

## Investigation of a Rubidium Fast Ionic Conductor [Cu<sub>2</sub>Hgl<sub>4</sub>:0.xRbI] where (x=0.1-0.3 mol. wt.%)

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### Abstract

An investigation has been taken for the development of new Rb<sup>+</sup> ion conducting composite fast ionic systems, [Cu<sub>2</sub>Hgl<sub>4</sub>:0.xRbI] (where x=0.1-0.3 mol. wt. %), were synthesized, using [Cu<sub>2</sub>Hgl<sub>4</sub>] ternary composite system as the host. [Cu<sub>2</sub>Hgl<sub>4</sub>] compound belongs to the fast ion conductors of A<sub>2</sub>Ni<sub>4</sub> (A=Cu; N=Hg and X=I). As the crystal lattice amends from a tetragonal to a hexagonal structure, the compounds [Cu<sub>2</sub>Hgl<sub>4</sub>] phase also demonstrates a change near 388 K. Near 445K, [Cu<sub>2</sub>Hgl<sub>4</sub>] is substituted by an equilibrium mixture containing α-CuI and Hgl<sub>2</sub>. Ionic conductivity increases by an order of magnitude or more at each of these phase transition. Along with the substitution of Hg<sup>++</sup> from Cu<sup>+</sup> in [Cu<sub>2</sub>Hgl<sub>4</sub>], there is also a slight increase in the room temperature conductivity, as a result of either increased crystalline defects or a characterized increased Rb<sup>+</sup> substitution. FTIR, FAR-IR, SEM and EDAX analyses were executed to confirm the formation of fast ion conductors [Cu<sub>2</sub>Hgl<sub>4</sub>:0.xRbI] (where x=0.1-0.3 mol. wt. %).



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### Introduction

Solid state sciences have exerted a broader impact on newer areas of science and technology, as evident by the shooting growth of the field observed during the last quarter century<sup>1</sup>. A phase transition is observed in the solid electrolytes A<sub>2</sub>Ni<sub>4</sub> (A=Cu; N=Hg) when put under a specific, static temperature. These solid electrolytes possess a certain kind of ion which is free from its confinement in specific

lattice sites, and moves liberally across the three dimensional structure. Solid composites have composed as a good system of multiphase ionic composite materials for developing solid state fast ionic devices<sup>4,5,6</sup>. Copper mercury iodide Cu<sub>2</sub>Hgl<sub>4</sub>, is unique amongst the fast-ionic conductors having a tetragonal unit cell that experiences an order-disorder phase transition at a given temperature of approximately 76°C, which, incidentally, is the

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standard temperature for  $\alpha$ -phase demonstrating mixed ionic and electronic conductivity. The stable  $\alpha$ -phase of  $\text{Cu}_2\text{HgI}_4$ , which is not fastionic, under encompassing conditions is tetragonal with space group. The iodine atoms adopt a some distorted face centered cubic (fcc) arrangement and the cations occupy 3/8 of the tetrahedrally coordinated positions in an ordered manner derived from the chalcopyrite ( $\text{A}_2\text{NI}_4$ ) structure by the accumulation of cation vacancies. Upon heating, the first fast-ionic  $\alpha$ -phase appears at 473K. The structure of  $\alpha\text{-Cu}_2\text{HgI}_4$  is similar to that of the  $\beta$ -phase, except that the iodine sublattice is an ideal fcc arrangement. Crystallography displays that  $\alpha\text{-Cu}_2\text{HgI}_4$  is in the cubic space group with each cation site having an average occupancy of  $\frac{1}{2}$  Cu and  $\frac{1}{4}$   $\text{Hg}^{2+}$ . Rietveld refinements of the diffraction data shows the lattice parameters as  $a=6.0672\text{\AA}$  and  $c=12.2266\text{\AA}$  at room temperature<sup>7</sup>. In various solid electrolytes, the method of chemical substitution has been broadly employed to alter the degree of ionic conductivity as well as the transition temperature which separates fast-ionic and covalent phases. The motive of this

paper is to demonstrate our investigation regarding the synthesis as well as the characterization of the  $\text{Rb}^+$  cation thus substituted  $[\text{Cu}_2\text{HgI}_4]$ . In the present investigation, although the primary purpose of this work is to detect the effects of cations as they are introduced in  $\text{Cu}_2\text{HgI}_4$  system, the result is found to be quite difficult to carry out. Instead, it is found that the pure  $\text{Cu}_2\text{HgI}_4$  systems are different from iodide fast ionic system which, in turn, leads to the phase separation structure.

#### Series Of Samples:

e.g. 1)  $[\text{Cu}_2\text{HgI}_4:\text{xRbI}]$  -

- $[\text{Cu}_2\text{HgI}_4]$
- $[\text{Cu}_2\text{HgI}_4:1\text{RbI}]$
- $[\text{Cu}_2\text{HgI}_4:2\text{RbI}]$
- $[\text{Cu}_2\text{HgI}_4:3\text{RbI}]$

$\text{A}_2\text{NI}_4$  (A=Cu; N=Hg) 005A In the  $[\text{A}_2\text{NI}_4]$  where (A=Cu; N=Hg) systems  $[\text{A}_2\text{NI}_4]$  are pure materials, In  $[\text{A}_2\text{NI}_4:\text{xRbI}]$  composite system (where  $x=0.1-0.3$  mol. wt.%, A=Cu; N=Hg),  $[\text{A}_2\text{NI}_4]$  considered as host doped with  $[0.\text{xRbI}]$  (where  $x=0.1-0.3$  mol. wt.%) as the dopant. In all the composite

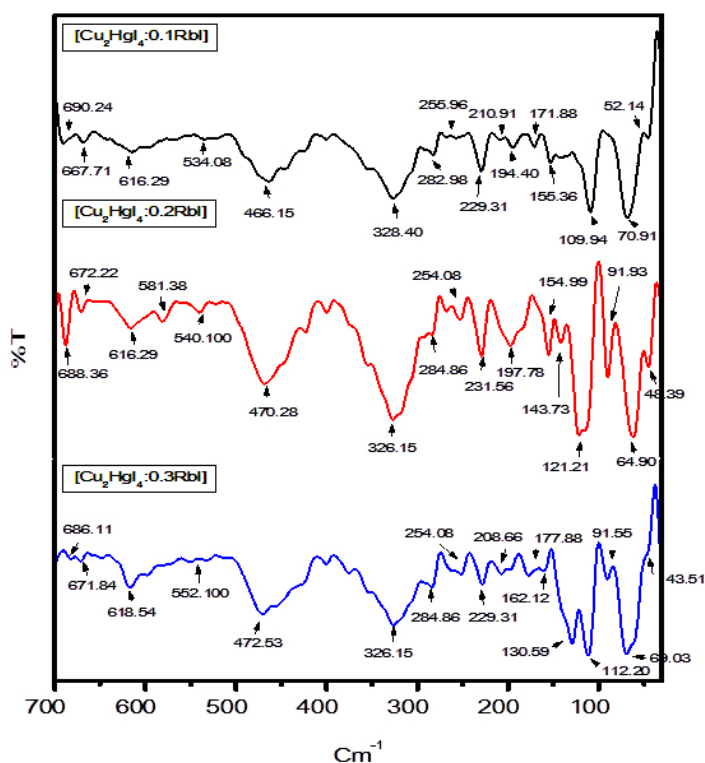


Fig. 1: FAR-IR spectrum for  $[\text{Cu}_2\text{HgI}_4:0.\text{xRbI}]$  fast ionic conductors

samples of  $[A_2Ni_4 \cdot xRbI]$ , the composition of the host  $[A_2Ni_4]$  was kept constant. A significant observation was that a better-quality solid electrolyte composite system can be prepared with the host  $[A_2Ni_4]$  (A=Cu; N=Hg) systems<sup>15</sup>.

### Experimental Procedure

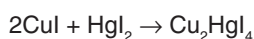
#### Material

The materials were used as received; copper [II] iodide and mercury [II] iodide were of S.d. fine-chem India, and C.D.H. Analar, each of which possessed a purity of 99%, 99% respectively.

#### Preparation and Characterization of Pure and Doped Samples

##### Preparation of Pure $[Cu_2Hgl_4]$ Host Sample

$Cu_2Hgl_4$  was synthesized through carrying out the reaction of a stoichiometric mixture of the component binary halides  $CuI$  and  $Hgl_2$  according to the equation:



Firstly, the powdery raw materials were mixed well. Then the fine pulverized stoichiometric mixture of the binary components was vacuum-packed in a silica crucible and was placed in an air oven

(CE0434NSW-144) at a temperature of approximately 200°C for 24 hours. Finally, the mixture was then cooled rapidly down to room temperature (removal from furnace at 200°C). After cooling, the dark red color changed to maroon.  $Cu_2Hgl_4$  is dark red below 76°C and maroon after 76°C<sup>16</sup>.

##### Preparation of Doped Sample $[Cu_2hgi_4 \cdot 0.Xrbi]$

To form  $[Cu_2Hgl_4 \cdot xRbI]$  composite compound,  $[Cu_2Hgl_4]$  host was doped with RbI in various  $x=0.1-0.3$  mol. wt. % respectively in an agate mortar. It was synthesized through solid state reaction. Silver tetra-iodocadmate 02 mol. wt. %  $[Cu_2Hgl_4]$  were mixed with  $x=0.1-0.3$  mol. wt. % (RbI) dopant solid respectively in an agate mortar, to form  $[Cu_2Hgl_4 \cdot 0.xRbI]$  fast ion conductor at room temperature. They were then heated at 100°C (373K) for 24 hrs in a silica crucible. After intermittent grinding, all the samples were finally prepared<sup>17</sup>.

##### Characterization of $[Cu_2hgi_4 \cdot 0.Xrbi]$ Composite Fast Ion Conductor

Scanning Electron Microscopic (SEM), Energy Dispersive Spectral (EDAX), FTIR spectral analysis were utilized to prepare and investigate the new composite fast ion conductors  $[Cu_2Hgl_4 \cdot 0.xRbI]$ .

Table 1: FAR-IR bands in  $[Cu_2Hgl_4 \cdot 0.xRbI]$  and assignments

Compound	$[Cu_2Hgl_4 \cdot 0.1RbI]$	$[Cu_2Hgl_4 \cdot 0.2RbI]$	$[Cu_2Hgl_4 \cdot 0.3RbI]$	Assignments	nature
CuI	109.94	121.21	112.2	$\omega_e$	Cu-I stretch of the monomer stretching motion of $(CuI)_2$ dimeric molecule bending motion of the $(CuI)_2$ molecule
	70.91	91.93	91.55	$B_{2u}$ and $B_{3v}$	
	52.14	48.39	43.51	$B_{1u}$	
$Hgl_2$	229.31	231.56	229.31	$\nu_3$	symmetric Hg-I stretch of the dimer I- Hg-I bend
	155.36	154.99	162.12	$\nu_2$	
$Cu_2Hgl_4$	328.4	326.15	326.15	-	Dissociation of the $[Cu_2Hgl_4]$ molecule Cu-I stretch $(Hgl_2)_2$ stretch
	466.15	470.28	472.53	-	
	616.29	616.29	618.56	-	
RbI	210.91	197.78	208.66	—	RbI-I stretch of the molecule RbI-I stretch of the molecule
	255.96	254.08	254.08	—	
$Cu_2Hgl_4 \cdot RbI$	534.08	540.1	552.1	—	formation of the $[Cu_2Hgl_4 \cdot 0.xRbI]$ band formation of the $[Cu_2Hgl_4 \cdot 0.xRbI]$ band formation of the $[Cu_2Hgl_4 \cdot 0.xRbI]$ band formation of the $[Cu_2Hgl_4 \cdot 0.xRbI]$ band
	667.71	581.38	671.84	—	
	690.24	672.22	686.11	—	
	—	688.38	—	—	

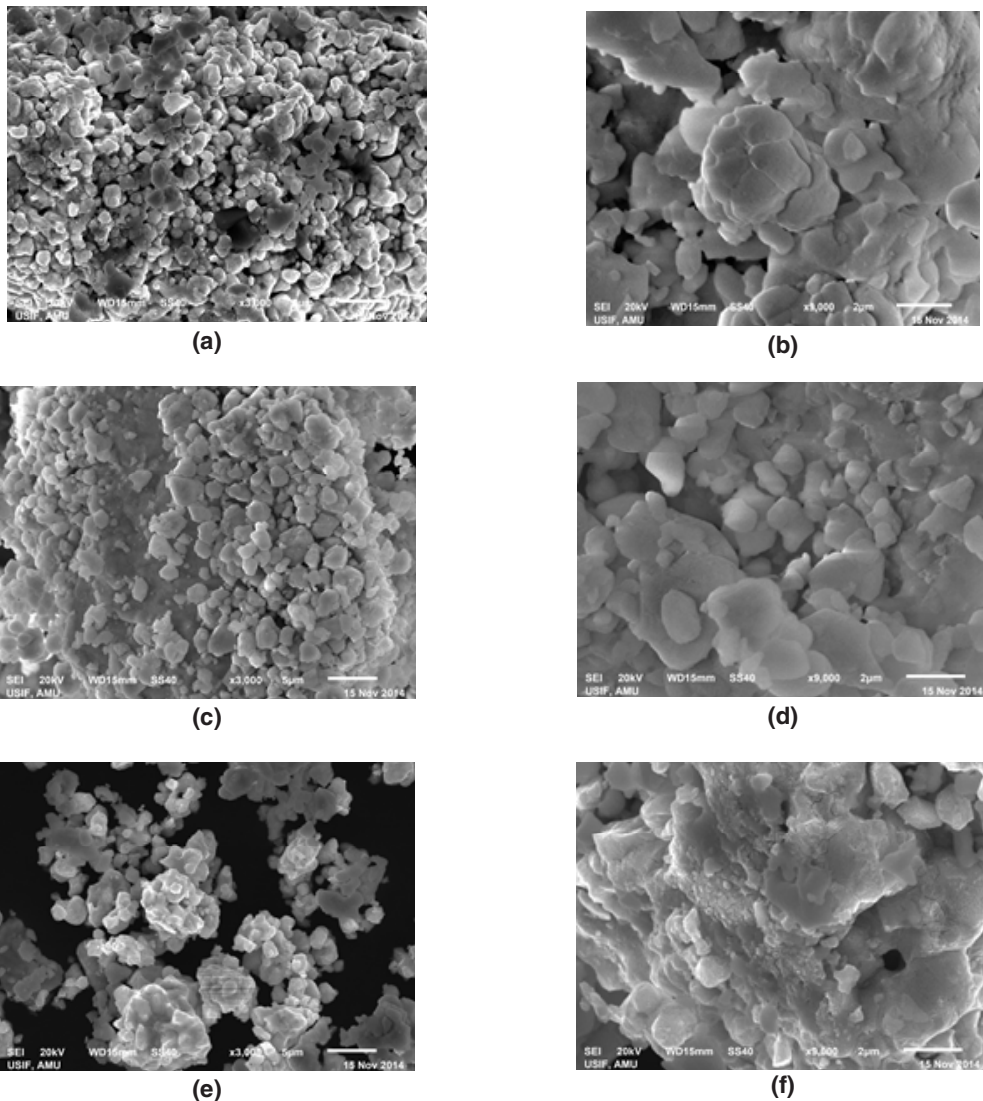
**Scanning Electron Microscopic (Sem) Studies**

The scanning electron microscopic (SEM) studies were conducted to get information regarding samples surface topography and composition of all the fast ionic composite systems  $[\text{Cu}_2\text{HgI}_4:\text{xRbI}]$  (where  $\text{x}=0.1\text{-}0.3$  mol. wt.% respectively). Afterwards, the reaction was accomplished using Jeol JSM 6510LV Scanning Electron Microscope at room

temperature and different magnifications (at 3000x and 10,000x).

**Energy Dispersive Spectral (Edax) Studies**

A chemical analysis of  $[\text{Cu}_2\text{HgI}_4:0.\text{xRbI}]$  (where  $\text{x}=0.1\text{-}0.3$  mol. wt. %) was conducted. The processed samples were carried out by EDAX to determine if there is any deviation from the initial composition.



**Fig. 3: SEM photographs of (a)  $[\text{Cu}_2\text{HgI}_4:0.1\text{RbI}]$  at 3000x and (b) at 10,000 x, (c)  $[\text{Cu}_2\text{HgI}_4:0.2\text{RbI}]$  at 3000x and (d) at 10,000 x, (e)  $[\text{Cu}_2\text{HgI}_4:0.3\text{RbI}]$  at 3000x and (f) at 10,000 x respectively. The particles are seen in a highly agglomerated state. The typical agglomerated particle size is in the range 0.825–1.164  $\mu\text{m}$ .**

Fig. 4 shows a typical EDAX result for  $[\text{Cu}_2\text{HgI}_4 \cdot 0. \text{xRbI}]$  sample.

### Far-IR Spectral Analysis

The Far-IR spectrum was recorded for all the fast ionic composite systems  $[\text{Cu}_2\text{HgI}_4 \cdot \text{xRbI}]$  (where  $\text{x} = 0.1\text{-}0.3$  mol. wt. %) in the far-infrared range  $30\text{-}400 \text{ cm}^{-1}$  at room temperature using a Perkin Elmer/FTIR Spectrometer measured in KBr.

## Results and Discussion

### FTIR analysis

#### Far-IR Discussion In $[\text{Cu}_2\text{HgI}_4 \cdot 0. \text{xRbI}]$

Figure 1 shows IR spectrum of the vapour over solid  $[\text{Cu}_2\text{HgI}_4 \cdot 0. \text{xRbI}]$  (where  $\text{x} = 0.1\text{-}0.3$  mol. wt. %) in the  $30\text{-}700 \text{ cm}^{-1}$  region at room temperature.

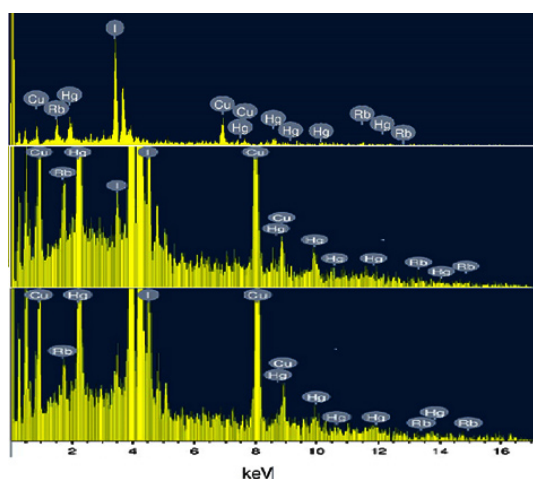
The IR spectrum of  $[\text{Cu}_2\text{HgI}_4 \cdot 0. \text{xRbI}]$  (where  $\text{x} = 0.1\text{-}0.3$  mol. wt. %) solid at room temperature showed two distinct absorption bands. The fundamental Cu-I stretching frequency of the monomer is assigned to the absorption around  $109.94 \text{ cm}^{-1}$ ,  $121.21 \text{ cm}^{-1}$ ,  $112.20 \text{ cm}^{-1}$  in  $\text{x} = 0.1\text{-}0.3$  mol. wt. % which shows typical PR band structure of a diatomic molecule. The  $(\text{CuI})_2$  dimeric molecule can be assigned to the band origin at  $70.91 \text{ cm}^{-1}$ ,  $91.93 \text{ cm}^{-1}$  and  $91.55 \text{ cm}^{-1}$  in  $\text{x} = 0.1\text{-}0.3$  mol. wt. % respectively<sup>24</sup>. Dimeric molecules are present in a considerable amount in the vapour formed over CuI at the temperature used in the present experiments by IR-measurements.

were present, which were at  $534.08$ ,  $667.71$ ,  $690.24 \text{ cm}^{-1}$ , in  $\text{x} = 0.1$ ,  $540.10$ ,  $581.38$ ,  $672.22$ ,  $688.38 \text{ cm}^{-1}$  in  $\text{x} = 0.2$ ,  $552.10$ ,  $671.84$  and  $686.11 \text{ cm}^{-1}$  in  $\text{x} = 0.3$  mol. wt. %. These absorption bands are in excellent agreement with those of the  $[\text{Cu}_2\text{HgI}_4]$  host composite. Another additional peaks that might indicate the presence of RbI species is at  $210.91$ ,  $255.96 \text{ cm}^{-1}$  in  $\text{x} = 0.1$ ,  $197.78$ ,  $254.08 \text{ cm}^{-1}$  in  $\text{x} = 0.2$ ,  $208.66$ ,  $254.08 \text{ cm}^{-1}$  in  $\text{x} = 0.3$  mol. wt. % have been observed shows Rb-I stretch of the molecule.

These results show that the successive release of  $\text{HgI}_2$  and  $\text{CuI}/(\text{HgI}_2)_2$  vapour species occurs during heating, thus indicating the dissociation of  $[\text{Cu}_2\text{HgI}_4 \cdot 0. \text{xRbI}]$  (where  $\text{x} = 0.1\text{-}0.3$  mol. wt. %) molecules under the conditions of the experiments (Table 1). Additional peaks that might indicate the presence of Cu-Hg-I species have been observed.

### Scanning Electron Microscopic (Sem) Studies

The scanning electron microscopic (SEM) studies were conducted to gain insight about samples surface topography and composition of all the fast ionic composite systems  $[\text{Cu}_2\text{HgI}_4 \cdot \text{xRbI}]$  (where  $\text{x} = 0.1\text{-}0.3$  mol. wt. %) after the reaction was completed (sintered) at room temperature. Fig.3 shows typical results at two different magnifications (at  $3000\times$  and  $10,000\times$ ), that confirms the highly accumulated nature of particles. The average size of accumulated particles is  $0.825\text{-}1.164 \mu\text{m}$ . It is worth noting here that the accumulation hence achieved is more evident at higher magnification. (Fig. 4b). Moreover, there is a major possibility of existence of nanosize amorphous particles. The sintering temperature of all the samples cannot be high enough to synthesize the pure phase of monoclinic  $[\text{Cu}_2\text{HgI}_4 \cdot \text{xRbI}]$  ( $\text{x} = 0.1\text{-}0.3$  mol. wt. %) sample as indicated by FTIR. The SEM images shown in Fig.3 suggest that the particles of all the samples are covered with a slice of RbI, sintered at a particular temperature. These slices are attributed to the residual RbI particles left over from the solid state reaction method<sup>24</sup>. The residual RbI particles can favor stabilization of  $\text{Hg}_2^+$  and facilitate the diffusion of  $\text{Cu}^+$ . As it is evident, it is the result of  $\text{Cu}_2\text{HgI}_4$  decomposition during preheating and sintering process, which then produces a composite powder



**Fig.4: EDAX results for  $[\text{Cu}_2\text{HgI}_4 \cdot \text{xRbI}]$  sample. Showing the presence of elements (Cu, Hg, Rb, I). The inset shows weight/atomic ratios of the four elements present in the sample are same as that in the unprocessed sample.**

[Cu<sub>2</sub>Hgl<sub>4</sub>:xRbl] (x=0.1-0.3 mol. wt. %) with superior conductivity<sup>25</sup>.

However, the particular temperature (100 °C) preparation causes an abrupt growth of particles with a smooth surface particle of ≈0.825–1.164. It has been reported that the morphology and surface area of the obtained particles bear a notable effect on the electrochemical performance of [Cu<sub>2</sub>Hgl<sub>4</sub>:xRbl] (x=0.1-0.3 mol. wt. %). Optimizing particle size or introducing conducting additives can improve the performance, as nanometer size of Rbl particles can be dispersed uniformly between [Cu<sub>2</sub>Hgl<sub>4</sub>] particles. It proves to be advantageous when it comes to attaining samples bearing small and uniform particle size as well as enhancing the general conductivity of the sample. Furthermore, the solid state reaction method can check agglomeration of the particles.

#### EDAX

EDAX analysis of [Cu<sub>2</sub>Hgl<sub>4</sub>:0.xRbl] (where x=0.1-0.3 mol. wt.%) processed samples was conducted by EDAX to determine whether there is a aberration from the initial composition or not. In a [Cu<sub>2</sub>Hgl<sub>4</sub>:0.xRbl] (where x=0.1- 0.3 mol. wt.%) samples shows a typical EDAX result, the wt.% of the four elements (Cu= 19.21%, Hg= 10.88%, Rb= 6.79%, I= 63.13%) in a [Cu<sub>2</sub>Hgl<sub>4</sub>:0.1Rbl], whereas the wt.% of the four elements (Cu= 23.05%, Hg= 12.75%,

Rb= 1.75%, I= 62.45%) and (Cu=27.27%, Hg= 8.45%, Rb= 0.95%, I= 63.32%) in a x= 0.2 and 0.3 respectively, is nearly same as in the unprocessed (Cu= 20%, Hg= 13%, Rb= 7%, I= 65%) material. Therefore, it can be concluded that the composition of the samples is not modified by the formation of all the fast ion conductors by solid state reaction<sup>27,28</sup>.

#### Conclusion

A novel composite superionic systems [Cu<sub>2</sub>Hgl<sub>4</sub>:0xRbl] (where x =0.1- 0.3 mol. wt.%), were investigated. An alternative ternary system [Cu<sub>2</sub>Hgl<sub>4</sub>] was used as host. In the host [Cu<sub>2</sub>Hgl<sub>4</sub>] structure, doping Rb<sup>+</sup> in the host was found to prompt a decrease in the mobile charge carriers, Rb<sup>+</sup> ions, which are in proportion with the increased 0.x mol. wt.% (x= 0.1-0.3 mol. wt.%) in the host mixed system.

The formation of a superionic phase in the composite system was confirmed by several FAR-IR, SEM and EDX studies.

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