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Preservation and Storage of Geological Samples: Implications and Suggested Methods

Decarbonisation and Resource Management programme

Open report OR/25/012

BRITISH GEOLOGICAL SURVEY

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Preservation and Storage of Geological Samples: Implications and Suggested Methods

EJM Bird, RJ Cuss, A Hines

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Front cover

Callovo-Oxfordian claystone sample vacuum-sealed in a foil bag.

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British Geological Survey offices

Nicker Hill, Keyworth,
Nottingham NG12 5GG
Tel 0115 936 3100

BGS Central Enquiries Desk
Tel 0115 936 3143
email enquiries@bgs.ac.uk

BGS Sales
Tel 0115 936 3241
email sales@bgs.ac.uk

The Lyell Centre, Research Avenue South,
Edinburgh EH14 4AP
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Tel 020 7942 5344/45
email bgslondonstaff@bgs.ac.uk

Cardiff University, Main Building, Park Place,
Cardiff CF10 3AT
Tel 029 2167 4280

Geological Survey of Northern Ireland, 7th Floor,
Adelaide House, 39-49 Adelaide Street, Belfast, BT2 8FD
Tel 0289 038 8462
www2.bgs.ac.uk/gsni/

Natural Environment Research Council, Polaris House,
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Foreword

This report is the published product of an internal study by the British Geological Survey (BGS). The work was conducted within the Fluid Processes Laboratory facility.

Acknowledgements

This study was undertaken by staff from the British Geological Survey (Radioactive Waste Team). The authors would like to thank Qian Zhang for his assistance in preparing samples and data collection.

The authors would also like to acknowledge the contribution of work experience student Andrew Hines of The King's School, Grantham who ran the Mx80 bentonite air-drying experiment during the summer of 2024.

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Summary

This report describes a study undertaken to determine the effectiveness of various preservation methods for reducing desaturation of geological samples held in long-term storage. A total of 24 samples of Callovo-Oxfordian claystone were tested using different storage methods. The change in water content was recorded regularly over one year and revealed that the best method is using a chamber vacuum sealer with sandwiched aluminium/mylar bags. Oven-drying two samples also revealed that it can take up to 7 days for all water to be removed from samples, so this should be the minimum drying time before geotechnical properties are determined. The moisture loss seen in a pre-compacted sample of bentonite showed that exposure to the atmosphere of just 1 hour is sufficient to reduce saturation sufficiently to change THMCG properties. Therefore, samples should be vacuum sealed in Mylar as much as possible during sample preparation.

1 Introduction

Clay-rich samples of rock which are stored long term, are susceptible to change due to atmospheric conditions. The primary issue is the loss of fluids to the environment due to a lower external humidity compared with the saturation of the rock. It is also possible for rocks to take on fluids from the environment if they have especially low water contents, or are stored in humid conditions. The saturation of a geological sample has a significant impact on its strength and flow properties, and is a strong control on thermo-hydromechanical-chemical-gas (THMCG). Therefore, water content strongly influences the effect of heat, mechanics (deformation), chemistry, and flow. Moisture content influences test behaviour which has implications for data collected during laboratory experiments, so it is therefore important to maintain the initial water content during storage as much as possible. In addition, exposure to the atmosphere can result in oxidation of minerals, altering the geochemistry of the rock. This too can have a profound impact on the THMCG properties of samples.

As preservation of samples is vital to understand THMCG properties of clay-rich rocks, it was necessary to determine how quickly samples lose water and the effectiveness of different preservation methods. This approach would allow recommendations to be made on appropriate preservation, and acceptable exposure times that samples can tolerate, before being degraded.

2 Starting material

Two studies were conducted. The first looked at the drying of Callovo-Oxfordian claystone with different sample preservation methods. The second was a study of moisture loss from an unpreserved sample of pre-compacted bentonite.

2.1 STARTING MATERIAL 1: CALLOVO-OXFORDIAN CLAYSTONE

The main study used two core barrels (EST68872 and EST67272) of Callovo-Oxfordian claystone (COx), supplied by Andra from the Meuse/Haute-Marne underground research laboratory at Bure in France. When the core was taken from its protective T-cell arrangement, it was found to be fractured and was not fit for THMCG testing. Opportunity was taken to use this core material for the current study instead of disposing of the fragments of rock. The UA (clay-rich) variety of COx (150-160 Ma) occurs at repository depth. Wenk *et al.* (2008) reports clay composition as 25-55 wt%, 23-44% carbonates and 20-31% silt (essentially quartz + feldspar). Clay minerals are reported to include illite and illite-smectite with subordinate kaolinite and chlorite. Yven *et al.* (2007) report three main mineral phases; clay minerals (20–55%), quartz and calcite (40–55%). Secondary mineral phases include dolomite, feldspar, pyrite, hematite and traces of siderite. Where necessary, large fragments of COx were broken up to create approximately equal sized fragments of rock with an average weight of 71.6 g (37-90 g, with two larger samples of ~170 g). It must be noted that the geometry of the samples was not identical, and therefore the surface area of the samples varied. It has been assumed that the water content of core was homogeneous, and all samples had the same starting water content.

Tests were conducted in two batches. The first commenced on 16th October 2023 and the second on 19th March 2024.

2.2 STARTING MATERIAL 2: PRE-COMPACTED VOLCLAY MX80 BENTONITE

The bentonite used was VolClay MX-80 (Johannesson, 2003, 2014) supplied by the American colloid Company (now Mineral Technologies Inc.) through Sibelco Nordic, who crushed and dried the material. Mx80 is a fine-grained, (16 to 200 μm) sodium-rich clay from Wyoming with a nominal dry density of 1506 kg.m^3. On average the material comprised (by percentage weight) 90.2% montmorillonite, 0.5% gypsum, 4.8% quartz, 0.1% calcite, 3.5% plagioclase and 0.9% muscovite. At a 5% solids dispersion in distilled water the pH of the mixture ranges between 8.5

and 10.5. As supplied, the moisture content is 12 %. The chemical composition data provided by the manufacturer and a complete description are given by Svensson et al. (2017).

A pre-compacted sample of bentonite was made by mixing the powdered clay with a known amount of deionised (DI) water to create a fully saturated sample of dry density of 1.7 g/cc. The wetted clay was vacuum packed and stored overnight in a fridge, before being compressed in cylindrical steel pressure vessel at 80 MPa axial pressure (with constant radial confinement) to produce a test sample at 100% saturation. During compression, the sample former was under vacuum to ensure that air was not trapped during pressing. The manufactured sample had a weight of ~185 g and dimensions of D62 mm x L31 mm.

3 Methodology

A total of 24 subsamples were taken from two cores of Callovo-Oxfordian claystone (COx) in two batches (Figure 1). The initial mass of each sample was measured using a Mettler Toledo AB304-S/FACT analytical balance which records mass to a resolution of 0.1 mg.

Figure 1 Various sample preservation methods (grid spacing = ½ inch). a) Oven-drying; b) Open air; c) Cling-film; d) Vacuum-sealed plastic bag; e) Cling-film + vacuum-sealed plastic bag; f) Aluminium foil + vacuum-sealed plastic bag; g) Vacuum-sealed, foil-lined plastic bag; h) Wax-sealed; i) Vacuum-sealed foil bag 1; j) Vacuum-sealed foil bag 2; k) Vacuum-sealed foil bag 3; l) Vacuum-sealed foil bag 4. *BGS © UKRI*

One subsample from each core was placed in an oven at 105°C to determine water content (Figure 1a). It was assumed that all samples had the same starting water content.

Six subsamples were not vacuum-sealed: two subsamples were left open to air as a control (Figure 1b), two were wrapped in clingfilm (Figure 1c) and another pair were wrapped in clingfilm before being sealed using molten paraffin wax (Figure 1h).

A further eight subsamples were vacuum sealed on the 16th October 2023, using an Andrew James AJ000641 Commercial Vacuum Sealer (Figure 2). Plastic seal bags were used, manufactured mainly for Sous Vide cooking. Two samples were placed directly into a plastic seal bag (Figure 1d), two were wrapped in clingfilm before being placed into a plastic seal bag (Figure 1e), two were wrapped in aluminium foil and placed into a plastic seal bag (Figure 1f) and two were placed into a Mylar foil bag, which itself was sealed within a plastic seal bag (Figure 1g). It must be noted that the Andrew James vacuum sealer was not an effective sealer of the foil vacuum bags, and experience had shown that the vacuum seal failed relatively quickly. The final sample was double sealed in foil and plastic bags as it was hoped the plastic bag would seal the foil bag for longer.

Figure 2 Andrew James AJ000641 Sealer. *BGS © UKRI*

The second batch of samples were produced on the 19th March 2024. A WeVac CV12 Chamber Vacuum Sealer (Figure 3) was used to seal four pairs of subsamples in four different mylar foil bags (Figure 1i-l). The WeVac vacuum sealer had been purchased as the Andrew James sealer could not seal the thicker Mylar-type bags. One type of foil bag was a metallised Mylar material where metal is sprayed onto plastic to give a foil effect (Figure 1i). This material is similar to bags used to preserve tea bags and crisps. The other three types were comprised of a layer of aluminium sandwiched between mylar plastic. Foil bag 1 was a metallised mylar type pouch produced by WACCOMT (Figure 1i). Foil bags 2-4 were sandwiched aluminium/mylar type pouches with foil bag 2 produced by Fresherpack (Figure 1j) and bags 3 and 4 produced by LUTER (Figure 1k and Figure 1l).

The two vacuum sealers operate differently. The Andrew James sealer directly draws air from the bag before heat sealing it whereas the WeVac device pulls a vacuum in the whole chamber, seals the bag and then repressurises the chamber.

The Mettler Toledo AB304-S/FACT balance was then used to record the mass of each sample at regular intervals, starting at an interval of one day and then changing to a one-week interval. The oven-dried samples were initially weighed three times per day before being recorded along with the other samples after several days. Samples were stored in an open plastic box on top of cupboards in the air-conditioned laboratory to ensure they were not accidentally damaged.

Figure 3 WeVac CV12 Chamber Vacuum Sealer. *BGS © UKRI*

To assess the loss of water in more detail, a cylindrical sample of Mx80 bentonite was used (Figure 4). The initial mass of the sample was recorded using a Mettler Toledo AB304-S/FACT analytical balance. The sample was left exposed to the atmosphere and weighed at regular intervals, initially at 5-minute intervals, increasing to 15 minute on day 2, 3-hourly on day 5, and then increasing to daily, weekly, and fortnightly as necessary.

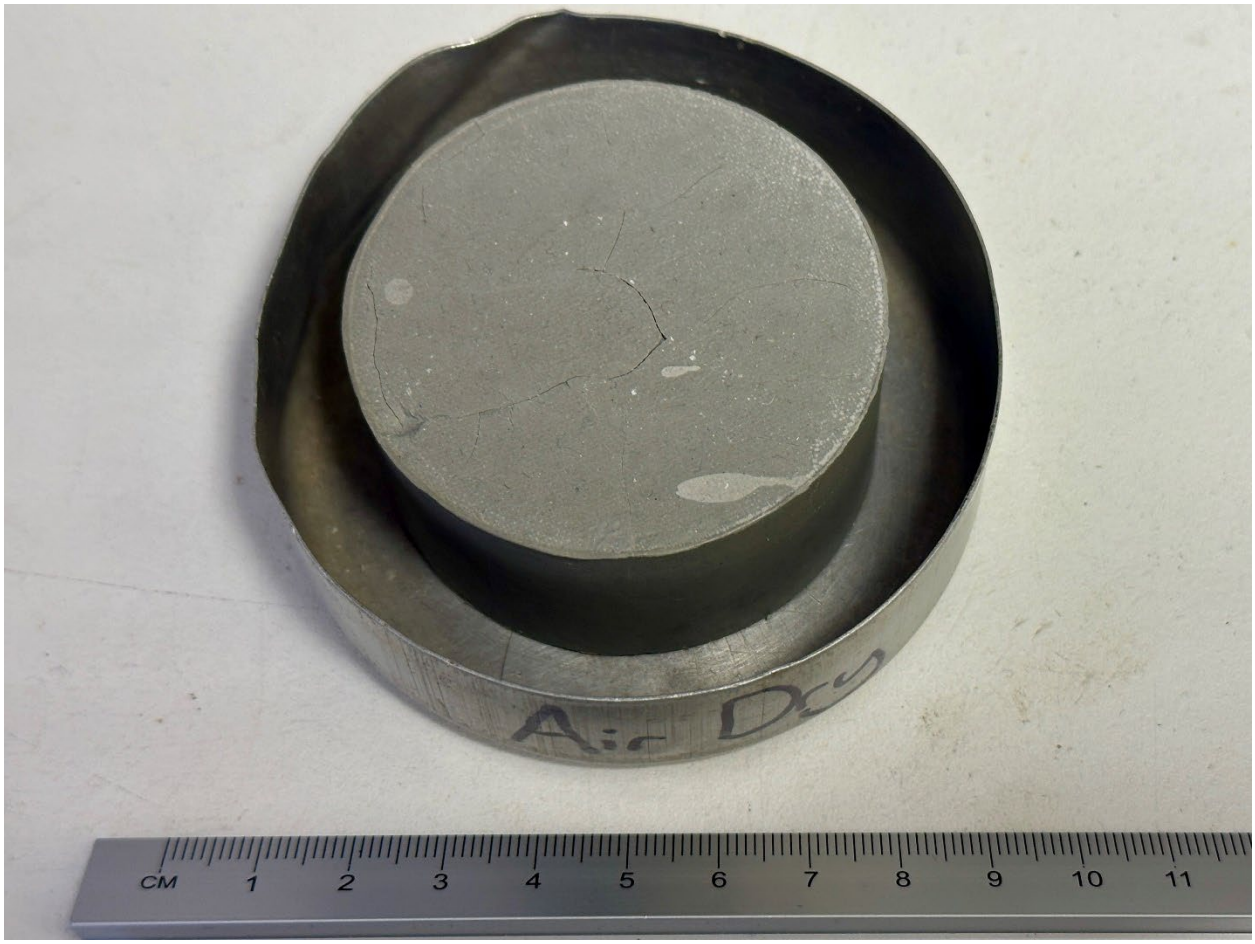


Figure 4 Cylindrical sample of Mx80 bentonite used to compare water loss with COx. *BGS © UKRI*

4 Results

4.1 OVEN-DRIED SAMPLES

Oven-drying two subsamples of the COx resulted in a 7.72% mass loss in the subsample from batch one samples (Figure 5). A 4.96% mass loss was observed in the second subsample which was produced from the second batch of samples. These values represent the water content of the samples.

A significant finding from the oven-drying of these samples was the duration taken to fully dry samples. Current practice for geotechnical observations involves placing samples into an oven at 105°C for at least 24 hours to evaporate water and determine water content (Franklin 1979). The COx samples used in this study lost between 96.6% and 98.9% of their water within 24 hours, and took between six and seven days for all water to be evaporated. This suggests that the minimum recommended time for sample drying to determine geotechnical properties may need to be increased. The time of oven-drying will be dependent on the size of samples being dried, and therefore longer drying time is recommended for large samples. The standard of 24 hours comes from drying of small quantities (a few grams) of soil and large, competent rock will take much longer to fully dry.

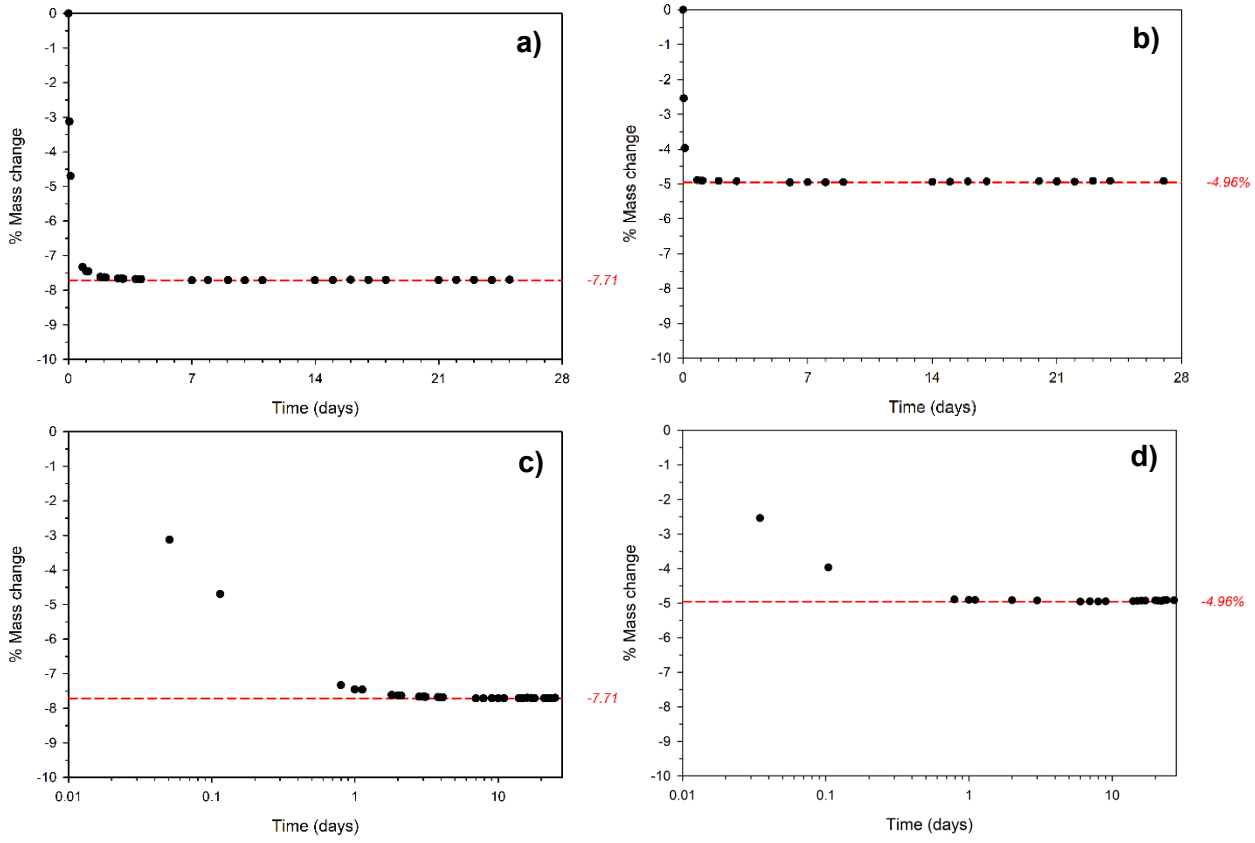


Figure 5 Mass change for two oven-dried Callovo-Oxfordian claystone samples over four weeks. a) Subsample from batch including bare unsealed, film unsealed, bare sealed, film sealed, foil sealed, foil-lined bag sealed and wax-sealed subsamples. b) Subsample from batch including mylar foil bags 1-4. c) As 4a above with log scale. d) As 4b above with log scale. *BGS © UKRI*

The first of the oven-dried samples was removed from the oven after it was established that all initial water had been removed. This resulted in a rapid uptake of water from the atmosphere as the sample regained 12.0% of its original water content within 24 hours (Figure 6). The saturation of the oven-dried sample quickly came within 10% of that of the air-dried samples within three weeks and continued to slowly approach a value 6% lower than the air-dried samples by the end of the study. The water content of the oven-dried sample also closely matched the erratic pattern of the air-dried samples as it balanced with the changing humidity of

the air-conditioned laboratory. The rapid uptake of water following the removal of the sample form the oven demonstrates the need to determine geotechnical properties immediately after removal.

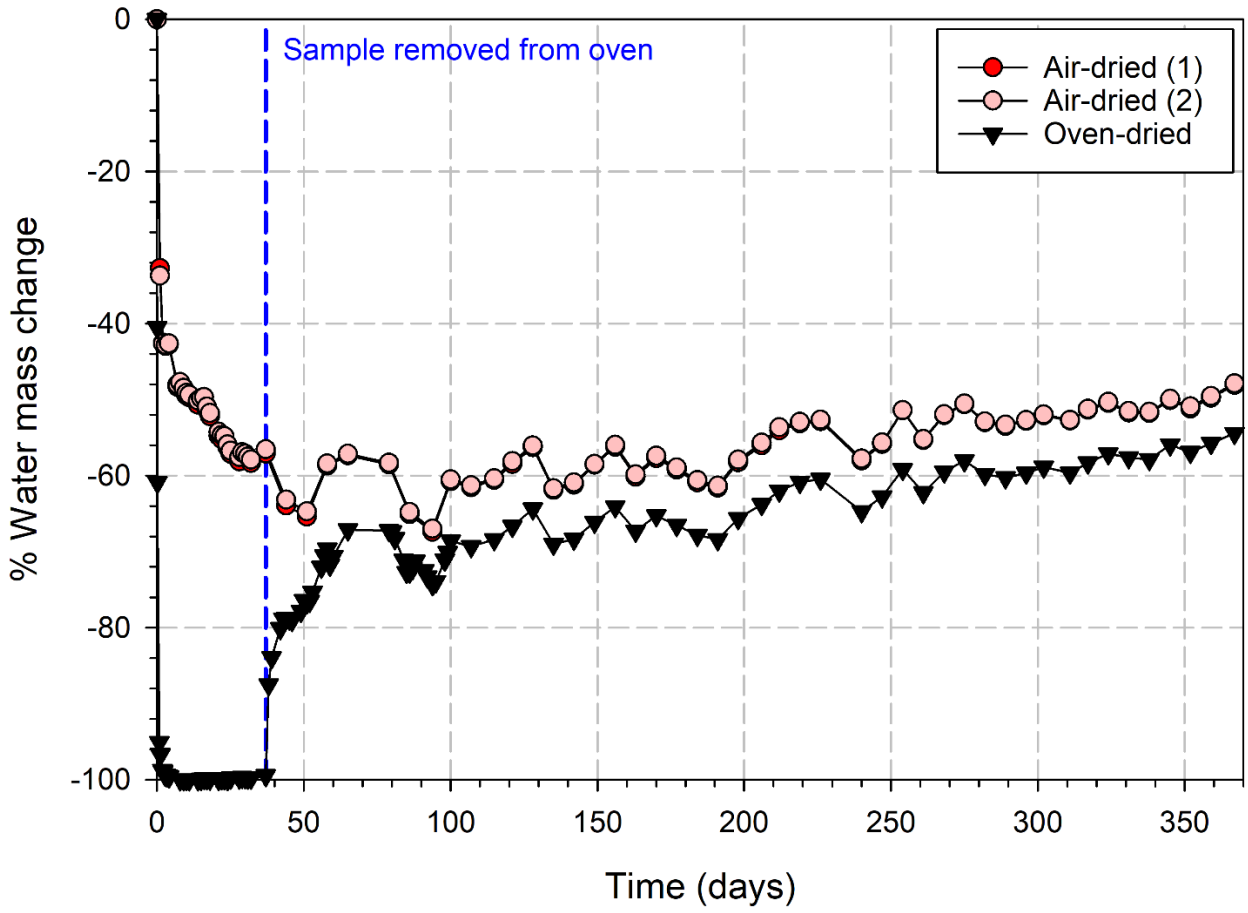


Figure 6 Response of water mass to removing a sample from the oven and comparison to air-dried samples. *BGS © UKRI*

4.2 SAMPLE PRESERVATION

A large range of desaturation rates were observed across the tested preservation methods. However, the pairs of samples stored with the same method formed close, discrete groups demonstrating a good level of repeatability (Figure 7). Slight differences can be explained by variations in surface area/volume ratio. Higher surface area proportions, particularly in smaller samples, facilitate greater rates of desaturation.

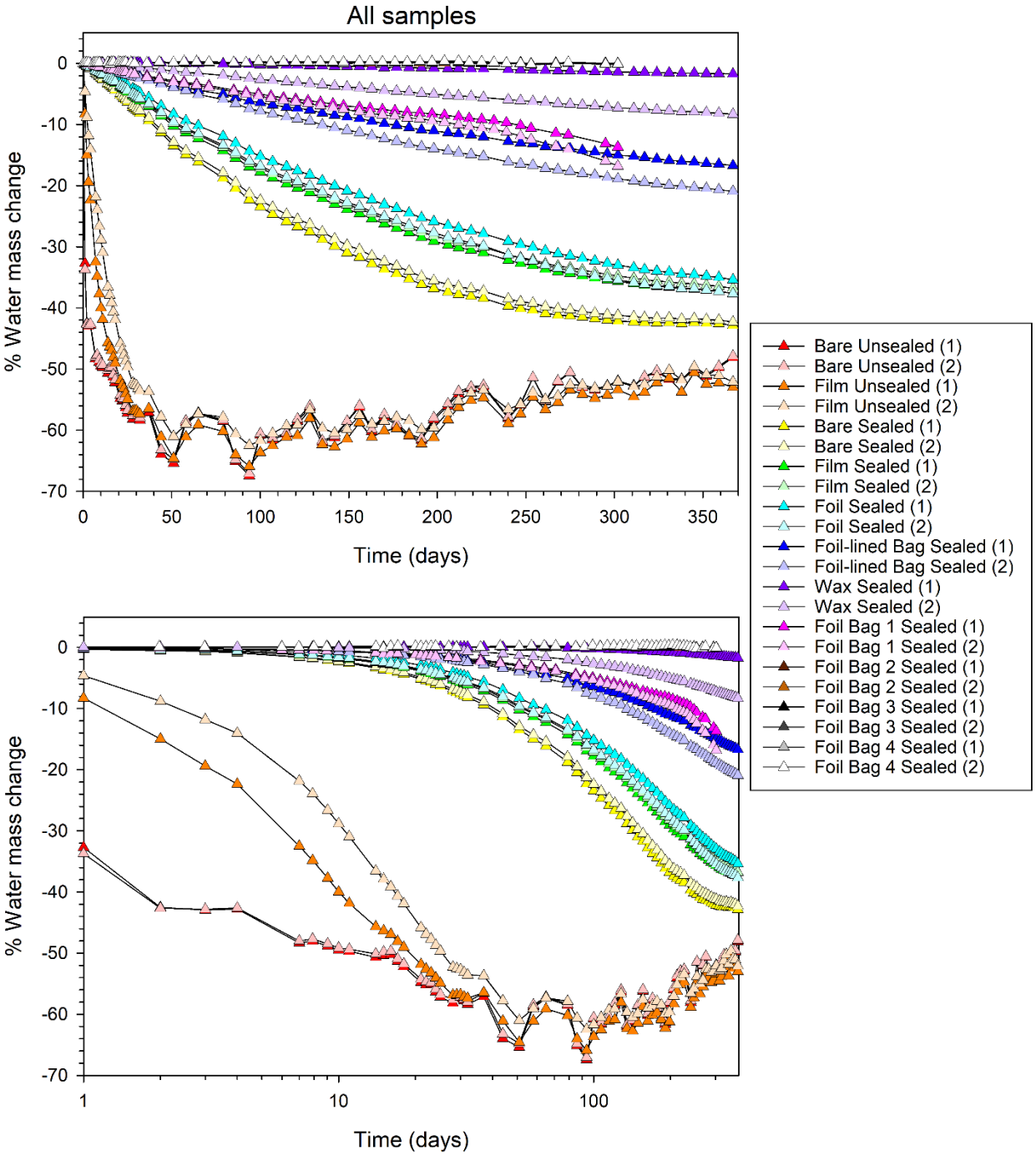


Figure 7 Change in water content observed in samples stored with various preservation methods. *BGS © UKRI*

4.2.1 Unsealed Samples

Rapid water loss was observed in both the bare, unsealed samples and the cling-film wrapped, unsealed samples (Figure 8). Within 24 hours, both bare samples had lost about a third of their original water content, and by 50 days they had lost approximately 65%. The use of clingfilm initially delayed the loss of water. However, after 50 days these samples experienced a similar level of desaturation to the bare samples. All four unsealed experiments exhibited fast responses to atmospheric changes with a jagged pattern observed after the initial sharp drop in moisture. The clingfilm-wrapped samples showed a slightly dulled, smoothed response to changing room conditions compared to the bare samples.

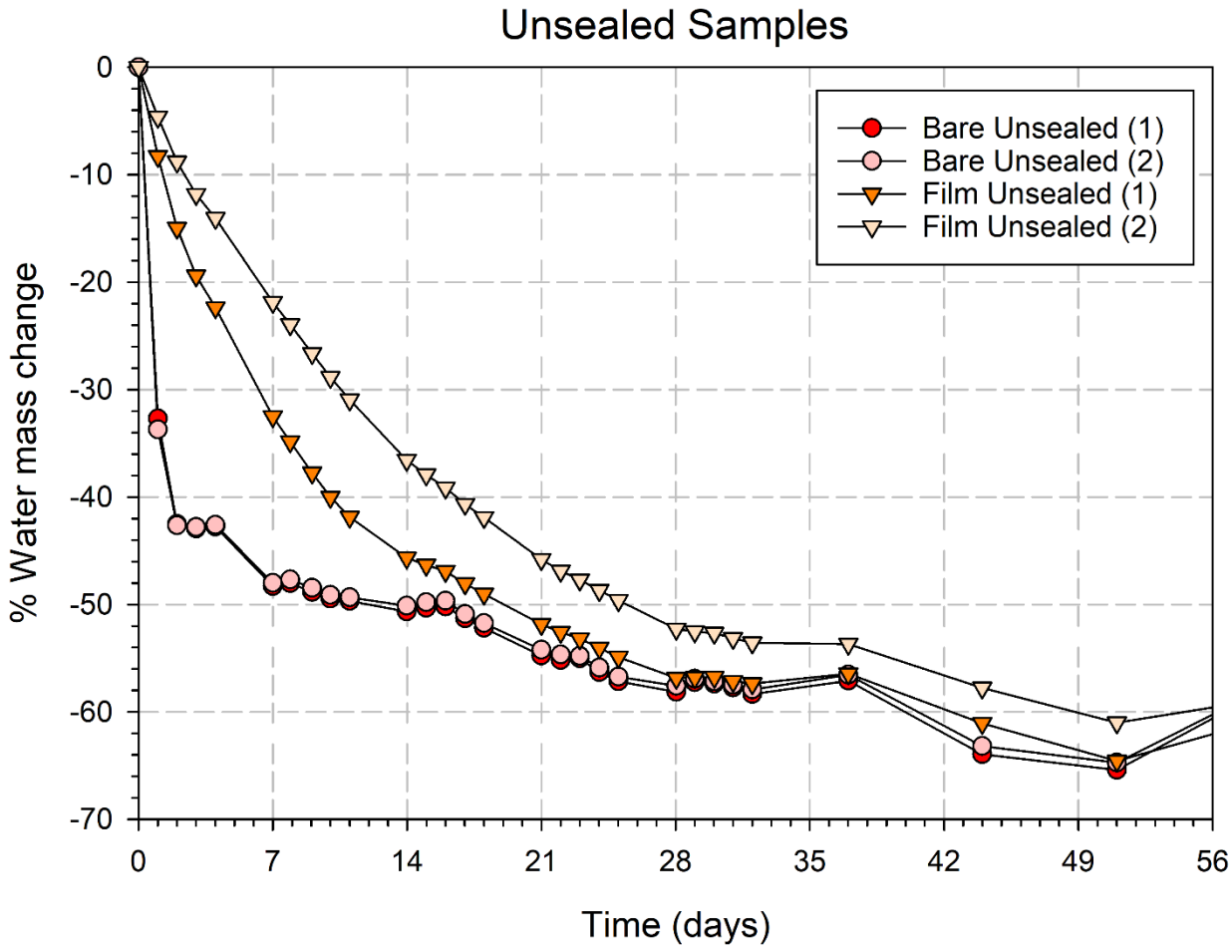


Figure 8 Water loss observed in unsealed samples air-dried over eight weeks. *BGS © UKRI*

4.2.2 Plastic vacuum sealed samples

Samples sealed using plastic Sous Vide seal bags using the Andrew James AJ000641 sealer experienced a steadier decline in saturation, with all samples characterised by a smooth curve of moisture loss across a year (Figure 9). The bare samples performed the most poorly of the plastic vacuum sealed samples, losing 42-43% of their original water content over the duration of the test (1 year). Both clingfilm wrapping and aluminium foil wrapping the samples prior to vacuum sealing yielded similar results, reducing desaturation by around 5%. The samples placed into foil-lined bags performed significantly better than the others. The use of foil-lined bags reduced water loss to between 17-21% over a year.

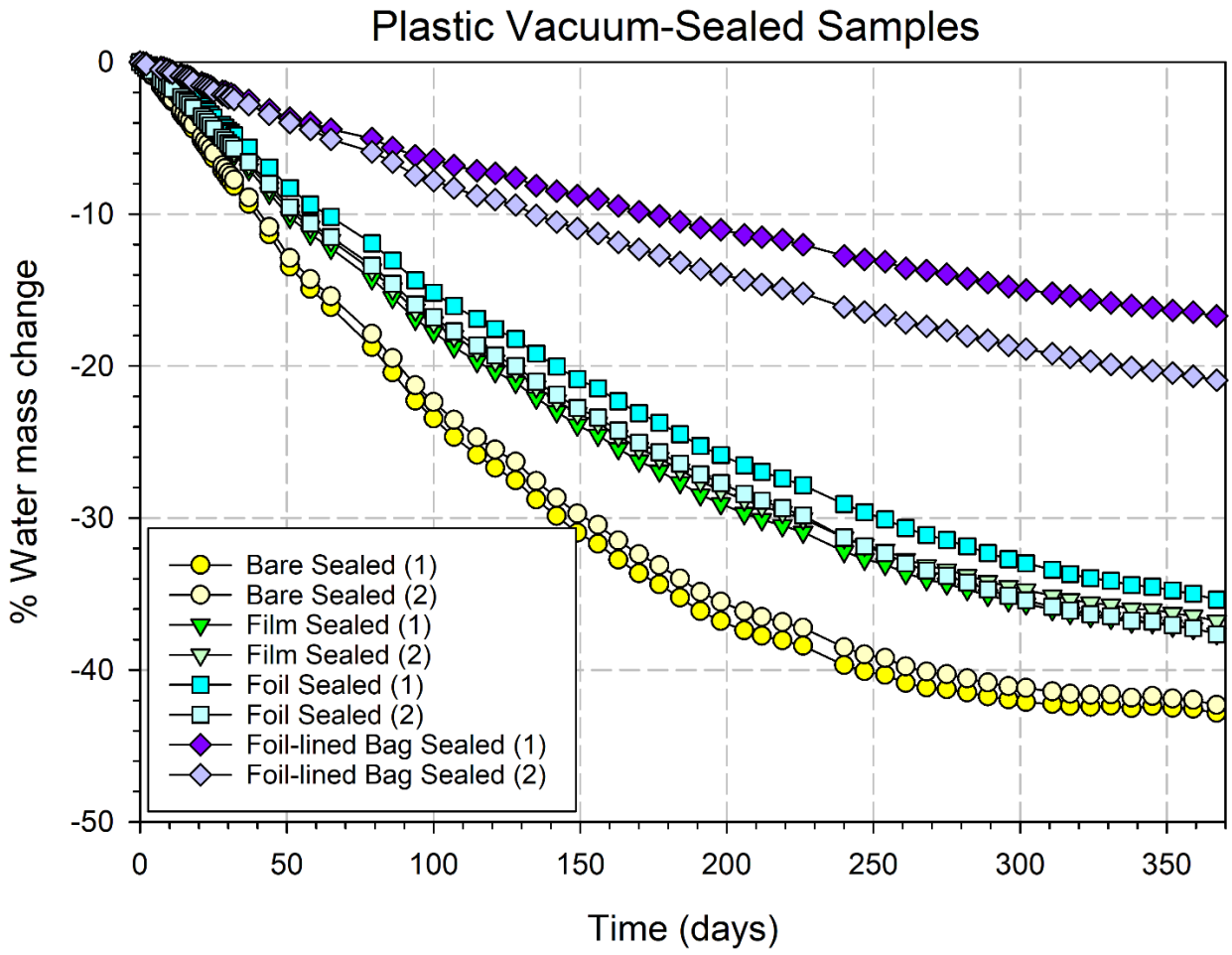


Figure 9 Water loss observed over one year in samples vacuum-sealed in plastic sous vide bags with different additional protection. *BGS © UKRI*

4.2.3 Wax-sealed Samples

The samples sealed in wax were able to retard moisture loss to a greater extent than those vacuum-sealed in plastic sous vide bags. Following wax-coating, both samples were measured and experienced a slight initial increase in bulk mass before decreasing in a fairly linear fashion (Figure 10). It is possible that the wax itself absorbed some water from the atmosphere which could explain the initial increase in mass.

The raw sample masses, without clingfilm or wax, were recorded before and after the study. This revealed a discrepancy between the raw and bulk sample mass changes with the raw samples losing 0.0179-0.0304% more mass than the bulk samples. The absorption of moisture into the paraffin wax coating could explain this. A large discrepancy in performance was observed in the wax sealed samples. These two samples had different sizes, with the first over twice as large, giving the second sample a significantly higher surface area/volume ratio. This could have enabled the higher desaturation observed. However, experience has shown that wax coating samples fully, is difficult, and that the clingfilm covering the sample can melt during the application of molten wax. This means that the complete coverage of the sample cannot be guaranteed and that differences in moisture preservation are not unexpected between samples. Over prolonged periods, wax can also crack, particularly over clingfilm which is a flexible material, resulting in a loss or reduction in protection.

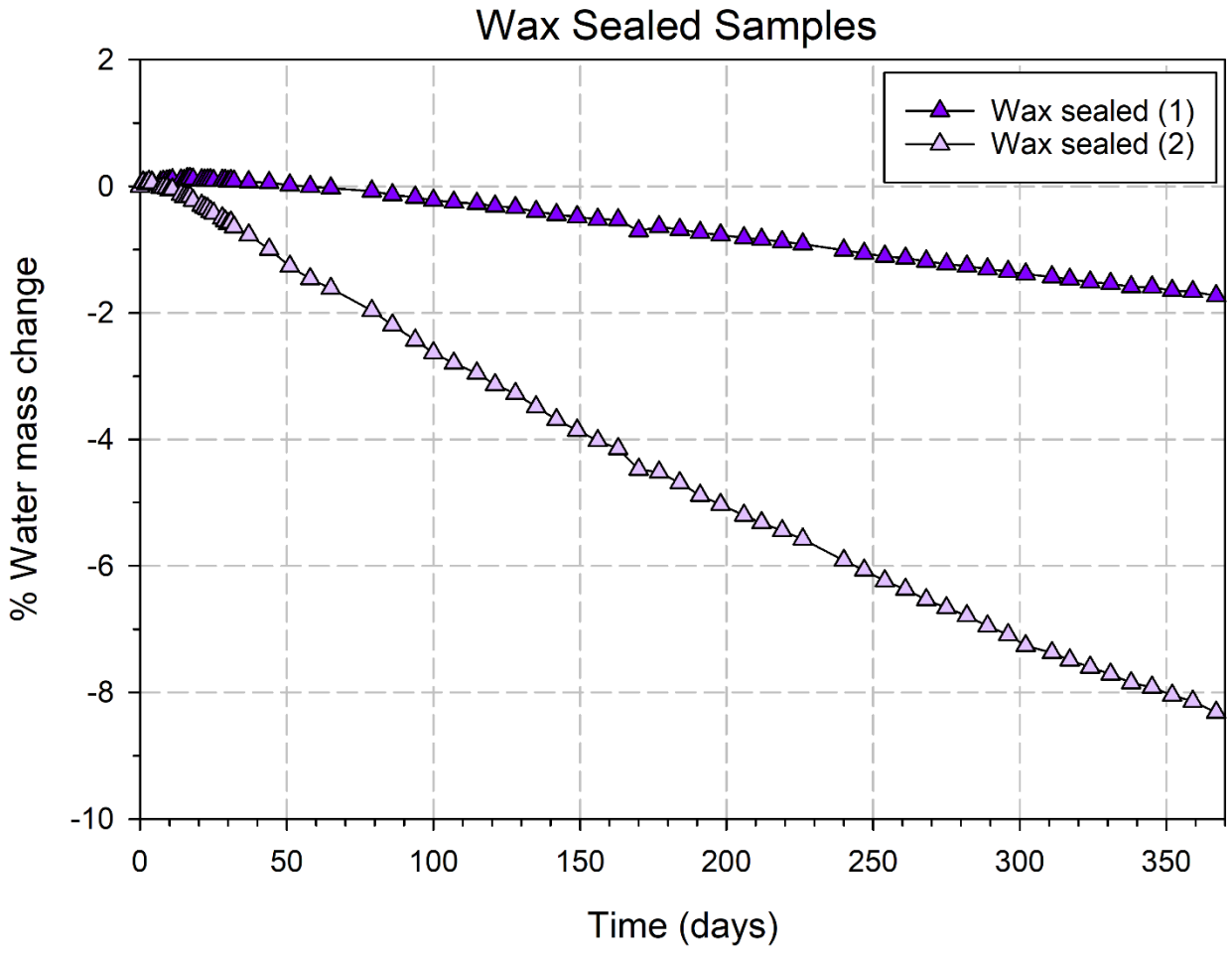


Figure 10 Water mass change observed in wax-sealed samples. *BGS © UKRI*

4.2.4 Mylar vacuum sealed samples

Four types of foil bags were tested with the WeVac sealer. Foil bag 1 was a metallised pouch manufactured by the spraying of metal onto plastic, whereas foil bags 2-4 were manufactured by sandwiching a layer of aluminium metal between heat sealing plastic. The metallised pouches experienced a steady decline in saturation, before an acceleration in desaturation rate towards the end of the test (Figure 11). This was likely caused by a loss of seal and subsequent introduction of air to the samples. The bags with a real aluminium layer performed significantly better than the metallised ones, with no significant mass change observed.

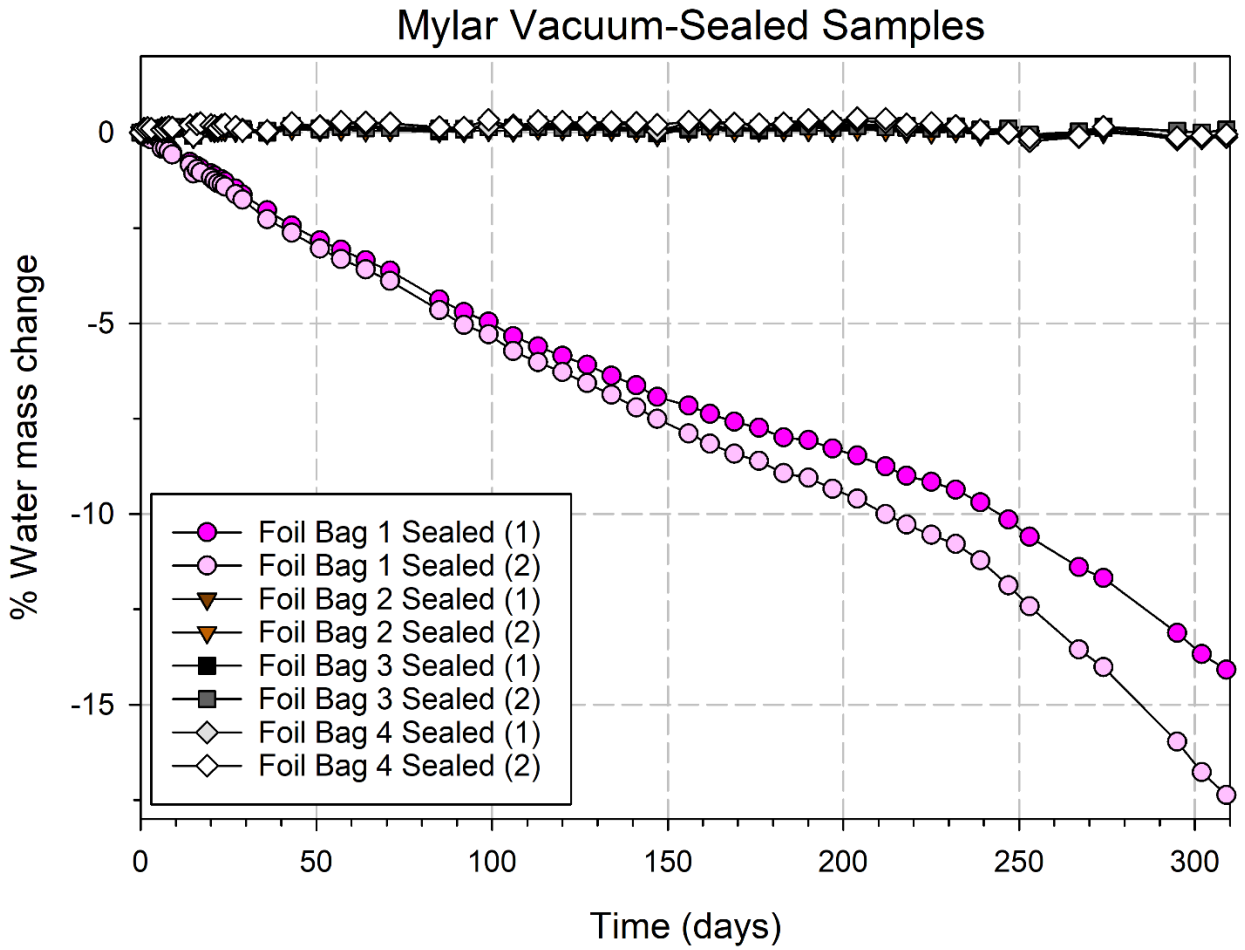


Figure 11 Water loss observed over one year in samples vacuum-sealed in foil bags. BGS © UKRI

Bags containing a sheet of aluminium sandwiched between heat sealing plastic performed the best of all samples (Figure 11). Across both manufacturers of this style of bag, the change in mass observed was negligible and was very close to the 0.1 mg resolution of the Mettler-Toledo balance (Figure 12). This corresponds to a maximum resolution of ±0.004% change in water mass. A slight increase was seen in all samples over the first 232 days. A similar pattern of peaks and troughs is seen for all test samples and therefore this variation has been interpreted as difference due to the balance, and not related to changes of the COx samples.

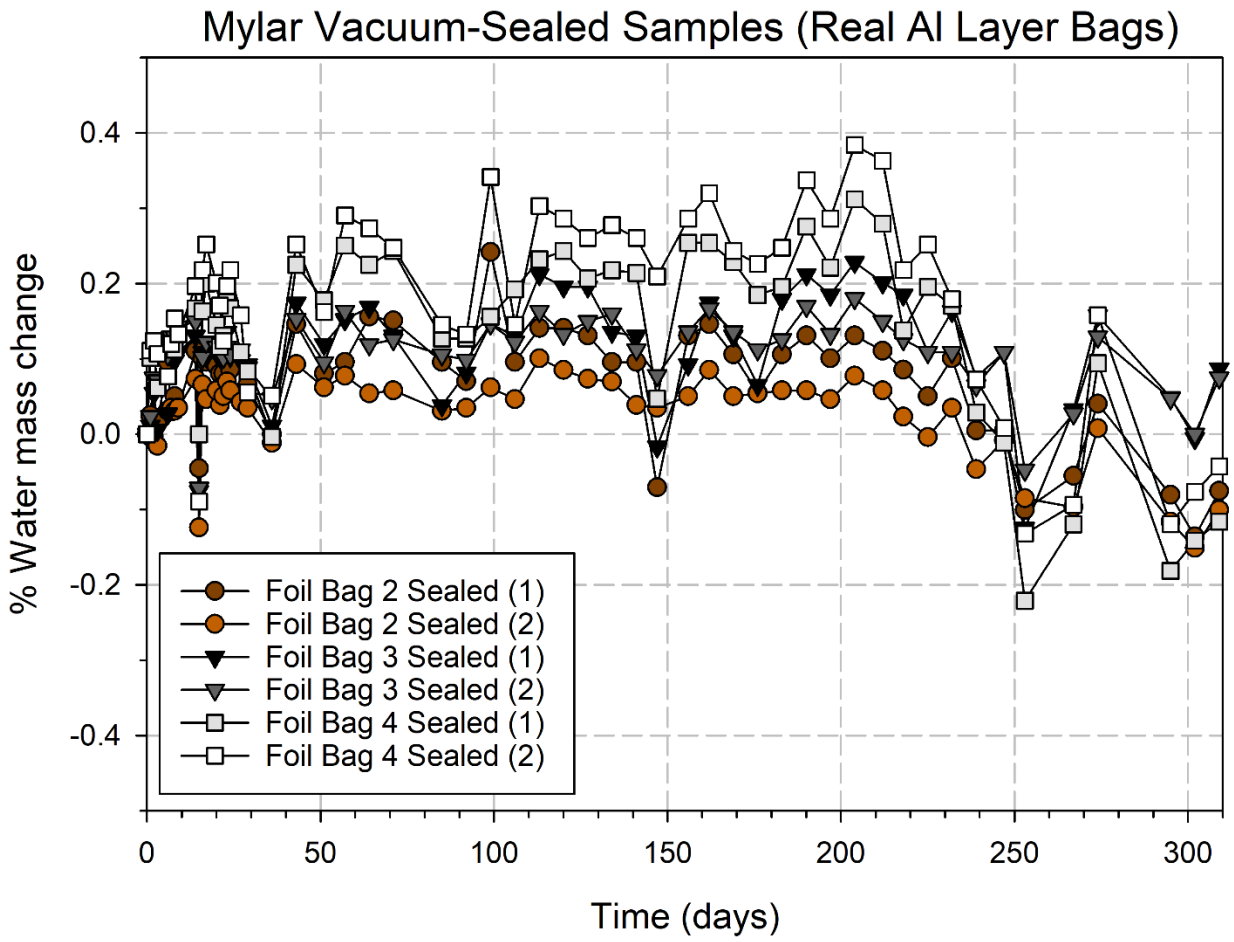
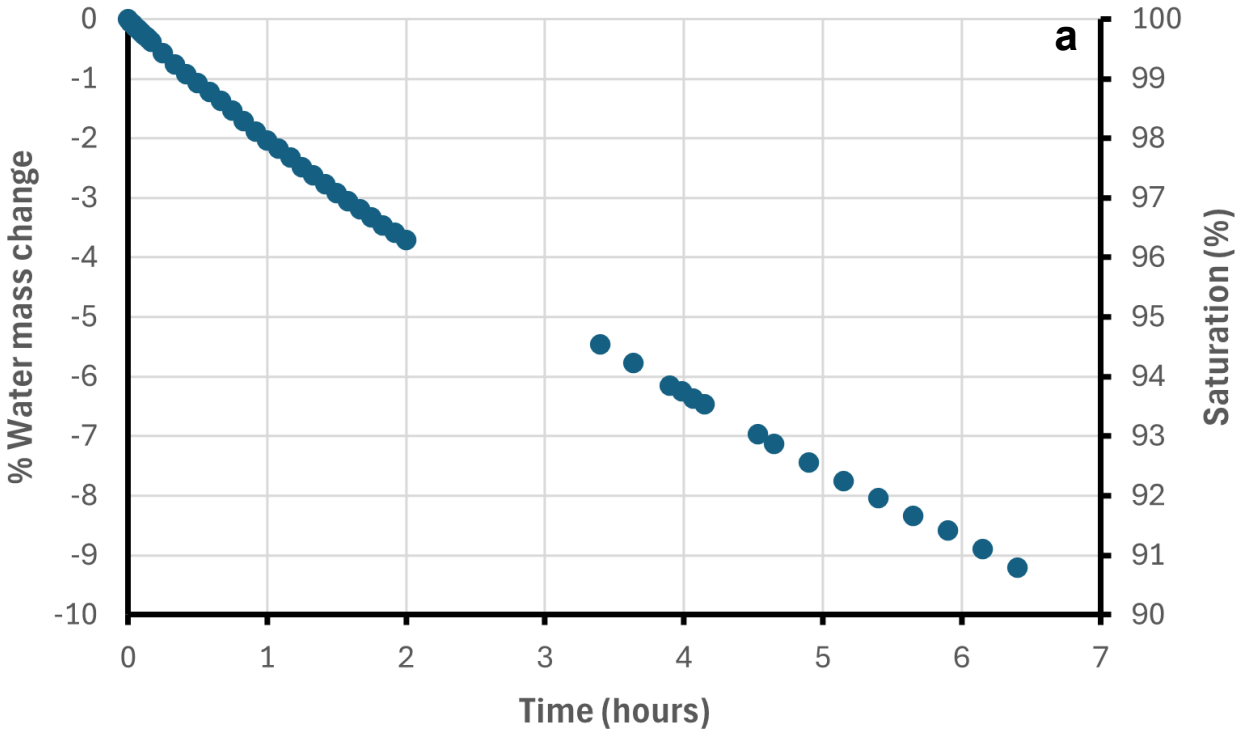


Figure 12 Water mass change observed in samples vacuum-sealed with different aluminium layer foil bags. *BGS © UKRI*

4.3 AIR DRYING OF MX80

A second study was conducted looking at water loss in pre-compacted bentonite in detail over a much shorter time period. As well as moisture loss, this study measured the colour of the sample to see if colour could be used as a proxy for moisture content. This study was conducted by work-placement student Andrew Hines between 1st and 5th July, with measurement continued by BGS staff.

Figure 13 shows the water loss and saturation of the sample within the first 7 hrs, and then for the first 7 days of measurement. Starting from an initial saturation of 100, over 2% saturation is lost in just one hour, with saturation reducing to 90% in 7 hours (Figure 13a). Within 1 week, saturation has reduced to below 50% (Figure 13b). THMCG properties in Mx80 have been shown to be very sensitive at saturations above 95%. Therefore, while the moisture loss highlighted may seem small, it will have profound influence on the behaviour of the sample.



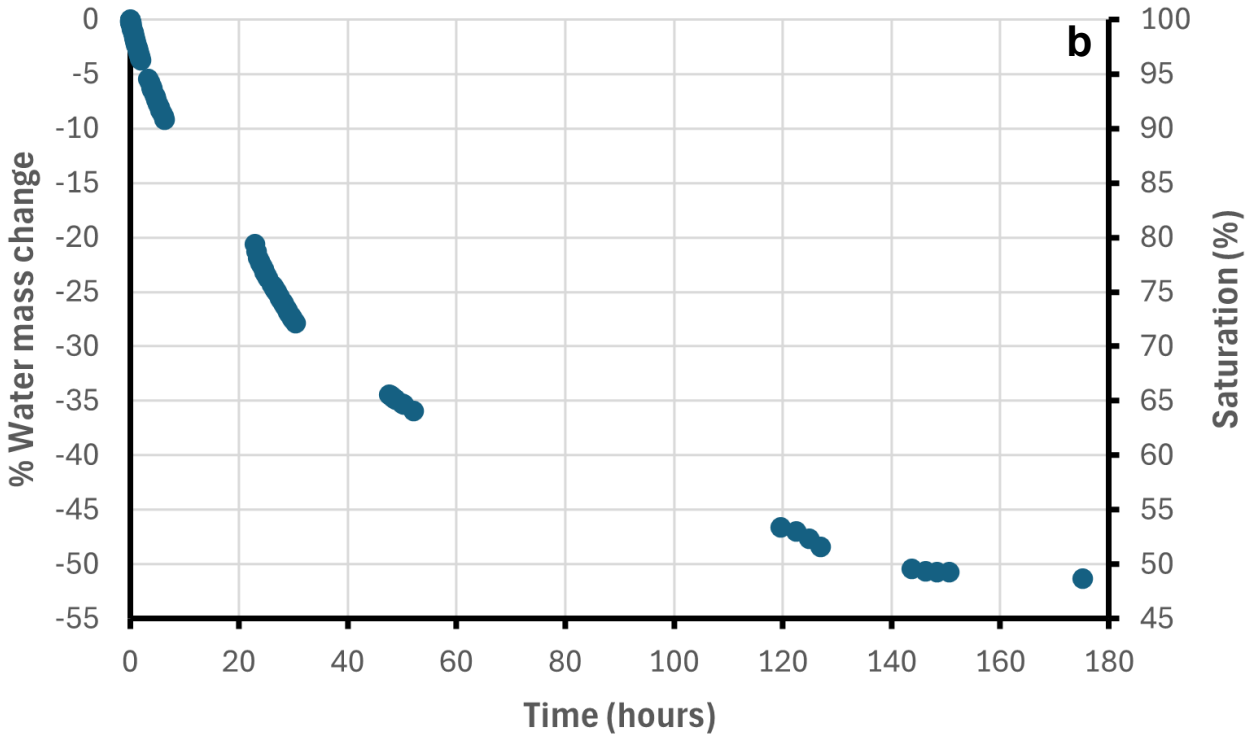


Figure 13 Water loss and saturation of sample of Mx80 exposed to the atmosphere; a) 7-hour duration; b) 7 day duration. *BGS © UKRI*

Figure 14 shows the water loss from the sample of Mx80 over 50-days, and compares this with the moisture loss seen in COx. The synthetic sample of Mx80 bentonite showed a very similar pattern of desaturation to the two COx samples. This demonstrates that the rate of water loss over time is broadly similar between different clay types. Therefore, the high moisture loss seen in Mx80 over the course of 1 hour is not unique to Mx80 and will be observed in clay-rich rocks.

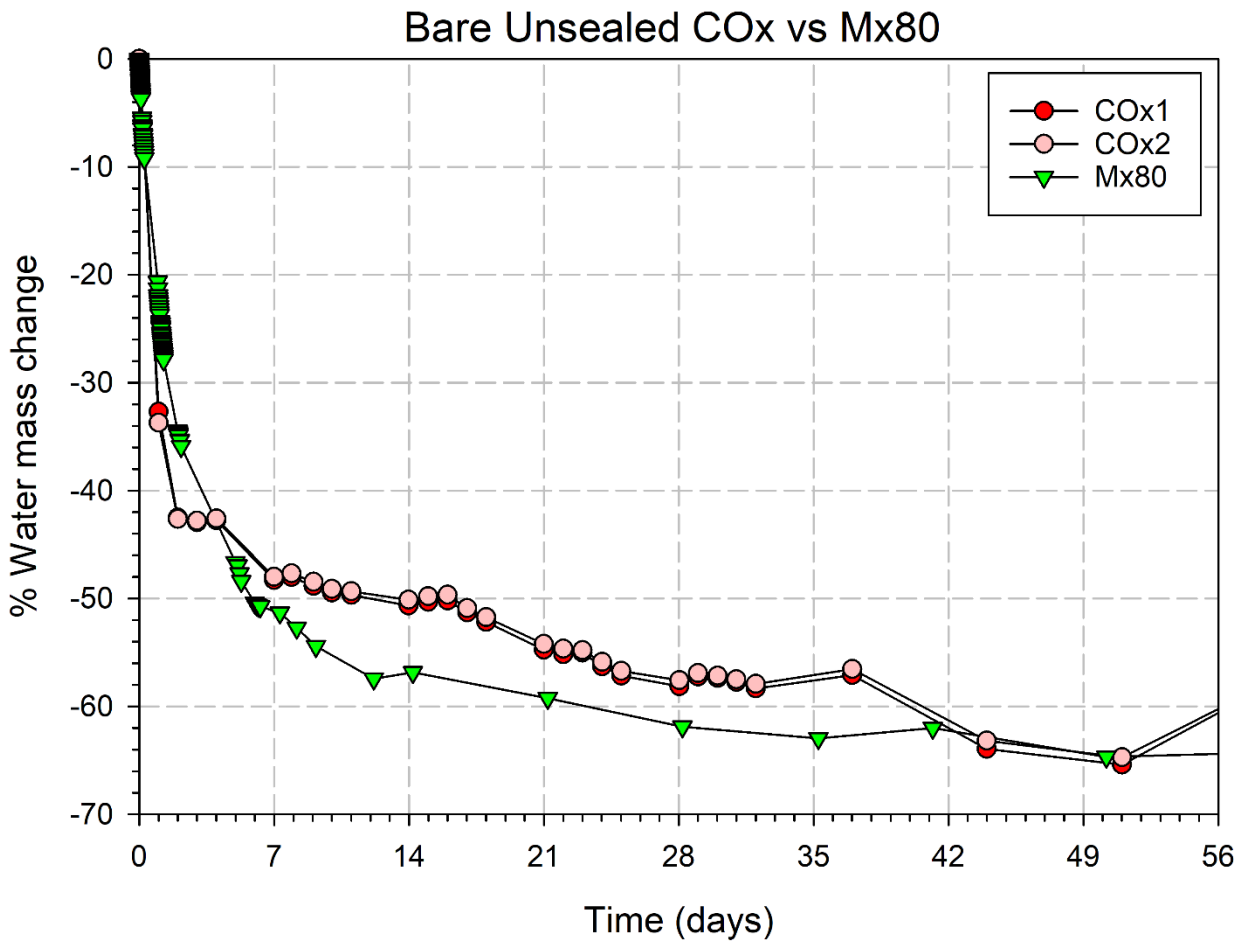


Figure 14 Comparison of water loss between two samples of Callovo-Oxfordian claystone (COx) and a sample of Mx80 bentonite (Mx80) over a period of 8 weeks. *BGS © UKRI*

4.4 DESATURATION PREVENTION FACTOR ($DSPF_{10}$)

One aim of the study was to quantitatively assess the effectiveness of different sample preservation methods. To do this, a desaturation prevention factor ($DSPF_{10}$) system has been implemented. Assuming an acceptable level of desaturation to be 10%, and taking the air-dried samples as a baseline, this system assigns a value to each method based on how long it can maintain an acceptable moisture level in a geological sample. DSPF is taken as the shortest time for 10% water mass to be lost (Figure 15), the results are shown in Table 1.

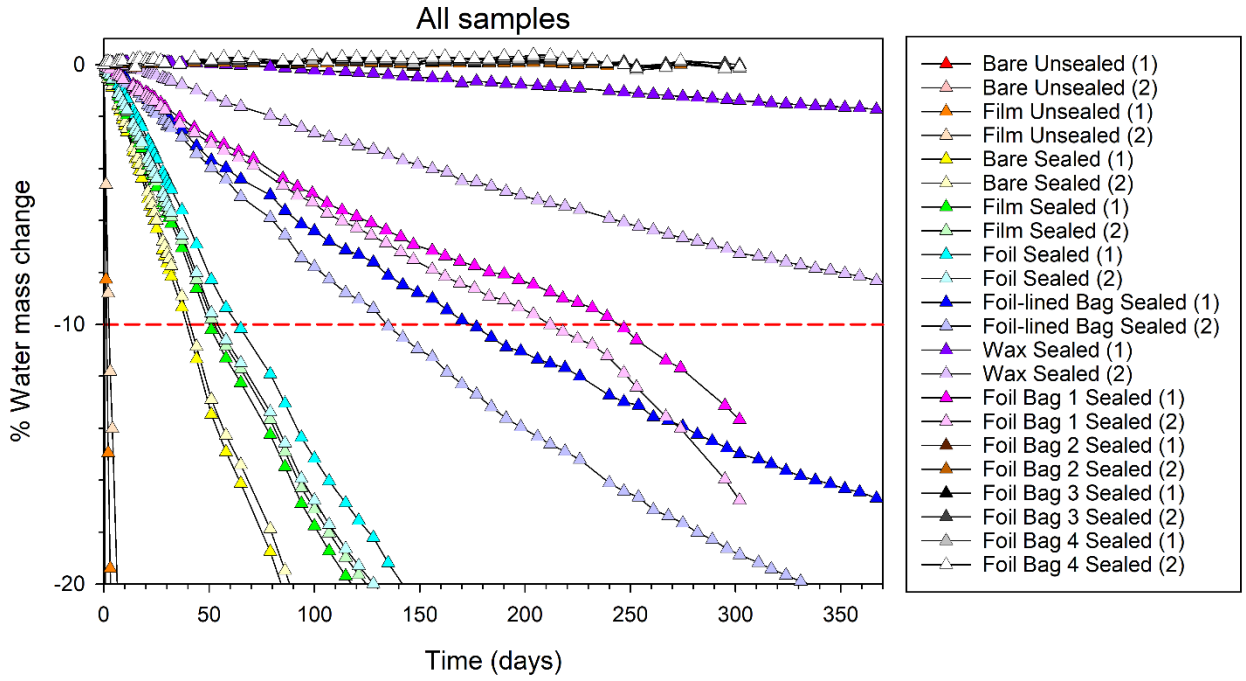


Figure 15 Water mass change observed across all samples with 10% water loss reference line for determining $DSPF_{10}$ factor. *BGS © UKRI*

Preservation Method	Fastest time to lose 10% water (days)	$DSPF_{10}$ Factor
Bare Unsealed	0.231	1
Film Unsealed	1.23	5
Bare Sealed	38.9	165
Film Sealed	50.9	215
Foil Sealed	53.8	227
Foil-lined Bag Sealed	135	570
Wax Sealed	493 (est.)	2086 (est.)
Foil Bag 1	212	897
Foil Bag 2	Not reached	2500+
Foil Bag 3	Not reached	2500+
Foil Bag 4	Not reached	2500+

Table 1 Average time taken to lose 10% water content and $DSPF_{10}$ Factor for each preservation method. *BGS © UKRI*

The $DSPF_{10}$ factor of each preservation method represents how many times longer that method can prevent a water loss of less than 10% of the original water mass when compared against leaving the sample open to air. Therefore, $DSPF_{10}$ represents how effective each method is. The bare, unsealed control samples therefore have a $DSPF_{10}$ factor of 1, losing 10% of its water within 0.23 days (Table 1). Wrapping the sample in clingfilm improved the storage time by approximately 5 times to 1.23 days before 10% of the original water has been lost. The introduction of vacuum-sealing increased the $DSPF_{10}$ factor to between 165-227 for bare, clingfilm-wrapped and foil-wrapped samples. The foil-lined bag had a significantly higher $DSPF_{10}$ factor of 570. The wax-sealed samples did not lose 10% of their water content within the year-long duration of the study but have been extrapolated to have a $DSPF_{10}$ factor of 2086. Of the four bag types sealed using the WeVac device, the three sandwiched aluminium/mylar type pouches did not lose more than 10% of their water so were assigned a factor of 2500+ and the metallised bag had a factor of 897. Figure 16 shows the data in graphic form. Note that foil bags 2-4 offer the greatest protection against sample drying, and the true $DSPF_{10}$ is much greater than 2500.

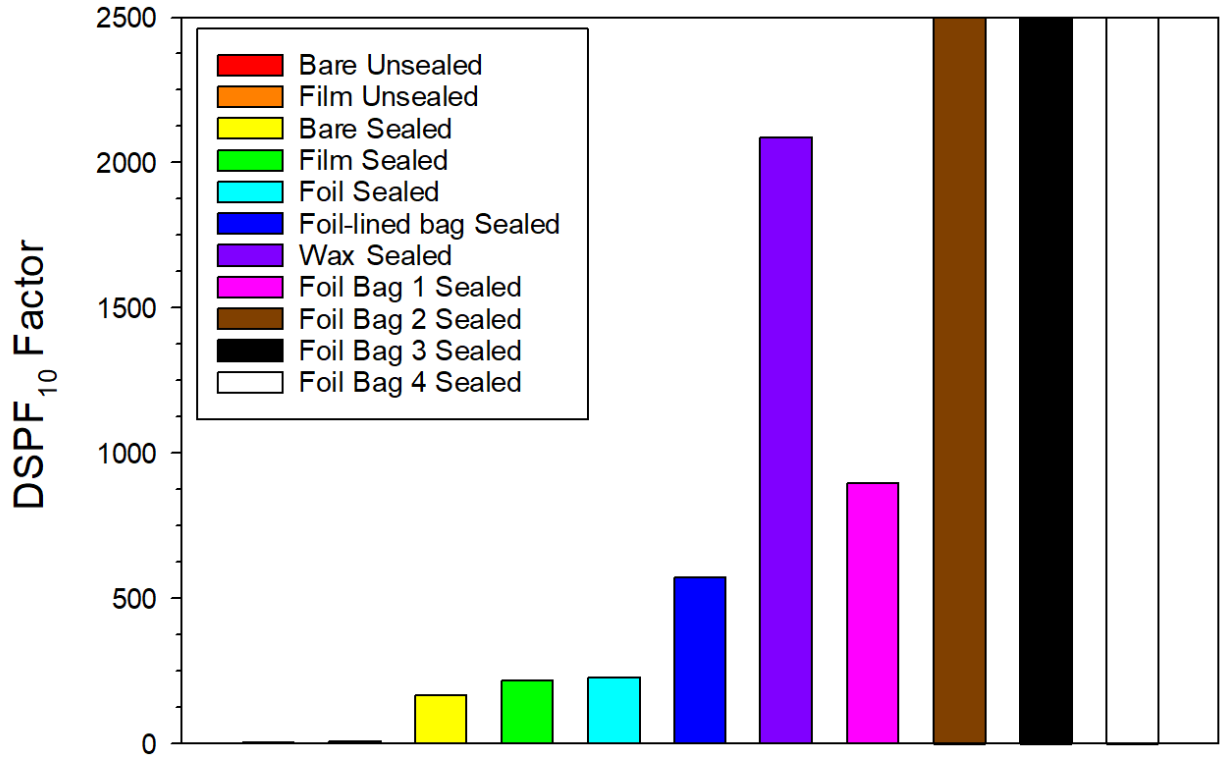


Figure 16 $DSPF_{10}$ factor for various preservation methods. *BGS © UKRI*

5 Discussion

The current study was not initiated to be a full scientific quantification of the effectiveness of different sample preservation methods. In this study we aimed to highlight the differences expected for different methods. Therefore, samples were not manufactured to be identical in size to remove this as a variable when comparing data. Each sample would have had a different surface area, and it has been noted that samples ranged in weight between 37g and 176 g, with an average of 71 g. Bearing this in mind, the study still achieved reliable data that shows the effectiveness of sample preservation techniques and highlights which of these are effective. It is concluded that the differences in the geometry of the starting samples did not have a detrimental influence on the results.

The study used two different clay-rich materials. The overall result seen after 50 days shows that moisture loss was consistent for COx and Mx80. Therefore, it is concluded that the type of clay-rich material may not be a significant influence on the performance of the different preservation materials. However, non-clay rocks may behave differently.

The current study has highlighted how important sample preservation is. In clay-rich rocks where considerable variations in THMCG performance can occur with small variations in saturation, even 1 hour exposure is enough to change clay-rich rocks. This needs to be considered when cores are logged and/or samples are manufactured. The use of clingfilm for short-term storage during sample preparation is not sufficient to protect the sample and it is advised that vacuum sealing in Mylar should be adopted during characterisation and sample preparation.

It must be noted that the term Mylar is not a guarantee of a good sealing material. All the foil bags used in this study were sold as being Mylar by different suppliers. One of these was a metallised mylar material where metal is sprayed onto plastic to give a foil effect (Figure 1i). This did not have a performance matching the other four foil bags, all of which comprised of a layer of aluminium sandwiched between mylar plastic. Therefore, care is needed when selecting a supply of foil bags.

6 Conclusions

This study has provided important implications surrounding the storage of geological samples as well as the measurement of geotechnics. When no mitigation measures are taken to prevent water loss from samples, rapid desaturation occurs, altering the properties of the rock. Exposure to the atmosphere for just one hour can have a noticeable change in sample saturation, and therefore exposure time should be minimised as much as possible. Various methods of preserving samples have been tested. Vacuum sealing in plastic sous vide bags and wax sealing both significantly improve the retention of moisture in samples. However, the best method has been found to be using a chamber vacuum sealer with sandwiched aluminium/mylar layer pouches. The loss of water in these samples was negligible over the duration of the study clearly demonstrating the effectiveness of this method. The desaturation prevention factor ($DSPF_{10}$) parameter was devised to quantify the improvement each sample preservation method provides. Short-term storage of samples in clingfilm was shown to only provide a marginal level of protection ($DSPF_{10}$=5), and it is advised that vacuum sealing in Mylar bags is adopted during sample preparation. The oven-drying of samples has determined that 6-7 days is required to remove all moisture from clay samples at 105°C (if preparing a sample for geotechnical analysis), although this is dependent on the size of samples. It is therefore recommended that samples are oven-dried at 105°C for at least one week to determine geotechnical properties for typical dimensions of samples used in THMCG experiments. If drying a sample at a lower temperature (e.g. to ensure no clay mineral alteration, or for a non-geotechnical technique), then longer drying times will be required.

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