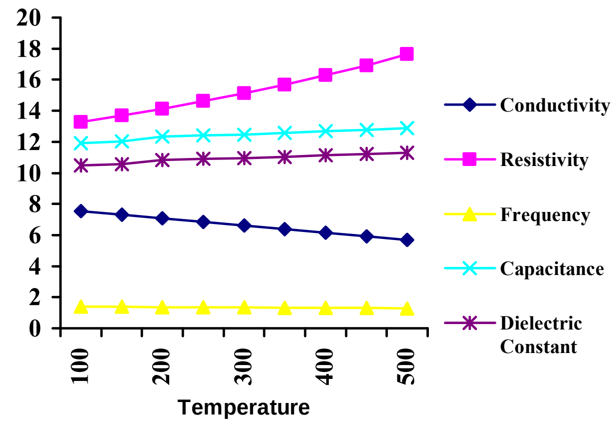

Fig. 13. Temperature Dependence of Conductivity , resistivity, Frequency, capacitance and dielectric constant 0.9 Nacl+ 0.1 KCl.

TABLE IV

TEMPERATURE DEPENDENCE OF CONDUCTIVITY , RESISTIVITY, FREQUENCY, CAPACITANCE AND DIELECTRIC CONSTANT 0.95 NACL+ 0.05 KCL.

Temper-ature (°C)	σ ($x10^{-5}$ Sm^{-1})	ρ ($x10^3$ Ωm)	F (MHz)	C (pF)	ε_r
100	7.306	13.687	1.411	11.81	9.955
150	7.083	14.118	1.383	12.05	10.14
200	6.859	14.579	1.355	12.30	10.35
250	6.635	15.071	1.327	12.56	10.57
300	6.412	15.595	1.299	12.83	10.81
350	6.188	16.160	1.271	13.11	11.03
400	5.964	16.767	1.243	13.41	11.28
450	5.741	17.418	1.215	13.72	11.55
500	5.517	18.125	1.187	14.04	11.83

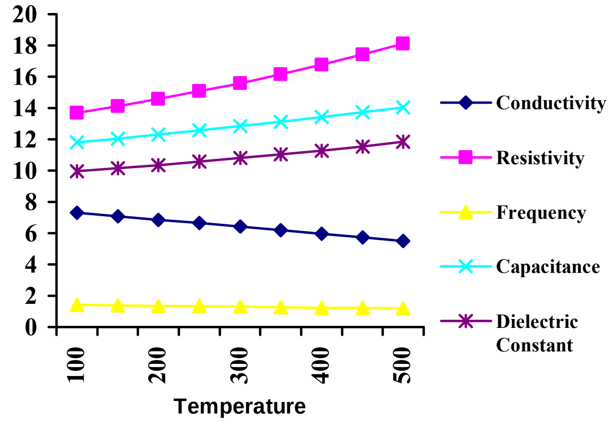

Fig. 14 Temperature Dependence of Conductivity , resistivity, Frequency, capacitance and dielectric constant 0.9 5Nacl+ 0.05 KCl.

IV. Conclusion

This research paper is about the study of (1-x)NaCl+xKCl solid solution preparation, X -ray diffraction analyses of this sample solutions for the identification and qualification of compounds by their diffraction pattern. Studying of temperature dependence of lattice parameter and relative intensity ratio of this research gives lattice parameter increase with increasing temperature and relative intensity ratio decreases in both (x=0.1 and x=0.05) ratios. It can also conclude from the results conductometer and crystal oscillator measurements that conductivity and frequency of these solid solution decrease with increasing temperature while the resistivity, capacitance and dielectric constants increase. It can also conclude that the measured frequency of this sample range in MHz, capacitance is in pF range. In 0.95NaCl+0.05KCl ratio the solution has single phase and have low electrical conductivity and good resistivity at high temperature and good electrical conductivity and low resistivity at low temperature. In place of Quartz crystal in crystal oscillator circuit, the sample solid solution is utilized as a crystal. So it is possible to produce and use the cheaper crystal like the sample (1-x)NaCl +xKCl solid solutions.

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