Growth, Morphology and Spectral Study of Gel Grown Copper Iodate Crystals

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Abstract

Copper iodate crystals are successfully grown in silica gel. The grown crystals are opaque having surf green color. The crystals have been characterized by FESEM, EDAX and FT–IR spectroscopy. A FESEM image shows that crystals are grown by layer deposition. EDAX study reveals the presence of copper, iodine and oxygen in grown crystal. FTIR spectrum clearly indicates that the iodate group is present in the copper iodate crystal.

Keywords: Gel method, FESEM, EDAX, FTIR.

Introduction

The solids are characterized by nearly perfect periodicity of atomic structure. The geometric regularity of atomic structure provides a simple picture of a crystal and helps a lot in gaining the knowledge of physical properties of the solid [1- 3]. In a crystalline solid, the atoms are arranged in a regular manner, i.e. direction of the crystal. The crystalline solid has directional properties, which are also called isotropic or anisotropic substances accordingly [4 - 6]. It is fact that number of important properties of solid material is understood when it is in the form of crystals. Crystals are proved to be the pillars of modern electronic and other industries. The supply of natural crystals is limited, at the same time, new materials or available materials in the modified form with crystalline form are required for modern development. The crystal can be grown by various transformations. The gel technique is one of the solution techniques and it is simply and can be used at room temperature or near the room temperature. It offers a good alternative for the growth of crystals of the material which are relatively insoluble or sparingly soluble in water [7]. which decompose before their melting points and do not have proper solvents. In fact gel act as 3- dimensional crucible which support the crystal and the same time yields to its growth without exerting major forces on it. Defect free which are not very large but appreciable size of crystal can be grown. Growth mechanism of crystal can be directly observed. Nucleation can be controlled by concentration programming and by using dummy gel too.

In the present work, single crystals of copper iodate [Cu(IO₃)₂] were grown by gel technique by single diffusion method and characterized by FESEM, EDAX and FT–IR spectroscopy.
Materials and Methods

The straight tube diffusion method was employed to grow copper iodate crystals in the gel medium. Silica gel was prepared by mixing Sodium Meta Silicate (SMS) of specific gravity of 1.04 gm/cc with 2N acetic acid. The inner reactant 0.1M Cu(NO₃)₂ was incorporated into the gel. This solution was then transferred to borosil glass tubes of 2.5 cm diameter and 15 cm length. After setting the gel, the second reactant, KIO₃ solution of concentration of 0.1M was slowly poured without disturbing the gel surface. Crystals are formed after 4-5 weeks onwards. The characteristic habit of the gel grown crystals is seen in the photograph of the grown crystals of copper iodate [see Fig. 1(a, b)]. The conditions for the growth of good quality crystals are optimized.

Fig.1 (a). copper iodate crystals in gel.                   Fig. 1 (b). As grown copper iodate crystals.

FESEM Analysis

In the present work powdered sample of copper iodate crystals was examined by using FESEM technique at the Institute of Chemical Technology, North Maharashtra University, Jalgaon. The study of the surface
of the crystal gives valuable information about its internal structure. Fig. 2 illustrates FESEM photographs of single crystals of copper iodate crystal. It shows plate like crystal morphology. These crystals are grown by layer deposition. Thick and thin layers are seen in figure.

![Fig. 2. FESEM images of copper iodate crystal.](image)

**Energy Dispersive X-Ray Spectroscopy (EDAX)**

Fig. 3 shows an EDAX spectrum of crystals of copper iodate. The peaks show the presence of copper, iodine, oxygen in the grown crystals. Table 1 shows the elemental and atomic percentage of the elements Cu, I, and O grown crystals. From the table, it was found that the weight % and atomic % of iodine is little low, it may due to the formation of white precipitate layer at the gel interface at the instant of pouring the upper reactant (KIO₃) and just below the precipitate layer concentration gradient goes on decreasing.

![Fig.3. Energy dispersive spectrum of copper iodate crystal.](image)
Table 1 Elemental composition of copper iodate crystal.

<table>
<thead>
<tr>
<th>Elements</th>
<th>Experimental values</th>
<th>Theoretical values</th>
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<tbody>
<tr>
<td></td>
<td>Mass[%]</td>
<td>Atomic[%]</td>
</tr>
<tr>
<td>I</td>
<td>62.24</td>
<td>25.21</td>
</tr>
<tr>
<td>Cu</td>
<td>18.45</td>
<td>13.65</td>
</tr>
<tr>
<td>O</td>
<td>19.31</td>
<td>61.14</td>
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</tbody>
</table>

FT-IR Analysis

The FT-IR spectrum of copper iodate crystals are shown in fig. 4. The FTIR spectrum of lead iodate crystals is shown in Fig. 4. The FT-IR spectrum shows strong band in the region 500-830 cm\(^{-1}\) indicating the presence of iodate and metal oxide [8-12]. Fundamental infrared frequencies, observed in all iodate compounds in general, are also found in present FT–IR analysis, which confirm the iodate group of grown crystals. Fundamental frequencies that have been observed are \(\nu_1\) at 719.47 cm\(^{-1}\) is due to O - Pb - O symmetric stretching and \(\nu_3\) at 771.55 cm\(^{-1}\) is due to the O – Pb - O asymmetric stretching. The dominant absorption bands are found at 700 – 800 cm\(^{-1}\) in all iodate compounds, and can be expected to contain \(\nu_1\) and \(\nu_3\) as well as possible splitting of \(\nu_3\). The FT-IR spectrum does not give any proof of the presence of combined water molecule in the copper iodate crystals.

Fig.4. The FT-IR spectrum of copper iodate.
Conclusions
The growth of copper iodate crystals was carried out by gel-aided solution technique. FESEM images shows plate like crystal morphology. Elemental analysis shows the presence of copper, iodine, oxygen in the grown crystals. The absence of water molecules in to the copper iodate crystals was confirmed by the FT-IR and thermal analysis.

Acknowledgement
The authors are much indebted to authorities of Institute of chemical technology, N. M. U. Jalgaon for help in FE-SEM, EDAX and FTIR analysis. One of the authors (KDG) is thankful to Dr. N. O. Girase, Principal, S.V.S’s Dadasaheb Rawal College, Dondaicha for his inspired suggestions.

References: