

Preventive Conservation of Salt-Contaminated Ceramics :

Establishing Safe Humidity Range Through Analysis of Crystallisation-Dissolution Behaviour of Salt Mixtures

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Introduction

This study aims to establish a safe relative humidity (RH) range for a salt-contaminated ceramic pillow in a museum to prevent it from further salt damages. Hygroscopic salts undergoing repeated crystallisation–dissolution cycles in response to RH fluctuations damages ceramics with porosity in nature by generating internal stress that leads to cracking, glaze flaking, and structural deterioration.

Conventional desalination methods, such as soaking or poulticing, would be too risky for this ceramic pillow due to its physical fragility and existing damages (Fig. 1). Preventive conservation to stabilise its condition through RH control is therefore critical to limit the re-occurrence of these salt phase transitions.

Three approaches were applied to identify the optimal storage RH thresholds, namely: (1) Literature-based inference, (2) Thermodynamic modelling (ECOS/RUNSALT), and (3) Dynamic vapour sorption (DVS). Their effectiveness and practicality are evaluated to support conservators in selecting appropriate methods in the future for determining precise RH thresholds for long-term preservation.



Fig.1 Condition of the ceramic pillow

Analytical Characterisation of Salts

Loose crystalline substance was sampled from the interior surface of the ceramic pillow.

1) X-Ray Diffraction (XRD) identified crystalline phases and their relative abundances. The salt types and crystalline structures identified by XRD were used for literature-based inference.

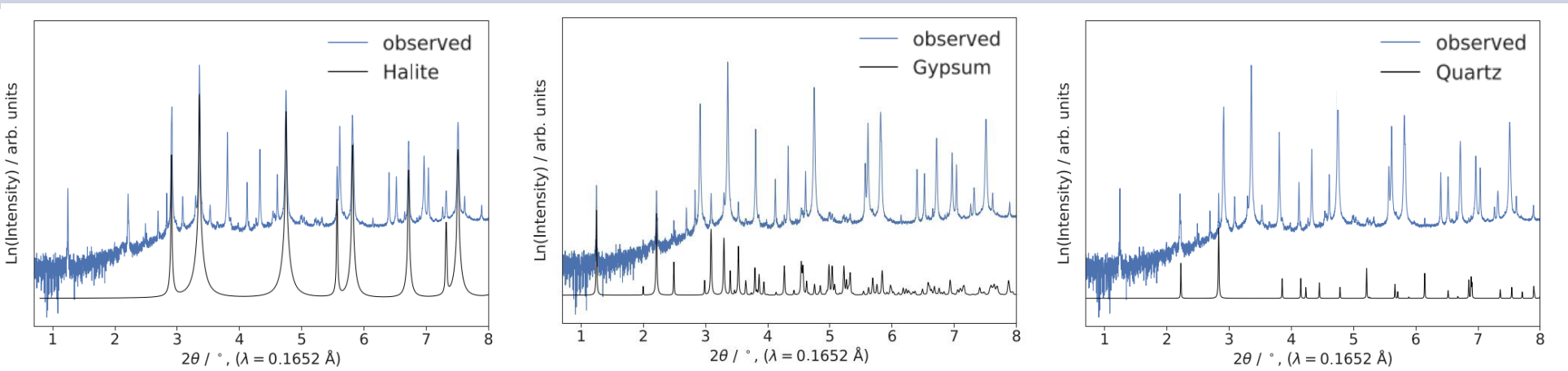


Fig.2 Comparison of simulated phases of the salt sample in log scale, from left to right: Halite, Gypsum, and Quartz

Phase	Composition	Wt. %
Halite	NaCl	97.2%
Gypsum	CaSO ₄ · 2H ₂ O	2.02%
Quartz	SiO ₂	0.47%

Table 1 Identified phases and composition of the salt sample by XRD

2) Ion Chromatography (IC) quantified soluble ions, providing compositional and concentration data. The salt concentrations and ionic species measured by ion chromatography were used for thermodynamic modelling using ECOS/RUNSALT.

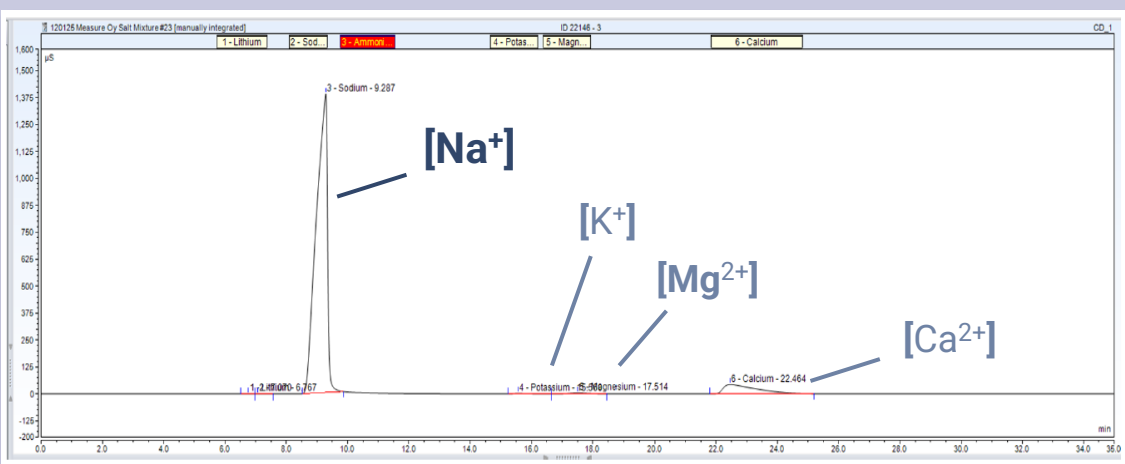


Fig.3 Chromatogram of cations identified from the salt sample

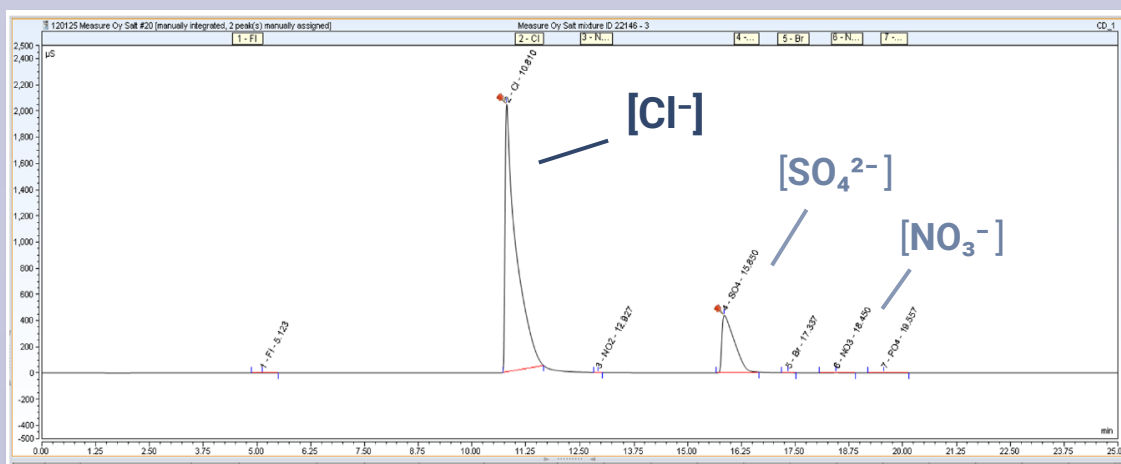


Fig.4 Chromatogram of anions identified from the salt sample

3) Scanning Electron Microscopy – Energy Dispersive X-ray Spectroscopy (SEM-EDS) characterised morphology and elemental composition, confirming co-localisation of NaCl, CaSO₄·2H₂O and SiO₂ with their associated atoms. These elemental profiles supported salt identification for literature-based inference.

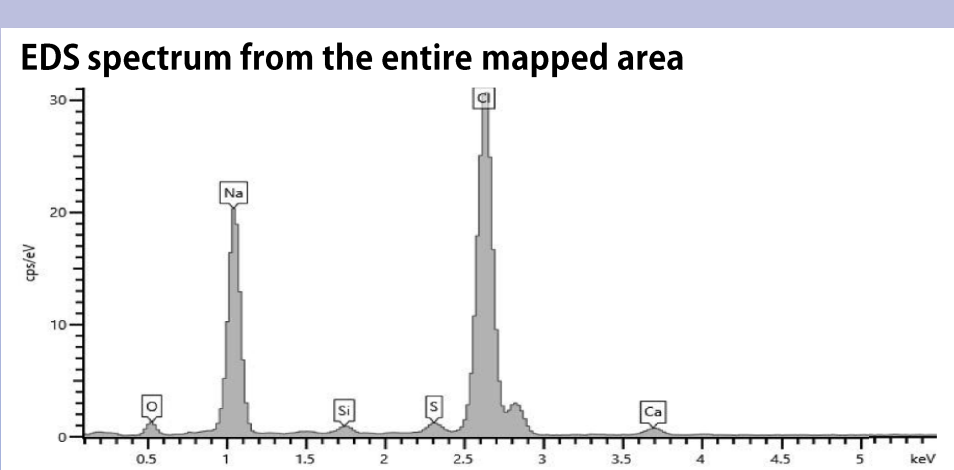


Fig.5 EDS spectrum from the entire mapped region

	Atomic %
Cl	52.5
Na	32.9
O	10.4
Ca	1.7
S	1.4
Si	1.0

Table 2 Elemental composition of the salt sample

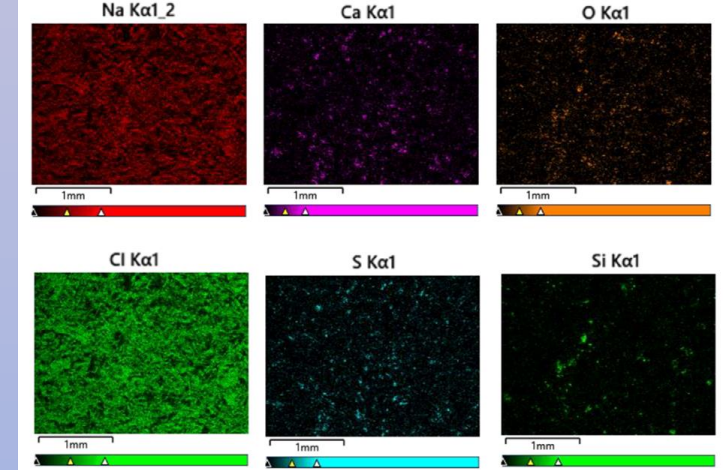


Fig.6 EDS elemental maps with individual elemental distribution

4) Fourier Transform Infrared (FTIR) Spectroscopy & Raman Spectroscopy provided molecular confirmation of CaSO₄·2H₂O, further supporting salt identification for literature-based inference.

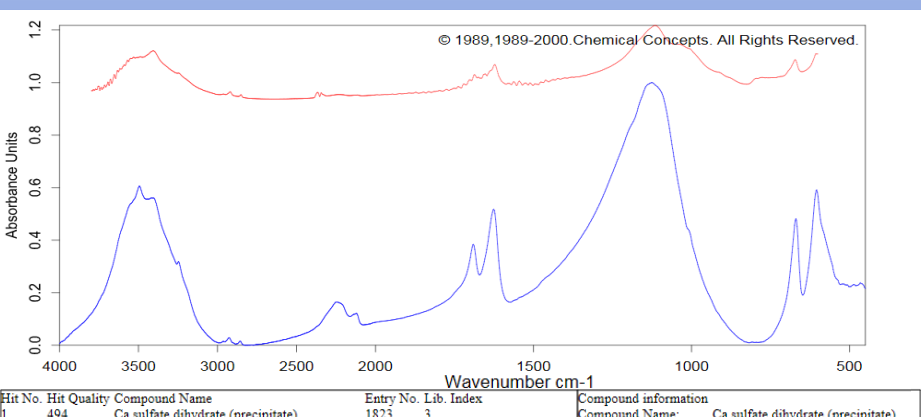


Fig.7 FTIR spectrum of the sample, confirming presence of CaSO₄·2H₂O

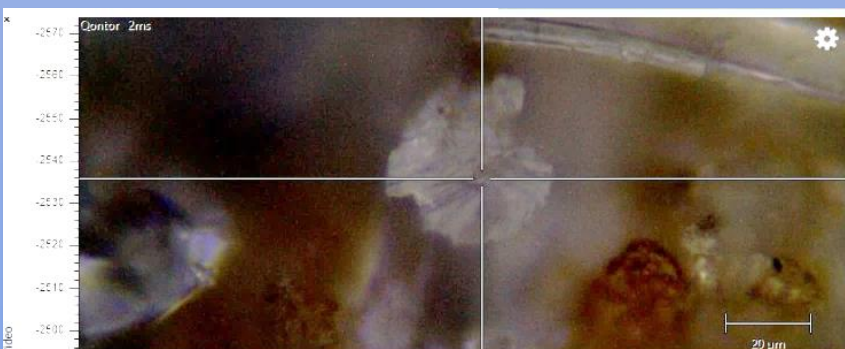


Fig.8a Raman spectroscopy measurement point on the sample

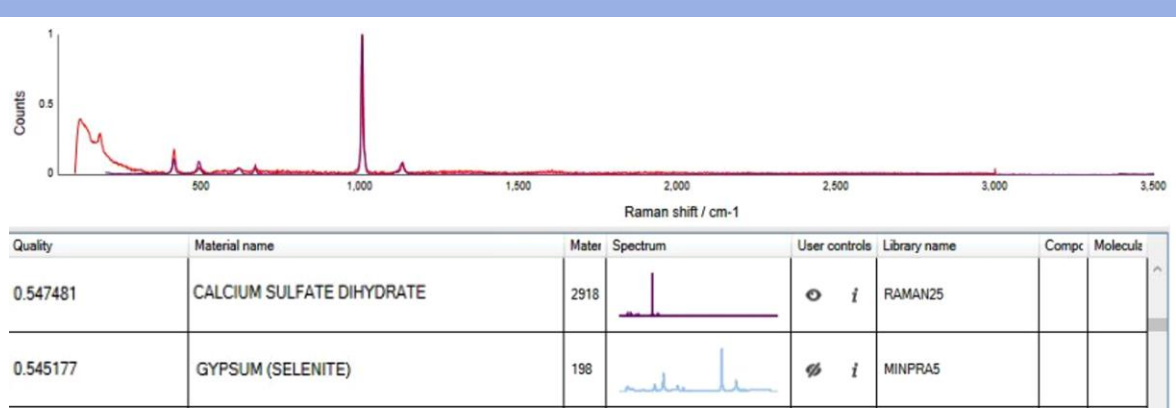


Fig.8b Raman spectrum of the sample, confirming presence of CaSO₄·2H₂O

Methodologies to Determine Safe RH Range

As the temperature in our museum collection stores and display areas is normally maintained between 20°C and 25°C, the analyses were conducted at both 20°C and 25°C.

1) Literature-Based Inference

Published data were used to infer crystallisation and deliquescence behaviour of identified salts. At 20-25°C, NaCl exhibits a deliquescence RH (DRH) at around 75%. Gypsum does not deliquesce below 95% RH. Quartz is not a salt and is insoluble.

Compounds	Deliquescence RH (DRH)
Halite (NaCl)	Around 73 - 77%
Gypsum (CaSO ₄ ·2H ₂ O)	Does not deliquesce Critical RH around 95% for dehydration to bassanite (CaSO ₄ ·0.5H ₂ O)
Quartz (SiO ₂)	Insoluble, No RH sensitivity

Table 3 DRH of the sample inferred from published data

2) Thermodynamic Modelling

IC-derived molarities was input into ECOS/RUNSALT to predict salt phase behaviour. After auto-balancing charge and excluding gypsum, model outputs indicate a DRH at approximately 71-75% at 20-25°C.

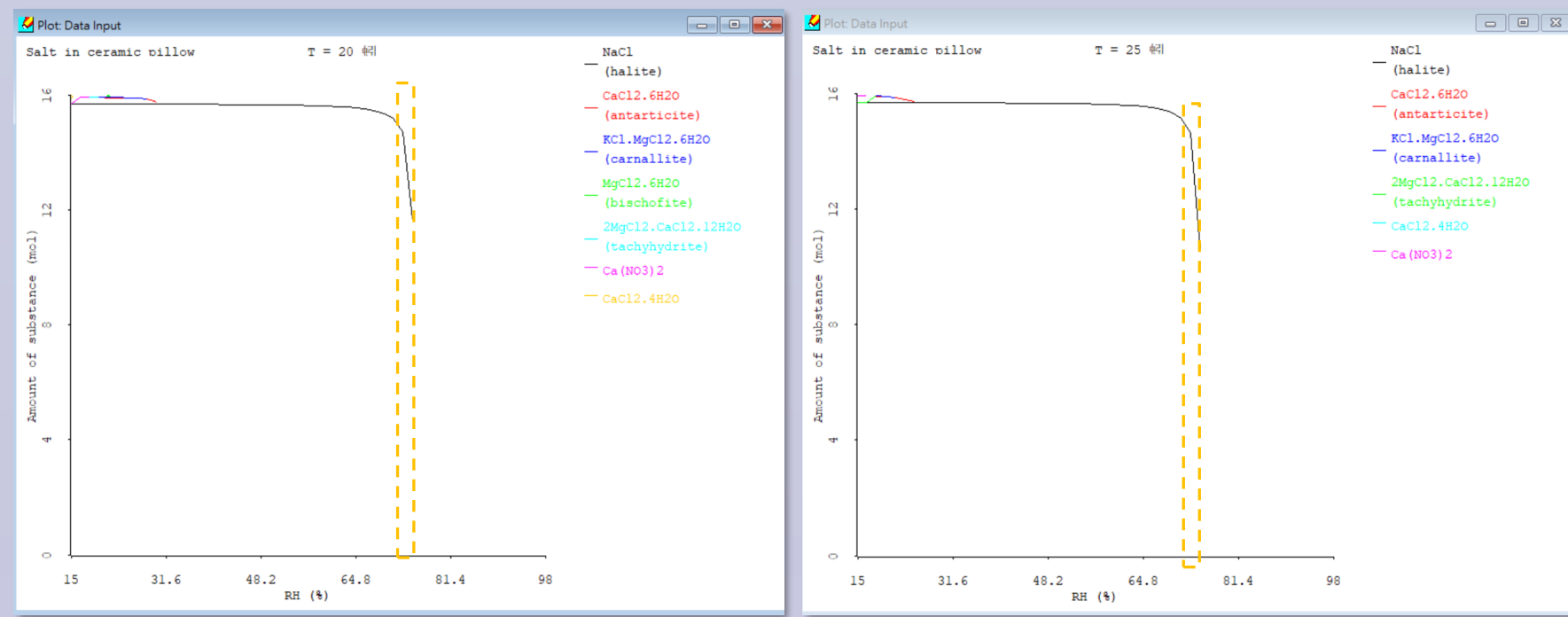
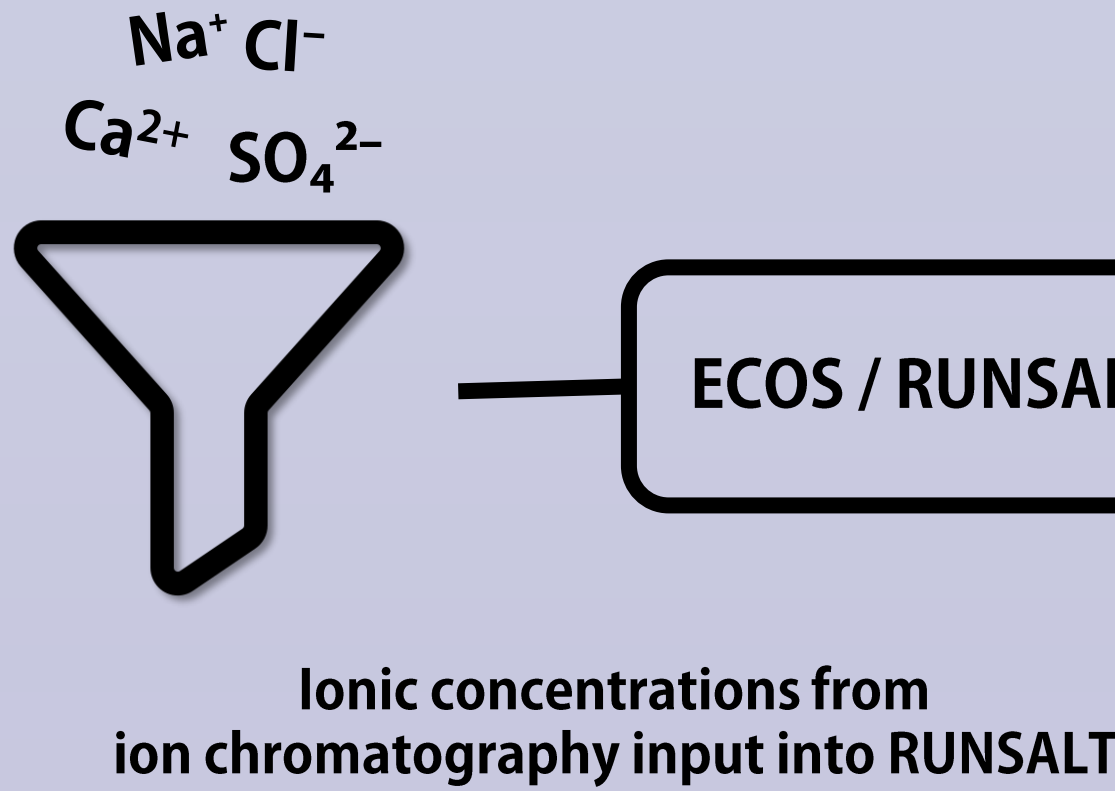


Fig.9 ECOS/RUNSALT output calculated at 20°C / 25°C. The dashed rectangle illustrates the DRH of the salt mixture

3) Dynamic vapour sorption (DVS) analysis

DVS measured moisture uptake under controlled RH. DVS isotherms showed deliquescence onset between 70% and 75% RH, with sharp and uncontrolled mass gain near 80% RH, indicating full dissolution. Desorption data revealed crystallisation upon drying starting from 70% RH at 20°C and 75% RH at 25°C, confirming reversible behaviour of the salt sample.

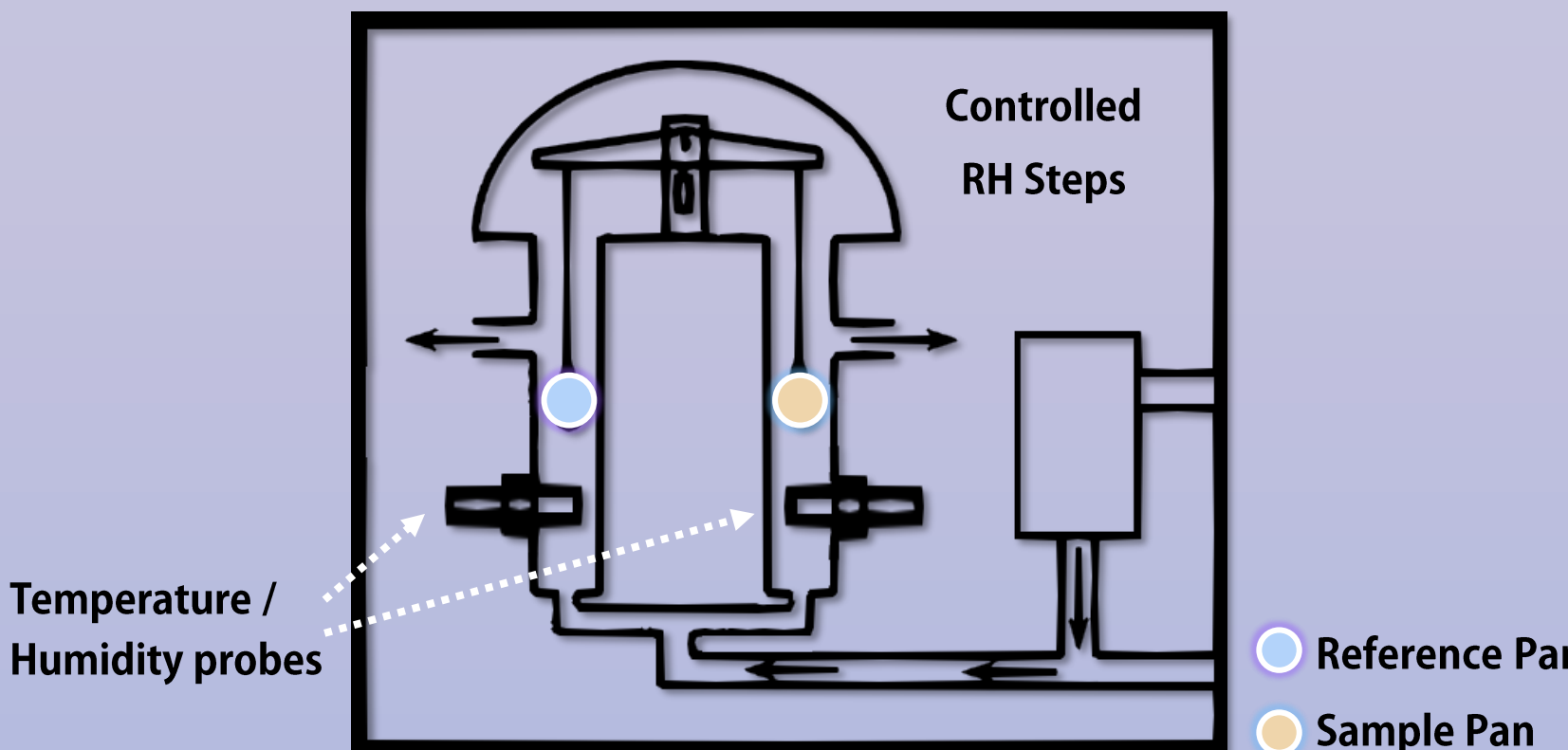


Fig.10 Schematic illustration of the basic principle of DVS device

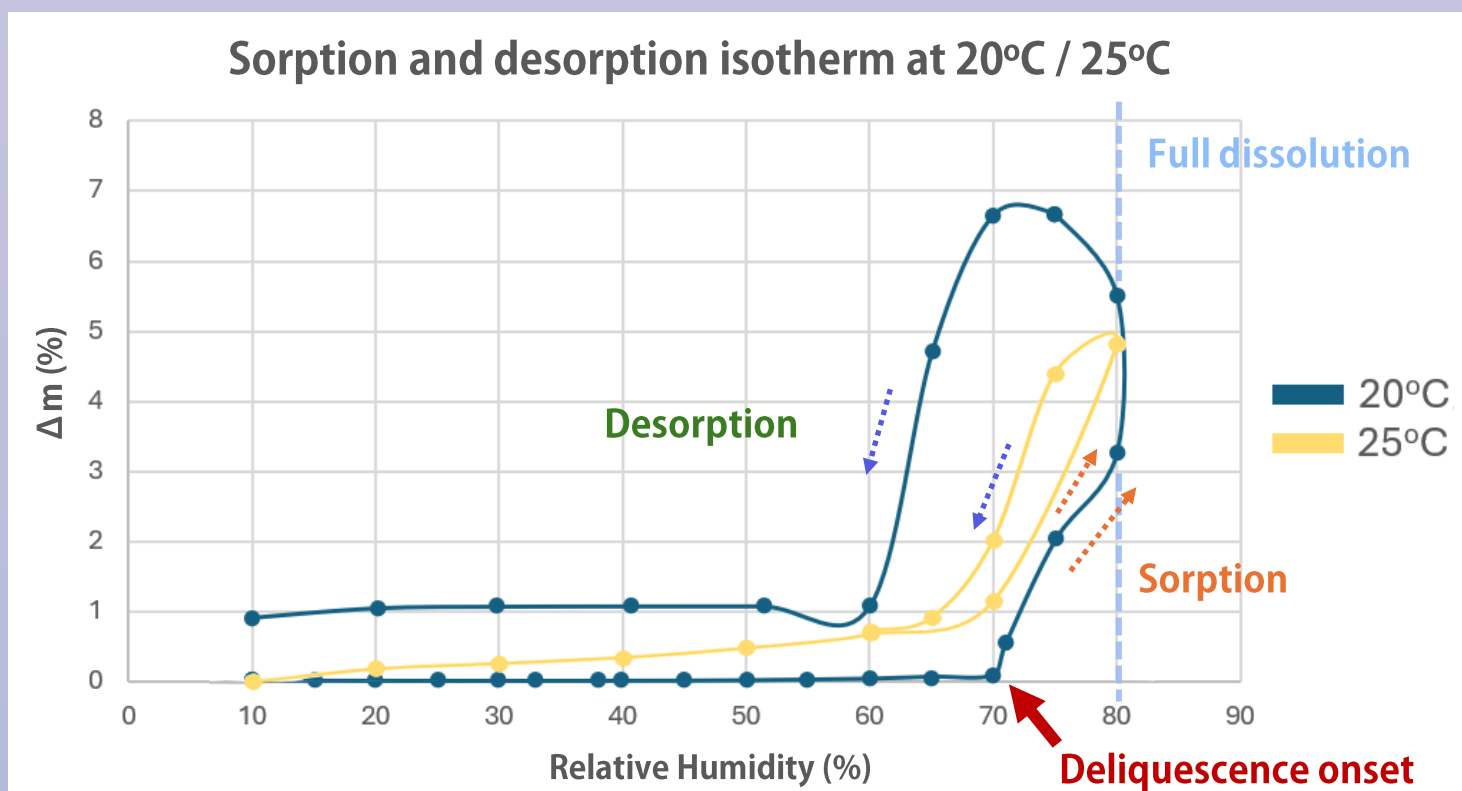


Fig.11 DVS sorption and desorption cycle curve of the sample [change in mass (%) vs RH (%)] at 20°C / 25°C

Comparative Evaluation

The three methods were compared in terms of accuracy, practicality, and limitations in defining the safe RH range.

Literature inference was the fastest but limited to single salts only. Thermodynamic modelling offered stronger predictive capacity for complex salt mixtures but required ion data and specialist software. DVS provided direct empirical result, but took the longest testing time and highest cost.

Methodology	RH Thresholds Accuracy	Analysis Required	Time Required	Cost	Optimal For
Literature Inference	Low for mixture	XRD Quantitative	Shortest	Lower	Pure dominant salts, by inference
Thermodynamic Modelling	Good, but assumes ideality	IC for Anions and Cations	Medium	Lower	Multi-salt system, predictive
DVS Analysis	Highest, capture kinetics	DVS Analysis Service	Longest	Higher	Complex mixture, empirical

Table 4 Comparison summary of the three methodologies adopted

Discussion

Differences Between Methods

DVS indicates crystallisation at a RH lower than the equilibrium predictions or reported values, reflecting kinetic effects, such as delayed nucleation and metastable supersaturation, which allow solutions to persist at a RH below the equilibrium level before crystallisation.

Effect of Temperature

Similar sorption-desorption behaviour at 20°C and 25°C suggests limited temperature impact on the deliquescence threshold of the salt mixture within this range.

Uncertainty

Uncertainty in this study arises from spatial heterogeneity of salt distribution, where limited sampling may not represent surface variability, and from differences between DVS conditions and real ceramic pore environments, which influence crystallisation behaviour, ion diffusion, and metastable states. Analytical limits may overlook trace hygroscopic components, while short-term experiments may not capture long-term in situ dynamics.

Additionally, the simplified salt composition (halite-dominated) represents an idealised system, leading to strong agreement across methods but limiting evaluation under more complex multi-salt conditions. Future work should include substrate-specific testing, long-term monitoring, and more compositionally complex systems to improve predictive reliability.

Conclusion

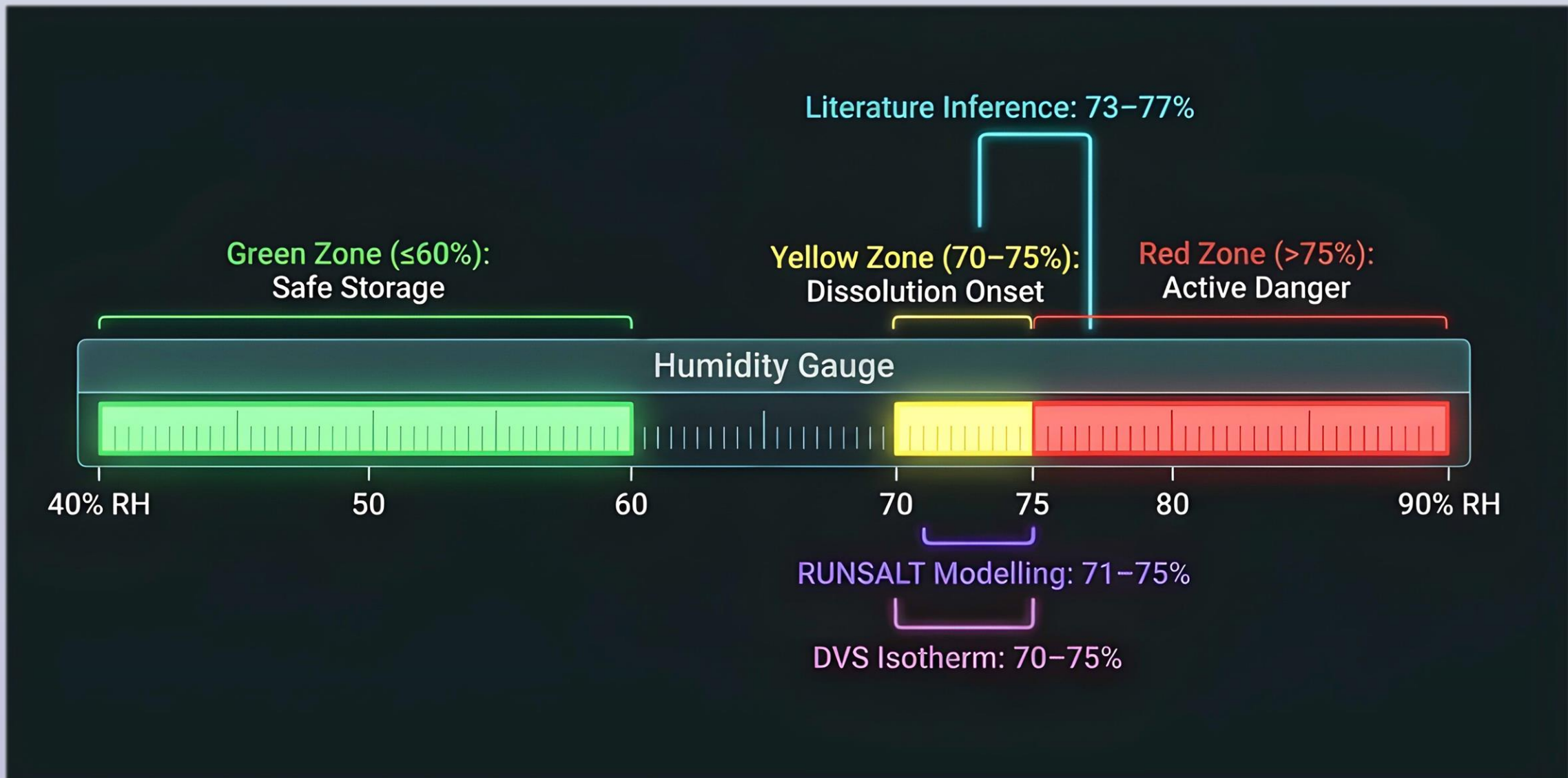


Fig.12 Summary of the study result and the recommended safe RH range for the ceramic pillow

All three methods indicate a consistent RH range. The integration of empirical and theoretical results substantiates a conservative limit of ≤ 60% RH for safe storage to prevent deliquescence–recrystallisation cycles and associated mechanical and surface damage.

This study shows that the combined in-situ experimental and thermodynamic modelling framework offers a reliable, evidence-based approach for defining RH control in preventive conservation of salt-contaminated porous materials. Conservators may adopt a single methodology or a hybrid approach based on resources available, accessibility to analytical facilities, and complexity of the salt system.

Selected Reference

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