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Growth of L-Valinium Aluminium Chloride single crystal for OLED and super-capacitor applications



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ABSTRACT

L-Valinium Aluminium Chloride (LVAC), a novel semi-organic material, was grown using slow evaporation under isothermal condition. The single crystal data reveal that the grown crystal belongs to monoclinic system. The SEM micrographs give clear picture about the surface morphology. Further, they confirm the inclusion of aluminium chloride into atomic sites of L-Valine. The compositional elements present in the crystal were identified through EDAX analysis. The mass spectral analysis was carried out to determine the molecular weight of the grown crystal. The optical transparency of the grown crystal was investigated by UV-vis-NIR spectrum. FTIR spectral study was used to identify the functional groups present in the grown material. The luminescence characteristics of grown material were analysed to confirm the effect of metal ion on the ligand. This property makes the material suitable for OLED application. The supercapacitive performance of the grown crystal was finally studied using cyclic voltammetry.

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1. Introduction

The ever-growing demands for the high quality NLO materials have led to tremendous research effort for the development of advanced semi-organic NLO materials. Those new class of materials intensely find extensive applications in optical communication. optoelectronic and super-capacitance, etc. [1-3]. L-Valine, a branched chain aminoacid, has been exploited for the formation of salts with inorganic acids which exhibit enhanced physiochemical properties [4]. Among all the metal dopants, aluminium chloride hexahydrate was chosen because of its cost effectiveness, abundant availability, ease of handling, good reactivity, experimental simplicity, capacitance capability and excellent solubility in water. The addition of metallic compound serves as a promising green reagent and catalyst during the synthesis process [5]. So far, no work has been reported on this material LVAC. However, work on some other compounds: L-Valinium picrate, L-Valinium monohydrochloride, L-Valine hydrobromide, L-Valine cadmium acetate, etc., have been reported [6-9]. In the present study, we report the growth and important characterization of the compound LVAC for OLED and super-capacitor applications.

2. Experimental method

High purity chemicals purchased from Merck were used for synthesising LVAC. L-Valine and aluminium chloride hexahydrate were dissolved in double distilled water in equimolar ratio and stirred at 70 °C for about 24 h in order to attain homogeneity throughout the solution. The obtained homogeneous solution containing pH 4.2 was filtered using Whatman paper and left free for slow evaporation to achieve supersaturation. The supersaturation of the solution leads to nucleation followed by growth. After a period of 45 days, good quality crystals of dimensions $7 \times 7 \times 2 \text{ mm}^3$ were obtained. The reaction scheme for the synthesising the material is given as follows: Fig. 1 shows the photograph of the as-grown LVAC single crystal.

$$C_5H_{11}NO_2 + \hspace{0.1cm}AlCl_3 \cdot 6H_2O \rightarrow C_5H_{11}NO_2 \cdot AlCl_3 + 6H_2O \downarrow$$

3. Result and discussion

3.1. Single crystal XRD

The grown crystal was subjected to single crystal XRD study to determine the crystal system with lattice parameters. Single crystal X-ray diffraction studies were carried out for the L-Valinium Aluminium Chloride employing ENARF NONIUS CAD4 X-ray diffractrometer. The unit cell parameters of L-Valinium Aluminium

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Fig. 1. As-grown LVAC single crystal.

Chloride are found to be $a = 11.23 \,\text{Å}$, $b = 5.17 \,\text{Å}$, $c = 12.89 \,\text{Å}$, $\beta = 111.46^0$ and hence, the crystal belongs to monoclinic system. The single crystal XRD result shows that LVAC crystal possesses cell parameters different from those of pure L-Valine. However, the crystal system remains the same. The deviation in the cell parameters clearly confirms the incorporation of aluminium chloride into the host lattice of L-Valine [10].

3.2. SEM

The morphologies of the micro structured L-Valinium Aluminium Chloride recorded by SEM with 5 μ m and 20 μ m resolutions are shown in Fig. 2(a) and (b) respectively. Fig. 2(a) clearly depicts the fairly smooth surface of the grown crystal along with

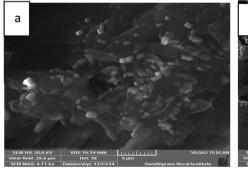
almost cubic microcrystals adhered to the base material L-Valine. Fig. 2(b) exhibits layers of wafers with bright shiny creeks which is due to the inclusion of brittle natured aluminium chloride with striations in the grown crystal. The micrograph image shown in Fig. 2(b) also shows 3-D frame work structure. The presence of 3-D frame work structure could lead to high electrochemical activity for super-capacitor application [11]. Of course, at certain regions, 3-D frame work has been destroyed due to localized thermal stress developed in the material.

3.3. EDAX

Fig. 3 shows the EDAX spectrum recorded for the grown crystal. The observed peaks clearly indicate the presence of constituent elements C, N, O, Al and Cl (except lighter element hydrogen) in the grown material.

3.4. Mass spectrum

The mass spectrum of L-Valinium Aluminium Chloride recorded in the range of 52–270 amu is shown in Fig. 4. In general, the ion with largest mass is often parent molecular ion without any fragmentations [12]. Hence, the peak against the largest mass 17.9 m/z corresponds to the parent molecule L-Valine. The exact mass of the LVAC molecular ion is observed as 255.74 (m/z) which is found to be closer to the calculated value 250.486 (m/z) (Table 1). The other peaks having smaller mass may be due to charged fragments of the ion. Thus, we precisely confirm the formation of the title compound.



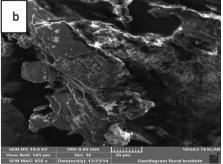


Fig. 2. SEM micrographs of LVAC.

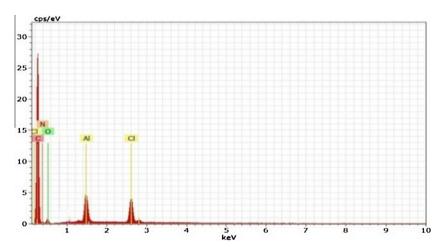


Fig. 3. EDAX spectrum of LVAC.

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