Preparation of R-methyl Imidazolium-Sodium Hexaflorosilicate Complex Crystals

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Room temperature ionic liquids (IL) are by definition ionic compounds - salts, which are liquid below 100 ^{o}C. Most applicable ionic liquids are based on imidazolium or quaternary ammonium cations with the BF_4^- anion. Properties such as inertness to moisture and oxygen, poor coordination ability and a weak ligation makes BF_4^- a superior anion for ionic liquids. During recent years investigations on stability of ionic liquids in combination with different solvents have been increased and this may lead to inappropriate experimental design and utilization of these chemicals.

Sodium hexafluorosilicate is an odorless, white crystalline powder of hexagonal structure which has widespread use in water fluoridation. The interest on pure Na_2SiF_6 has been increased from this decade due to the similar refractive index with the natural ice crystals in visible light. Crystallization behaviour of sodium hexafluorosilicate is not well described and by our knowledge the crystallization in presence of ionic liquids is not mentioned at all.

The regular complex crystals of Na_2SiF_6 have been obtained in the aqueous solutions of different methylalkylimidazolium (ethyl-, butyl- and decyl-) tetrafluoroborate ionic liquids. (Fig.1.) It is demonstrated that sodium hexafluorosilicate crystalline compounds with good regularity and narrow size distribution containing dialkyl imidazolium ions between the nano hexagonal crystalline clusters interconnected to each other to a whole hexagonal aggregate can be obtained in large quantities. This characteristic phenomenon of crystallization of ionic liquids containing BF_4^- ions is reported for the first time. The mechanism of formation of various [RMIm]BF_4-Na_2SiF_6 microcrystalline morphologies and the influence of temperature on growth kinetics are discussed. Crystallographic studies of the product were carried out by X-ray diffractometer (XRD), characterization by scanning electron microscopy (SEM) and optical microscopy; also infrared spectra (IR) were recorded. Thermal analyses were performed by differential scanning calorimetry-thermogravimetric analyser (TGA-DSC). Presence of ionic liquid cations was confirmed by high resolution mass spectrometry (HRMS).

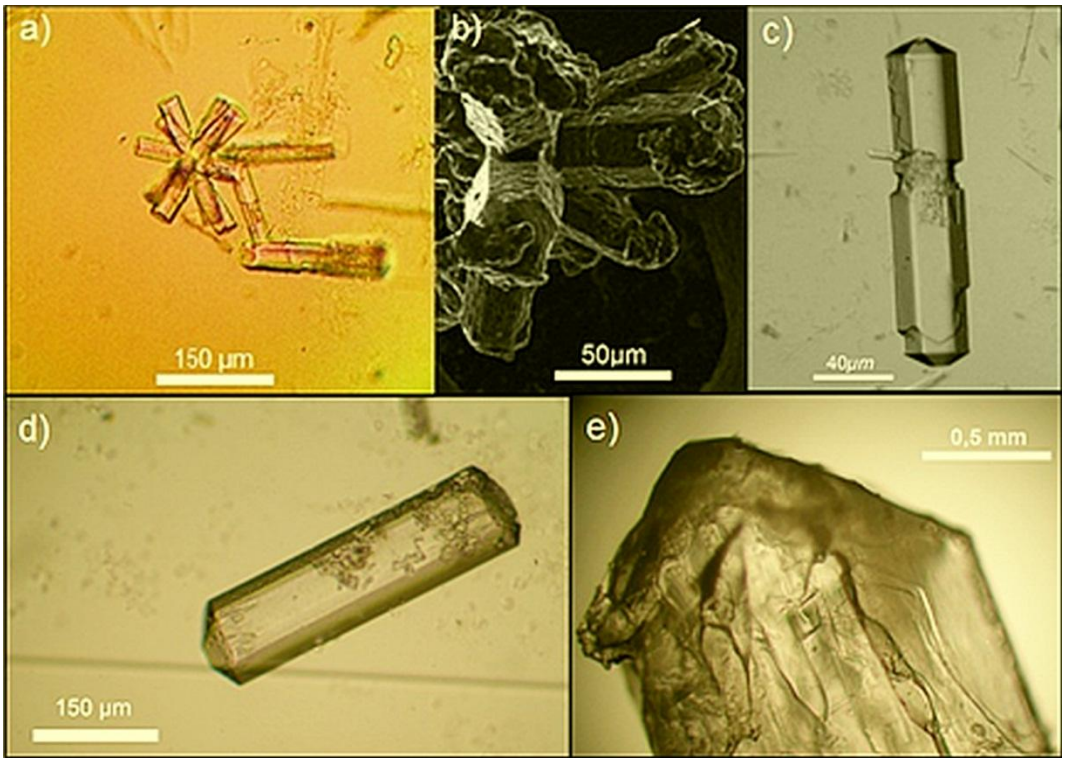

Fig. 1: Optical and SEM images of different shape and size crystal structures: a), b), c) - [EMIm]BF_4-Na_2SiF_6 microcrystals; d) [BMIm]BF_4-Na_2SiF_6 microcrystal - synthesized at room temperature in low concentrated ionic liquid aqueous solutions; e) optical image of [EMIm]BF_4-Na_2SiF_6 macrocrystal, synthesized at 50^{o}C in concentrated ionic liquid aqueous solution.

Described cluster structures can have several potential applications in different fields due to their nano comb-clustered complex structure. The developed, method allows easy production of Na_2SiF_6 structures with a narrow size distribution. There is a hypothetical possibility that these two-component systems have variable refractive index. The presence of conductive ionic liquid between the crystalline layers makes them attractive in fields like optics where they may be applicable as conductive sensors.