ABSTRACT

Concrete derived hyper-alkaline leachate creates calthemite straw stalactites, properties of which are compared to speleothem straws

GARRY K SMITH, Newcastle–Hunter Valley Speleological Society

Calthemites are secondary deposits, consisting primarily of calcium carbonate ($CaCO_3$), derived from concrete, mortar or lime. They are very similar in composition and form to speleothems in limestone caves, however beneath human-made concrete structures. $CaCO_3$ deposition occurs when carbon dioxide (CO_2) is a reactant as opposed to a product. Calthemite deposits typically take on the shapes and forms of speleothems e.g. stalactites, stalagmites, straws and flowstone.

This study compares calthemite straw stalactites with speleothem straws of comparable outside diameter and length. Calthemite straws grow in length, hundreds of times faster than speleothem straws in caves (Smith 2016). Measurements of both types of straws found that on average, outside diameters were within an equivalent range, however calthemite straws had a far thinner wall thickness. This physical attribute equated to calthemite straws on average having less than 50% the mass of speleothem straws. Hypotheses explaining the reason for such a disparity are considered.

Also measured was the carrying capacity and subsequent mass of $CaCO_3$ deposited from hyperalkaline solution (pH 13) leaching from a concrete structure.

Leachate solution drop breaking free from a calthemite straw.

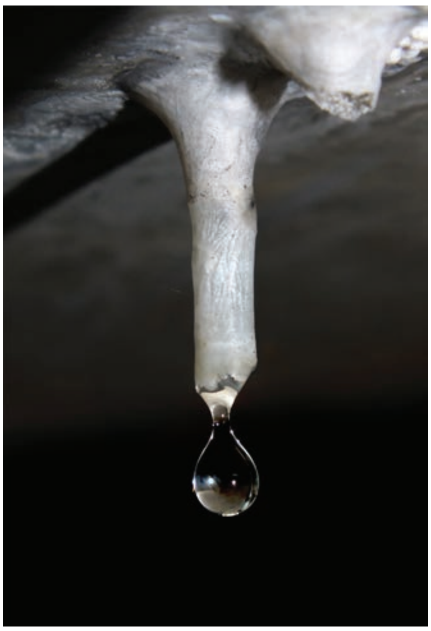
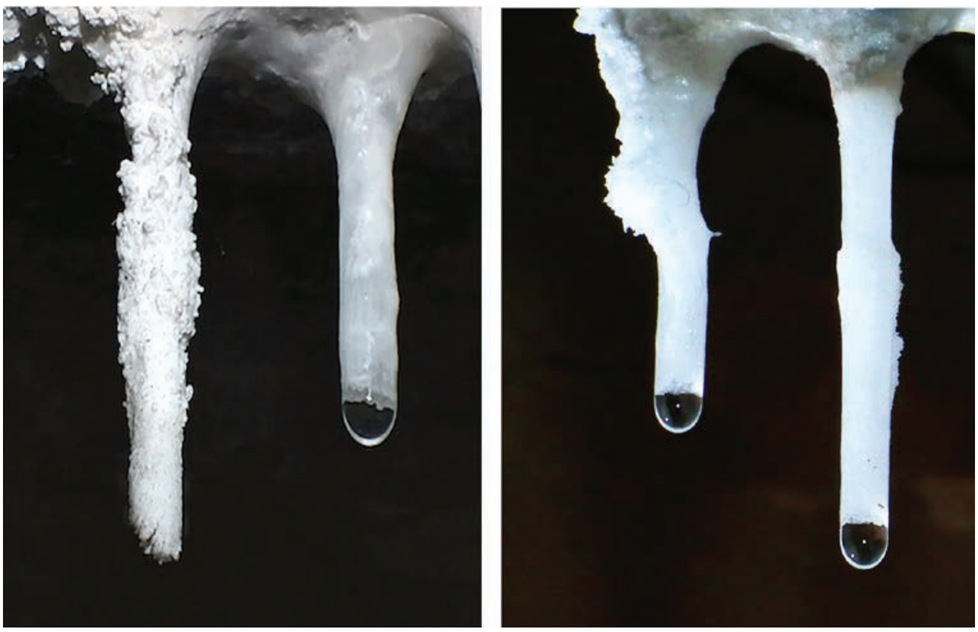


Figure 1. Calthemite straws on left, are similar to speleothem straws on right. Both are composed of calcium carbonate and approximately the same diameter, but the linear masses are significantly different.



Comparing calthemite to speleothem straw stalactites; solution drop mass and calcium ion saturation

Garry K. Smith

3 Spinnaker Ridge Way, Belmont NSW 2280 Australia
E-mail: gksmith29@icloud.com

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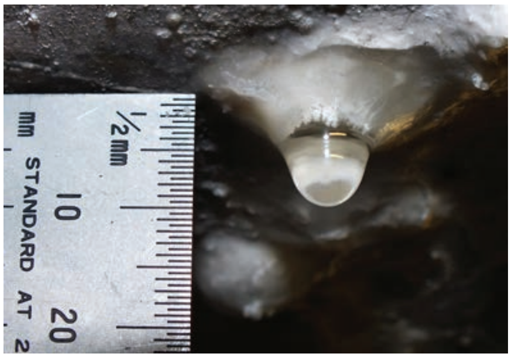


Figure 2. A cone shape deposit forms as the base of the new straw transitions into a parallel tube.

Introduction

Beneath concrete structures, calcium carbonate is precipitated from hyperalkaline solution to create calthemite deposits which mimic cave speleothems (Smith, 2016). Under ideal conditions, calthemite straws can grow in length hundreds of times faster than speleothem straws due to the greater calcium ion (Ca^{2+}) carrying capacity of the hyperalkaline leachate solution and different chemistry involved.

Ford and Williams (2009) state that, ‘Rates of growth are usually quoted in terms of the extension of a given form rather than its accumulation of mass. Straw stalactites “grow” fastest because they have the greatest extension per unit of areas deposited.’ Growth rates between 0.2 and 2 mm per year are quoted for speleothem straws (Ford and Williams 2009), where as calthemite straws can grow at rates up to 2mm per day (Smith 2016).

At first glance calthemite and speleothem straws look very similar (Figures 1), but on closer investigation there are quite a few physical differences, despite the fact that both are primarily made of calcium carbonate (usually calcite) and deposited from dripping leachate solution. This study compares the physical attributes (mass and diameter) of calthemite and speleothem straws. Also investigated, is the mass of calcium carbonate deposited by hyperalkaline leachate, discharging from straws beneath a concrete structure.

Many samples of different diameter were measured to determine the average mass per linear length of calthemite and speleothem straws. Individual solution drops were accurately

Figure 3. Typical cross sections of calthemite straws. They have a thinner wall, are more fragile and have a less dense crystal structure than speleothem straws. Image scale divisions are in mm.

Figure 4. Typical cross section of speleothem straws.

	Calthemite Straws		
Straw Length mm	Weight in g	Average weight (g) / unit length (mm)	Average straw diameter in mm
17.0	0.092	0.0054	3.70
23.0	0.079	0.0034	3.90
44.0	0.213	0.0048	4.10
33.5	0.288	0.0086	4.30
19.0	0.240	0.0126	4.35
58.5	0.460	0.0079	4.45
28.5	0.124	0.0044	4.50
16.0	0.084	0.0053	4.60
14.5	0.146	0.0101	4.60
10.0	0.061	0.0061	4.60
35.0	0.239	0.0068	4.90
20.0	0.263	0.0132	5.00
21.0	0.208	0.0099	5.10
38.5	0.607	0.0158	5.20
30.0	0.386	0.0129	5.30
27.5	0.489	0.0178	5.40
Total 436.0	3.979		
	Average g/mm using overall length		
		0.0091	

Table 1. Measurements of calthemite straws

	Speleothem Straws		
Straw Length mm	Weight in g	Average weight (g) / unit length (mm)	Average straw diameter in mm
45.5	1.096	0.0241	4.50
44.7	1.166	0.0261	4.60
36.0	0.608	0.0169	4.90
33.8	0.769	0.0228	4.90
60.7	1.572	0.0259	5.00
54.5	1.444	0.0265	5.00
29.2	0.872	0.0299	5.00
46.8	1.201	0.0257	5.10
35.8	0.943	0.0263	5.15
50.0	1.457	0.0291	5.25
66.0	1.604	0.0243	5.30
60.5	1.429	0.0236	5.35
46.5	1.217	0.0262	5.35
47.0	1.327	0.0282	5.55
49.5	1.707	0.0345	6.00
36.5	1.435	0.0393	6.45
Total 743.0	19.847		
	Average g/mm using overall length		
		0.0267	

Table 2. Measurements of speleothem straws

weighed to determine the relationship between a calthemite straw's diameter and the solution drop mass. Solution was collected and evaporated to obtain the mass of calcium carbonate deposited from solution, taking into consideration any deposition on the straw. These data are cross-referenced to a previous study at the same location, where calthemite straw growth rates versus drip rates, was recorded (Smith, 2016).

Straws of both types begin their development as a large diameter calcium carbonate ring around the area which has been wetted by solution on the underside of the concrete structure or cave ceiling. The exact size of the $CaCO_3$ crystal ring depends on the wettability of the host surface and surface tension supporting the drop. Over time a cone shaped $CaCO_3$ deposit forms (Figure 2), as the base of the new straw transitions into a cylindrical parallel tube growing down from the face of the host ceiling. The tube becomes parallel when an equilibrium is reached between the straw diameter, solution surface tension and other influencing factors as identified in this paper.

Study Site

A concrete building constructed in Belmont, NSW, Australia during 2008 (9 years old at the time of this study) included a partly enclosed undercover car park with supermarket area above. Straw stalactites began growing within months of the building being completed. Poorly constructed roof guttering traps rainwater and leaks a continuous flow through a minute hole, onto the concrete structure. The water then finds its way into the concrete, following microscopic cracks and internal porosity, gaining solutes until it emerges from cracks in the car park ceiling, where the stalactite straws are growing.

Although the stalactites were in a difficult location to access due to vehicle movement, the constant supply of solution water all year round, made it ideal to study the mass of $CaCO_3$ deposited from hyperakaline solution and drip mass compared to calthemite straw diameter.

Samples and methods

Fifteen calthemite straws were removed from the underside of the concrete structure. Many were still active straws so extra care was taken to avoid the highly corrosive hyperalkaline solution (pH 13) contacting hands or skin. Once removed the straws were placed in an oven at 60–70°C, to evaporate any solution still inside the straw. The straw length was trimmed to remove any portion with significant variation in outside diameter. The weight (mass) and length of each parallel straw section, was accurately measured with jewellers scales and vernier callipers to obtain average mass per unit length (mg/mm).

Several of the caves at Timor, north of Newcastle had been vandalised many years ago and provided an opportunity to make measurements of broken speleothem straws in situ. In addition, permission was granted to collect some broken straws from Cliefden Caves, NSW. This allowed a meaningful comparison between speleothem and calthemite straws.

Both speleothem and calthemite straws have small irregularities inside and outside, making neither absolutely uniform (Figures 3 and 4). Only straws with a near parallel outside diameter were considered in this study. Any straw showing signs of diameter enlargement due to $CaCO_3$ deposited from solution film or solution trickling down the outside was rejected from the sampling. The outside diameter of all straws sampled, varied between 3.7 to 6.45 mm (Tables 1 and 2).

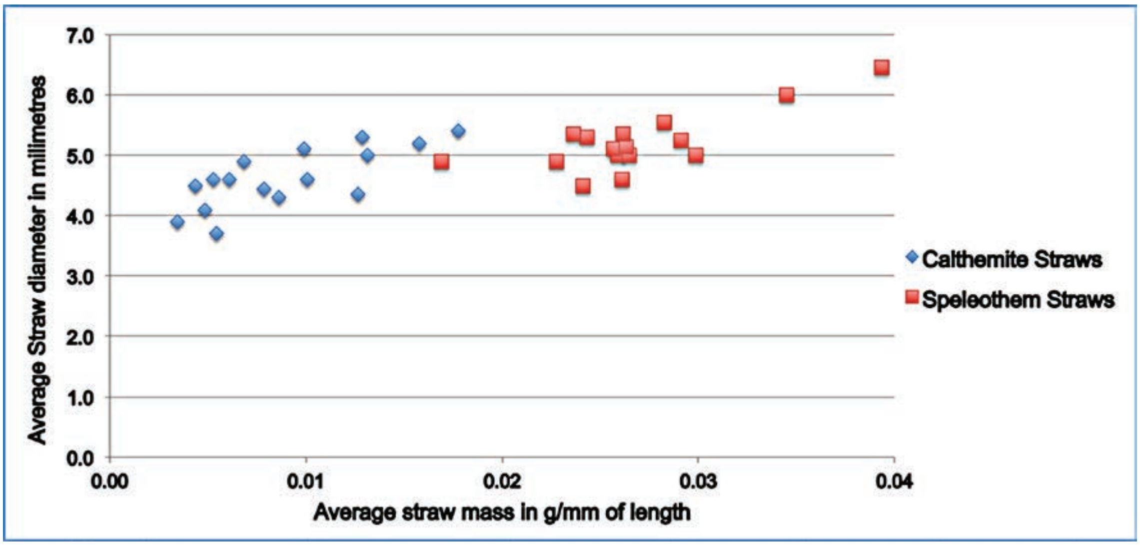
Two precision jeweller scales (0–10g and 0–30g), capable of weighing to 0.001g, were used to measure the mass of containers and their content. A precision engineering ruler and vernier calliper were used to measure the lengths and diameters of straws to within 0.05mm.

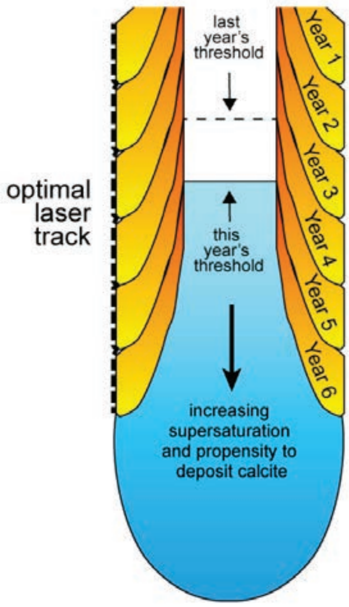
Results of straw linear mass measurements

Measurements over all straws sampled revealed speleothem straws are on average 2.9 times heavier than calthemite straws of equivalent outside diameter. Speleothem straws averaged 26.7 mg/mm (Table 2), while calthemites straws averaged 9.1 mg/mm (Table 1). This comparison is however biased toward the speleothems as two significantly larger diameter samples were collected (Figure 5). Considering only straws with outside diameters ranging between 4.9mm–5.1mm, the average speleothem straw is 2.47 times heavier than calthemite straws. Calthemite straws are on average just 40.7% the mass of speleothem of equivalent outside diameter.

In general speleothem straws have a denser calcite structure and a greater wall thickness, and thus a smaller solution canal down the centre than calthemite straws (Figures 3 and 4). The calthemite straws are generally quite fragile due to their thin wall thickness.

Figure 5. Straw mass (gm) per unit length (mm)





Left: Figure 6. Speleothem straw growth pattern. Image by Paul et.al. 2013. As the drip hangs from the tip, a combination of greater CO_2 degassing and lower nucleation energies occurs at the drip/straw-tip interface, producing wider layers at the edge of the straw.

Figure 7. Masking tape holds container against underside of concrete to collect hyperalkaline solution dripping from a straw. The mass (gm) of clean container is written on outside.

The large disparity in straw mass per mm, between calthemite and speleothem straws, appears to be due to the difference in the $CaCO_3$ deposition process, as discussed by Smith 2016. When $CaCO_3$ deposition occurs on a speleothem, CO_2 must defuse out of the leachate drop, thus the diffusion of the gas from the drop occurs slowly and more evenly throughout the drop. This causes $CaCO_3$ to be deposited along the inner wall of the straws solution canal as well as at the straw tip (Paul et al. 2013, Figure 6).

Therefore, the speleothem straw grows with a smaller canal and greater wall thickness than a calthemite straw. On the other hand, calthemite leachate has a higher Ca^{2+} carrying capacity and its chemistry facilitates much faster deposition of $CaCO_3$ at the drop surface in contact with atmospheric CO_2. There is not enough time for CO_2 to diffuse evenly throughout the solution drop to cause deposition for much distance inside the straw's solution canal. Therefore, the straw is fast growing in length with little $CaCO_3$ deposition in the solution canal. These two factors cause calthemite straws to grow more quickly in length but lack the wall thickness of speleothem straws.

Solution drop mass

As part of this study it was decided to investigate what factors influenced the mass of a leachate solution drop falling from a calthemite straw. In particular. to identify how or if, a straw's outside diameter is governed by the solution's surface tension, which in turn may be influenced by Ca^{2+} ion saturation and environmental parameters.

Collection containers were held with masking tape, hard against the underside of a concrete structure to capture a counted number of drops falling from a calthemite straw of known diameter (Figure 7). Evaporation of solution was negligible as atmospheric air could not freely enter the container during collection of the drops (less than 30 minutes). These samples were only collected in the late evenings after the shopping centre had closed, when there was minimal air movement, or vibration in the concrete structure due to vehicle movement and staff moving heavy stock pallets. This provided more consistent drop samples.

There were 48 samples collected covering a range of different straw diameters and drip rates. From the counted number of drops collected in each-container, the average drop mass was plotted against the straws diameter (Figure 8). The observed relationship is approximately linear.

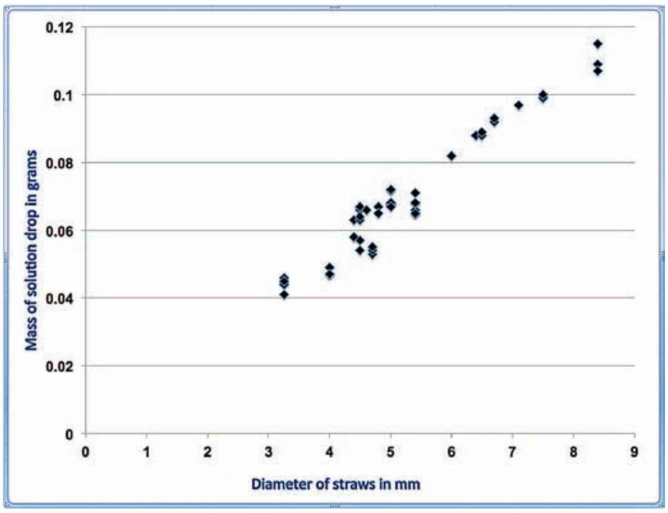


Figure 8. Graph comparing the mass of calthemite solution drops (grams), to the outside diameter (mm) of the stalactite straws from which they fell. The solution mass included Ca2+ and any other dissolved minerals.

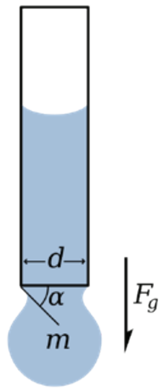


Figure 9. Solution drop mass calculation diagram. Sourced from Wikipedia. https://en.wikipedia.org/wiki/Drop_(liquid)

The theoretical mass *m* of a drop hanging from the end of a straw (Figure 9) can be found by equating the force due to gravity ($F_g = mg$) with the component of the surface tension in the vertical direction ($F_\gamma \sin \alpha$) giving the formula;

$mg = \pi d\gamma \sin \alpha$

where α is the angle of contact with the tube, and g is the acceleration due to gravity. Where d is the tube diameter in metres.

The limit of this formula, as α goes to 90˚, gives the maximum mass of a pendant drop for a liquid with a given surface tension γ. Note that the SI units for γ are millinewtons per metre (mN/m).

$mg = \pi d\gamma$

This relationship is the basis of a convenient method of calculating surface tension. More sophisticated methods are available, to take account of the developing shape of the pendant as the drop grows. More information can be sourced from 'Pendant drop test' https://en.wikipedia.org/wiki/Drop_(liquid)

Curl (1972) found that surface tension is sensitive to temperature changes and impurities in the solution. Impurities may be in the form of calcite crystals (rafts) which have been observed on the calthemite solution drip surface (Smith 2016) and their presence influenced by drip rate. There may well be minerals or other impurities within the solution, which influence the surface tension. Other impurities in speleothem straw solution may include: Mg, Sr, SiO_2 and SO_4, clay particles and organic matter (Borsato 2016)

In theory the 'drop mass' from a known diameter stalactite straw can be calculated using the formulae as detailed above, however there are many variables influencing solution 'surface tension' across a range of calthemite straws. Surface tension influencing factors may well include; saturation of Ca^{2+}, solution pH and impurities, serration of crystal structure around the straw tip (altering length of contact surface), solution temperature and $CaCO_3$ rafts on drip surface (Figure 10). Drips may be induced to fall prematurely by: solution flow rate, pulsation of solution, concrete structure vibration (movement of goods and people in supermarket) and air movement. If a drop is induced to fall prematurely, without reaching its maximum potential mass, this would translate into a false surface tension calculation when data is entered into the formulae.

Calculated 'surface tensions' from the collected calthemite leachate solutions, varied between 35.88 and 43.72 mN/m over an atmospheric temperature range of 15 to 25°C. As a comparison pure water at 20°C is 72.86 ±0.05 mN/m (Pallas and Harrison, 1990).

Data collected did not definitively indicate that leachate 'surface tension' had any appreciable influence on a straw's outside diameter. However, as determined in a previous study (Smith 2016), slow dripping calthemite straws tended to be slightly larger in diameter than fast dripping straws. This may well be due to the drop surface angle α remaining larger for a longer period as the drop forms and deposits $CaCO_3$ at the straw tip. An example of a calthemite straw with changes in diameter, is shown in Figure 10 and an example of a straw growing in diameter in Figure 12. It is most likely that observations of calthemite straw diameters having a relationship to drip rate, may also be mirrored in speleothem straw's diameter also being influenced by solution drip rate.

However, an extra fast drip rate does not instantaneously create a small diameter straw or vice versa for a slow drip rate. A straw changes diameter gradually as it grows in length. If we look at growth rates (Smith 2016), it may take a matter of days or weeks for a calthemite straw to significantly change diameter as a result of a change in drip rate. A speleothem straw may take many months to significantly change diameter.

Figure 10. Variations in calthemite straw diameter, due to changes in solution surface tension, influenced by solution saturation of $Ca(OH)_2$ and usually associated with changes in solution supply (drip rate).

Background and methodology to determine $CaCO_3$ deposition from Hyperalkaline Leachate

This part of the study was undertaken in order to try and understand how much $CaCO_3$ is deposited by hyperalkaline leachate as it is often cited that the appearance of calthemite deposits under concrete structures is a sign of degradation of concrete, causing a loss of strength. A search of literature found that according to Fagerlund (2000), *'About 15% of the lime has to be dissolved before strength is affected. This corresponds to about 10% of the cement weight, or almost all of the initially formed $Ca(OH)_2$.'* This would mean that a large amount of $Ca(OH)_2$ must be leached from the concrete before structural integrity is affected. The other issue however is that leaching away $Ca(OH)_2$ may allows the corrosion of reinforcing steel to affect structural integrity.

The Ca^{2+} carrying capacity of speleothem leachate is approximately 200 times less than calthemite leachate (Sefton 1988), so only calthemite drip water was sampled in this study. Small containers were taped over short active calthemite straws on the underside of the concrete structure, so as to capture solution drips over periods of time ranging between 1–3 days. The containers were held with masking tape, hard up to the flat underside of the concrete to restrict the ingress of fresh air containing CO_2, which would cause deposition of $CaCO_3$ at the straw. This attachment method did not provide a perfect airtight seal, so variations between container and atmospheric pressure could equalise, without influencing the outflow of leachate solution from the straw. This attachment method also minimised solution evaporation, however it was noted that on each occasion upon removing a container, there was a thin calcite raft floating on the collected solution. This indicated that some atmospheric CO_2 was entering the containers and allowing $CaCO_3$ to precipitate at the solution surface.

The length of each straw was recorded prior to and upon removal of each leachate collection container. These measurements were critical when identifying if $CaCO_3$ was deposited on the straw instead of remaining in solution collected in the container.

Sample number	Mass (g) of calthemite solution collected	Mass (g) of CaCO3 remaining after solution evaporated	Calculated mass of CaCO3 deposited as straw growth	Mass (g) CaCO3/kg of solution (including straw growth).	Calculated average Time (min) between drops
1	7.304	0.018	0.0036	2.9500	9.03
2	16.830	0.041	0.0071	2.8580	3.98
3	6.434	0.017	0.0071	3.7460	10.70
4	11.909	0.030	0.0142	3.7110	11.70
5	28.740	0.101	0.0142	4.0080	7.14
6	12.924	0.039	0.0142	4.1160	10.84
7	13.203	0.052	0.0107	4.7450	15.64
8	48.692	0.039	0.0000	0.8010	0.15
9	28.964	0.020	0.0000	0.6905	0.15
10	2.659	0.003	0.0000	1.1282	1.55
11	1.748	0.001	0.0000	0.5721	2.46

Table 3. Calthemite leachate samples were evaporated to determine $CaCO_3$ deposited from solution. Also considered is deposition of $CaCO_3$ at the straw tip. Samples 8 –11 had very fast drip rates with no measurable $CaCO_3$ deposition at straw tip.

The collected solution was weighed in the container and the empty container mass deducted to ascertain the solution mass. The solution was left in the container and allowed to evaporate in the sun over several days. The dry container (with $CaCO_3$ deposited inside), was then accurately weighed and the container mass deducted to determine the $CaCO_3$ mass. The accurate mass of each collection container had been recorded prior to commencing the study.

The diameter and change in length of straws was recorded and factored into calculations to arrive at the overall mass of $CaCO_3$ deposited from the hyperalkaline solution (Table 3).

Samples 1 to 7 (Table 3) were collected during a relatively dry period when drip rates were slow at between 4 and 16 minutes per drop. It took several days to collect sufficient sample in the containers. Samples 8 to 11 were collected after a severe rain event, which significantly increased the drop rate of all active straws. Those sampled were faster than one drop every 2.5 minutes and even as fast as one drop every 3 seconds. This meant there would be no straw growth (Smith 2016) and sufficient solution could be collected from each straw in less than 30 minutes.

As was expected the mass of $CaCO_3$ deposited per kg of hyperalkaline solution was significantly less in the period with an abundance of leachate. The greater flow rate through the concrete after the rain event, meant there was limited time to leach calcium hydroxide from cracks and micro pores within the concrete and transport Ca^{2+} to the under surface of the structure.

The linear relationship of time between drips and $CaCO_3$ deposited from solution (Figure 11), depicts the dissolution kinetic of the

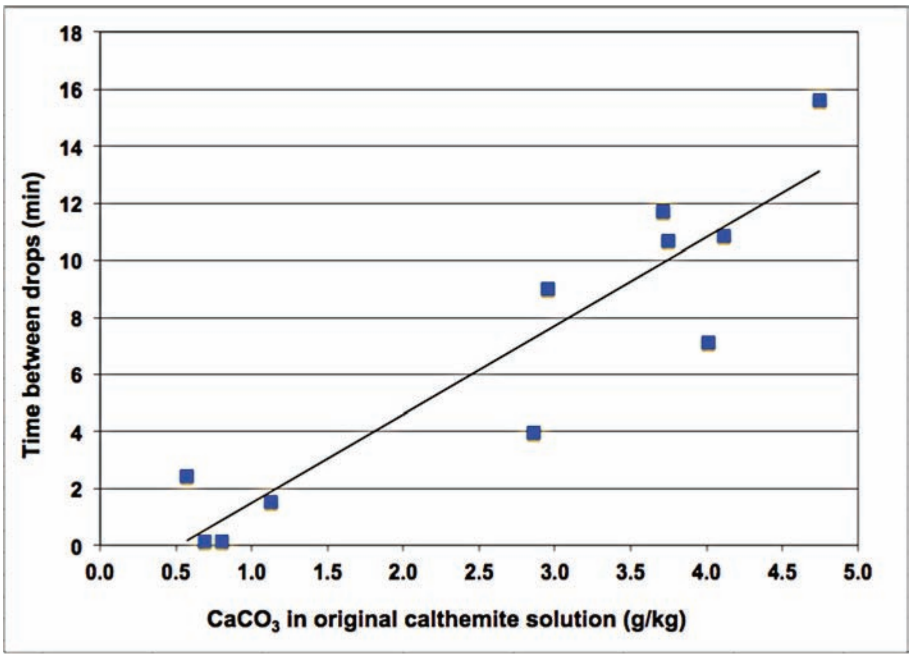


Figure 11. Relationship between time (min) between drops and mass (g) of $CaCO_3$ precipitated from mass (kg) of solution. Linear regression is shown.

concrete: as the residence time of the fluid inside the concrete increases we observe a steady and linear increase in the Ca^{2+} concentration in solution (deposited as $CaCO_3$). Overall the mass of $CaCO_3$ originally present in the hyperalkaline solution varied greatly from 0.572 to 4.745g/kg of leachate.

The regression line on the graph (Figure 11) highlights that there is a reasonable deviation in sampled solution concentrations, which are likely influenced by other factors besides drip rate (flow-rate). It is reasonable to surmise that solution seepage path, resonance time, and availability of Ca^{2+} along the seepage path play a large part in the leaching of Ca^{2+} from concrete structures. These results indicate there is no simple way of accurately calculate how much Ca^{2+} is being leached from concrete and deposited as $CaCO_3$ by measuring leachate flow rates.

As a comparison, Moore (1962) collected solution from a speleothem stalactite dripping at a 23 second interval and measured the flow rate at 30 ml/hour, in a cave atmosphere at 12.7˚C and near 100% humidity. Calcite rafts were forming on the surface of the pool beneath the stalactite, so he assumed that the drip solution was near 100% saturation. Moore calculated that the total calcite deposition from the solution was 0.014g/day which equates to 0.0194g/kg of speleothem leachate. This figure is in line with the expectation that speleothem solution Ca^{2+} ion saturation is an order of magnitude hundreds of time less than calthemite solution.

Conclusion

On average calthemite straws examined had thinner wall thickness and a less dense calcium carbonate structure than speleothem straws of equivalent diameter. It appears that the chemistry and slower deposition rate of calcium carbonate from mildly alkaline solution (low Ca^{2+} saturation) associated with limestone cave (speleothem) straws, creates a more-dense structure than hyperalkaline solution creating calthemite straws. This is well explained by the speleothem straw growth pattern image by (Paul et.al. 2013). Measurements revealed that calthemite straws are on average just 40.7% the mass per linear length of speleothem straws of equivalent outside diameter.

Calthemite straws can grow up to 2mm per day when the drip rate is 11 minutes between drops. As determined (Smith 2016), when the drip rate is more frequent than one drop per 11 minutes, the deposition rate (gain in length) is reduced. This present study identifies that the changes in leachate residence time within the concrete (expressed by the drip rate), greatly influences the uptake of calcium ions in solution and subsequent amount of $CaCO_3$ deposited at the straw tip. Hence in periods of fast flow, the concentration of Ca^{2+} in solution is less than when there is a lower solution flow rate.

The time a drop remains at the tip of a calthemite straw affects the ability of solution to uptake carbon dioxide from the atmosphere and deposit $CaCO_3$, however the leachate saturation also plays a significant roll. The Ca^{2+} ions carried by solution is influenced by the leachate solution pH, flow rate, length of seepage path and time taken to travel through the concrete's micro cracks and pores, and availability of Ca^{2+} along the seepage path.

The mass of a leachate drop falling from a known diameter calthemite straw is directly proportional to the end diameter of the straw from which it fell; i.e. the larger the straw diameter, the greater the drop mass.

Figure 12. A longer period between drips allows more time as a developing drops bulges, to deposit $CaCO_3$ at a greater circumference to increase straw OD. Note the calcite raft lattice on the calthemite straw drop.

The drop mass could not be accurately predicted without knowing the solution surface tension at the precise time. However, there are many variables (temperature, impurities etc), which can influence surface tension and in turn the drop mass. Provided the possibility of a drop prematurely falling because of vibration, air movement or other factors, a drop mass could be approximately calculated using the formula $mg = \pi d\gamma$ if the straw diameter and leachate solution surface tension γ is known.

There appears to be sufficient variation in leachate surface tension to have a small influence over the maximum diameter range of calthemite compared to speleothem straws. Calthemite leachate drip rate appears to influence the resulting calthemite straw outside diameter, and the drip rate may well influence a speleothem straw's diameter.

Sampling and analysis of solution drip rate from straws and the Ca^{2+} ions leached from concrete (precipitated as $CaCO_3$) showed that a slower drip rate had a higher solution saturation. However, the deviation of results, indicated that other factors such as solution seepage path, resonance time and availability of Ca^{2+} along the seepage path, has an influence over the calthemite leachate saturation.

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