Determination of the deliquence point in salt mixtures by utilizing the dynatic vapour sorption method

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ABSTRACT

Background and objectives
Salts are generally recognized to be one of the main courses for deterioration of porous materials. The damage and eventual removal of the salt is dependent on whether the salt is crystallized or dissolved. In relation to desalination of church vaults for future preservation of the murals it is of interest to limit the applied liquid during the desalination e.g. to prevent drying cracks and creating conditions for microbial growth [1]. At present climate chambers with a specific temperature and relative humidity are used to prevent salt induced deterioration and also in this relation it is of interest to find the deliquescence point since the relative humidity can be kept on the most effective level without causing damage. The deliquescence point for single salts is well known; however, in case of salt mixtures the combinations are comprehensive. In order to find deliquescence points for specific salt mixtures different simulation programs have been used as e.g. RUNSALT [2], ECOS [3], however several assumptions have to be made and therefore these programs are by several considered as limited reliable. In [4] phase diagram for the Na₂SO₄·H₂O system was calculated on basis of thermodynamic data as a function of temperature. In [5] a model approach was made to predict phase equilibria in case of salt mixtures and safe ranges of environmental conditions taking temperature, varying ions, moisture contents and activities into account. The model was based on the semi-empirical model from Pitzer where some parameters were experimental determined. The results were given in RH as a function of temperature. In order to understand the fundamental mechanisms such a model as deduced in [5] is essential. The need for incorporating the transport properties of the porous material and dissolution and crystallization kinetic of the various salts is mentioned to be strongly depended to the transport properties of the various salts in reality [5]. Measurements of the deliquescence point and the kinetics for some specific salt mixtures in materials at a specific temperature would further contribute to the understanding of salt mixtures and this is the content of the present work by utilizing dynamic vapor sorption method (DVS).

The Dynamic vapor sorption method
The deliquescence point can empirically be found by producing water vapor sorption isotherms which previously were found by time and labor intensive desiccators and saturated salt solutions. A further development of the original water vapor sorption isotherm method is the dynamic vapor sorption method (DVS). The DVS method is a gravimetric technique measuring the velocity and the extent to which a solvent is absorbed by a sample. In a DVS experiment this is accomplished by exposing a sample to a series of step changes in relative humidity and monitoring the mass change as a function of time. The sample mass must be allowed to reach gravimetric equilibrium at each step change in humidity before progressing to the next humidity level. Then, the equilibrium mass values at each relative humidity step are used to generate the isotherm. The DVS method has previously been used for identifying a stable crystalline salt in order to allow clinical development activities. During this work a hysteresis effect was measured when exposed to high humidity’s which they propose was caused by a change from an anhydrous salt to a more hydrated salt [6].

Expected results
The DVS will be used to determine the sorption isotherm for NaCl (for comparison with reference values), Na₂SO₄·H₂O (for comparison with [4]) and salt mixtures taken from three different positions from the vault of Rørby Church, Denmark (to be used during future desalination). On basis of the results which are to be measured in August 2012, it will be discussed whether satisfactory results can be obtained with the DVS method for determination of the deliquence point in relation to salt mixtures at a specific temperature.

It has to be discussed whether it is of interest to determine a precise deliquence point of a salt mixture in practice or whether the deviation between the three taken samples in Rørby church makes such a precise determination meaningless.

Also in case of establishing a climate chamber to prevent further salt induced deterioration a RH could be chosen to the climate chamber on basis of determination of the deliquence point in a limited temperature intervals. Since temperatures are most often recorded in advance to desalination the deliquence point at one specific temperature would give valuable information.

In a previous work the known value for the deliquence point of the single salt NaCl was confirmed by very primitive measurements in a microclimate and it was shown that in case of dissolved ions (and possibly a limited contribution from the compress plaster) a high desalination efficiency was obtained by use of a new electrokinetic method [7-9].

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References