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Complete List of Authors:	Acikel, A. S.; Monash University, Civil Engineering Gates, Will; Deakin University Singh, R.M.; University of Surrey, Civil and Environmental Engineering Bouazza, Abdelmalek; Monash University Fredlund, Delwyn; Golder Associates Ltd., Geotechnical Engineering Rowe, Kerry; Queen's University, Civil Engineering
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Time-Dependent Unsaturated Behaviour of Geosynthetic Clay Liners

A. S. Acikel[1], W. P. Gates[2] R. M. Singh[3], A. Bouazza[4], D. G. Fredlund[5], R. K. Rowe[6]

[1] Formerly Monash University, Melbourne, Australia; Tel: +61 3 9905 0538; Fax: +61 3 990 5 4944; Email: asliacikel@gmail.com

[2]Deakin University, Burwood, Australia; Tel: +61 3 9246 8373; Email: will.gates@deakin.edu.au

[3]University of Surrey, Surrey, United Kingdom; Tel: +61 3 9905 4981; Fax: +44 01483 68 9280; Email: r.singh@surrey.ac.uk

[4]Monash University, Melbourne, Australia; Tel: +61 3 9905 4956; Fax: +61 3 9905 4944; Email: malek.bouazza@monash.edu

[5]Golder Associates Ltd., Saskatoon, Saskatchewan, Canada; Tel: 1-306-667-1251; Fax: 1-306-665-3342; Email: delwyn.fredlund@gmail.com

[6]Queen's University, Kingston, Canada; Tel: +1 613 533 3113; Fax: +1 613 533 2128; Email: kerry.rowe@queensu.ca

Abstract

Three different chilled-mirror hygrometer test procedures were developed to investigate the time-dependent unsaturated behaviour of powdered and granular bentonite based needle punched geosynthetic clay liners (GCLs) on both the wetting and drying paths of the water retention curve (WRC). The GCLs structure and bentonite forms governed the effect of measurement time and duration as well as the time-dependent suction changes of the bentonite component at a constant gravimetric water content. A conceptual model is proposed to explain the observed time-dependent unsaturated behaviour of the GCLs. The model suggests that the cross-over points on WRCs correspond to the point where bentonite crystallite separation is maximised within the crystalline swelling regime of smectite, forming a 4-layer hydrate state where smectite interlayer spaces are filled with water. At gravimetric water contents below this point, the interlayer space dominated the suction while at higher water contents, meso- and macro-pores played increasingly important roles in determining the suction. The results reported herein provide further proof that the unsaturated behaviour of GCLs is largely controlled by the bentonite component.

Keywords: *Geosynthetics, GCL, unsaturated, bentonite, water retention curve, time-dependency*

1. *Introduction*

Geosynthetic clay liners (GCLs), typically 5 to 10 mm thick, are composed of a thin layer of sodium bentonite contained between two layers of geotextile, commonly held together by needle-punching (Bouazza 2002). They are widely used in lining systems of waste containment facilities to minimise the escape of pollutants into soil and groundwater. (Hornsey et al. 2010; McWatters et al. 2016; Rowe 2014; Touze-Foltz et al. 2016). GCLs are well suited for this application due to the low permeability of bentonite (the active component of GCL) when hydrated, usually by uptake of water from the underlying soil (Daniel et al. 1993; Rowe 2007; Rayhani et al. 2011; Rowe et al. 2011; Anderson et al. 2012; Chevrier et al. 2012). The final equilibrium gravimetric water content reached by the GCL component via hydration from the subsoil is therefore critically important to its success as a hydraulic barrier (Rayhani et al. 2011; Liu et al. 2015; Bouazza et al. 2017). Key to the hydration process is the GCL water retention curve (WRC) relationship which is fundamental for estimating the ultimate degree of saturation attained by GCLs and the equilibrium times required for hydration or dehydration.

While complex, quantifying the water retention behaviour of GCLs is crucial for the evaluation of their unsaturated hydraulic performance (Abuel-Naga and Bouazza 2010; Beddoe et al. 2010; Bouazza et al. 2013; Bannour et al. 2014; Acikel et al. 2015; Rouf et al. 2016). In this respect, several factors need to be taken into consideration including: (1) the time-dependent unsaturated behaviour of the bentonite component, (2) the time dependency of the suction measurement/control techniques when considering a partially confined highly plastic material such as bentonite, and (3) remembering that the GCL is a composite manufactured material with a tri-modal pore structure. Currently, there are insufficient published studies that specifically investigate the impact of the time dependency of the many factors influencing the unsaturated behaviour of GCLs as a composite material. The previous studies mostly aimed at obtaining water retention curves (WRCs) of GCLs at homogenized condition and did not point out the importance of the impact of measurement on the reported suction values. The suction values of unsaturated GCLs are not always in a homogenized condition in practice but they are in a time dependent suction range at given gravimetric water content due to the aging behaviour of the bentonite component (Schanz et al. 2010; Delage et al. 2006). Considering the conditioning (aging) and testing time dependencies of WRCs can lead to more analysis accuracy when the WRC is implemented in numerical analysis. Thus, the time dependency of unsaturated GCLs needs to be further investigated to improve in numerical modelling accuracy and the understanding of the unsaturated performance of GCL in field applications.

The objective of this paper is to explore the time dependency of the GCL unsaturated behaviour as affected by conditioning time, testing time and duration. Three different chilled mirror hygrometer test procedures were used on both the wetting and drying paths of the water retention curve of three GCL types to investigate the effect of conditioning and testing time and duration on total suction measurements with different levels of partial-confinements provided by the needle-punched fibres. A conceptual model is presented to describe observed time dependent unsaturated behaviour of GCLs in terms of conditioning and measurement time dependencies based on the chilled mirror hygrometer test results.

1.1. *Background – bi-modal vs tri-modal characteristic of bentonite and GCL*

Bentonite has a large range of pore sizes due to its structure which is comprised of quasi-crystals (discreet collection of crystallites or 2:1 layers), particles (collection of quasi-crystals), and aggregates (collections of particles) (Figure 1). Most clay mineralogists differentiate between interlayer (slit) pores and inter-particle pores, whereas in this usage inter-particle pores include all the pores that are not interlayer pores (e.g., Gates et al. 2012). Such differentiation has been useful to many researchers (Gens and Alonso 1992; Alonso et al. 1999; Sanchez et al. 2006; Delage 2007; Villar and Lloret 2008; Romero et al. 2011; Seiphoori 2014; Seiphoori et al. 2014; Navarro et al. 2015; Cui 2016; Sanchez et al. 2016) who described bentonite as having a bi-modal pore structure. The pores between bentonite aggregates (inter-aggregate pores) are generally within the macropore size range, and the pores within aggregates (intra-aggregate pores) are of micropore sizes. Other researchers have used a tri-modal pore structure to define bentonite pores (Yong 1999; Pusch and Yong 2003; Salles et al. 2009; Nowamooz and Masrouri 2010). Herein, the pores within bentonite - interlayer, inter/intra-particle and inter/intra-aggregate – are considered to provide a dual (macro and micro) porosity within the unsaturated regime.

GCLs have a more complex porosity pattern over the range of sizes with average pore sizes that are different for the bentonite and the geotextile components of GCLs. In this context, the whole GCL can be considered as having a tri-modal pore structure which, in addition to the interlayer, intra- and inter-aggregate pores of bentonite described above, includes the geotextile pores. In the current study, the geotextile pores are referred to as “macro”, the inter-aggregate pores as “meso”, the intra-aggregate pores and all pore size smaller than intra-aggregate (such as inter-particle, intra-particle and interlayer pores) as “micro” pores (Acikel et al. 2018) . A representative micro-structure of bentonite units and porosities in a bentonite grain, as well as GCL tri-modal pore structure, are shown in Figure 1. It is important to note that the “macro” and “meso” pores terminology used for *GCLs* is not the same as that used to describe the *bentonite* tri-modal pore structure, as the macropores also contain contributions from the geotextile component.

1.2. ***Background – time dependency of equilibrium among tri-modal pores***

The time dependency of measured suction values is essential in terms of GCL water retention testing. Time-dependent suction changes in GCLs affect water retention measurements through: (i) the conditioning time of bentonite (ageing), and (ii) the test equilibration time. The suction value of the specimen changes during the conditioning period due to diffusion-limited migration of water into smaller less accessible pores. While theoretically, it may be possible for a fast and non-destructive test to accurately measure the suction value at any specific time, all measurements regardless of the method chosen, exert a change in the suction value due to water re-distribution during the time required for equilibrium to be established. Yong (1999) observed that measurements of the equilibrium energy status might correspond to the conditions existent within the macropores; therefore, measurements may not represent the true equilibrium of meso- (inter-aggregate) and micropores (inter-aggregate). Later, Gates et al. (2017) stated that the equilibrium condition could be driven in both directions between the pore sizes by the vapour phase. The time-dependency of water redistribution within the pore structure means that a change in suction values may take place until equilibrium is re-established, particularly for bentonites and other clay-enriched materials, even if the change in gravimetric water content is negligible.

The effects of suction changes on the drying path are shown diagrammatically in Figure 2. When the gravimetric water content change is complete, and the system has reached a new constant gravimetric water content, the suction value will continue to change due to bentonite conditioning. The measurement procedure also changes the measured suction value slightly or significantly depending on moisture exchange during the measurement. Also, the magnitude of the suction change depends on when the measurement is made and how much time is required for the measurement. At any specific time during the conditioning, the measured suction value will be the accurate value if the measurement is instantaneous and the measurement itself has not changed the suction. However, the measurement dependency (the suction changes due to measurement time and duration) changes the actual suction value meant to be measured at a specific time during the conditioning period. To improve the accuracy of the suction measurement within a conditioning period, optimum testing times need to be determined to minimize the measurement impact which will make the instant (or relatively faster) measurements inapplicable. Alternatively, the numerical impact of the measurement needs to be estimated and taken into account to calculate the actual suction value which would not incur measurement impact.

2. *Material and Methodology*

2.1. *Material*

Three needle-punched GCLs were used in this study and are referred to as GCL1, GCL2 and GCL3, respectively. GCL1 (Elcoseal X2000) and GCL2 (Elcoseal X1000) are powder bentonite based GCLs. GCL1 had a scrim reinforced carrier geotextile consisting of both woven and nonwoven geotextiles and a nonwoven cover geotextile. GCL2 had a woven carrier geotextile and a nonwoven cover geotextile. Both GCLs were thermally treated. The bentonite component of GCL1 and GCL2 (referred to herein as B1) originated from the same source. GCL3 (Bentomat ST) is a granular bentonite (referred to herein as B2) based GCL. It had a woven carrier geotextile and a nonwoven cover geotextile and was not thermally treated. The major properties of the GCLs used in this study are shown in Table 1.

2.2. *Methodology*

2.2.1. *Equipment*

A WP4C dew point potentiometer (Decagon Devices, Inc., Pullman, WA, USA) was used in the present investigation to measure the GCL total suction (Fig.3). The potentiometer uses chilled mirrors to determine the dewpoint of an enclosed atmosphere brought to vapour-phase equilibrium with a soil specimen and measures total suction. The measurement chamber contains a mirror, a photoelectric cell, a thermocouple attached to the mirror and a fan. The chamber is sealed after the test specimen is placed within a specifically designed sample container which has an outer diameter of 40 mm and a height of 10 mm.

The suction measurement range of WP4C is 0-300 MPa (Decagon Devices Inc 2010). The instrument has an accuracy of ±0.05 MPa for the suction range 0-5 MPa and 1% for the suction range of 5-300 MPa. Moreover, the device has a temperature control feature allowing tests to be conducted in the 15-40 °C range. The WP4C was designed to be compatible with ASTM-D6836 (2007).

When the system in the chamber reaches equilibrium, the water potential of the air in the chamber is assumed to be equal to the water potential of the specimen. The specimen temperature is controlled by an internal thermo-electrical module, whereas the temperature of the mirror is controlled by a thermoelectric cooler. The photoelectric cell observes the point when condensation first appears on the mirror through the change in reflectance of the light when condensation occurs on the mirror. The internal fan circulates air within the specimen chamber to reduce the time to reach vapour equilibrium. Since the dew point and specimen surface temperatures are measured simultaneously, the need for complete thermal equilibrium is eliminated. The experimental program reported in this study was conducted at room temperature at 22±1˚C and 60±5% relative humidity (RH).

2.2.2. *Test Procedures*

Three different test procedures were used to obtain the GCL WRCs with a particular focus on the impact of the GCL structure and the bentonite form (i.e. granular versus powder) on the time dependence of the total suction measurements.

Suction measurements were performed on both the wetting and drying paths. The first and second test procedures differed in the amount of conditioning time allotted to each GCL specimen. The difference between the second and the third test procedures was the suction equilibrium time associated with testing within the measurement chamber of the WP4C device. Table 2 summarises the test procedures used on the wetting and drying paths. The dew point potentiometer was also set at 22°C for the suction measurements. The test procedures are detailed below.

Tests performed along the wetting path

Tests were commenced using as-received GCL specimens (i.e., at a gravimetric water content of 10 to 12%). Before starting the wetting phase, the total suction value of the as-received specimens was measured. The initial step for all test procedures involved the hydration of the GCL specimens that were placed in containers specially designed for the WP4C device. The specimens were hydrated by injecting a pre-determined amount of de-ionised water to increase the gravimetric water contents by ~15-20% incrementally. In the course of the study the behaviour during the conditioning duration is investigated instead of only homogenized condition. Thus, the calculated amount of water injection was preferred for the hydration process rather than vapor equilibration or misting to avoid and control homogenization. Duplicate specimens were used for each GCL type. The first test procedure, termed herein as "1st test", used one of the two specimens, whereas the second and third test procedures (which are referred to, respectively as, 2nd and 3rd tests) were conducted in sequence on the second specimen. The process was as follows for the first specimen of each GCL type used for the 1st test. After each hydration step, the specimen was sealed for 2 hours for conditioning before initiating suction measurements. A conditioning time of 2 hours was chosen to study the suction responses of the specimens that were close to the initial stage of water re-distribution. Once the suction measurements were performed, the specimens were then further hydrated for the next gravimetric water content target, these steps were repeated until the completion of the wetting phase. The suction measurements took between 20 minutes and 2 hours to complete.

For the second specimen of each GCL type, used for the 2nd and 3rd tests (Table 2), the process was as follows: The gravimetric water content (GWC) was increased gradually to a target value, once this value was reached the specimens were kept sealed for 6 days. Six days of conditioning was chosen to study the suction responses of the specimens that are closer to the final suction equilibrium. After the conditioning phase, the specimens were placed in the chilled mirror device to conduct a measurement (2nd test). The specimens were then left undisturbed in the device for a further 1 day. Then three subsequent measurements were made without removing the GCL specimens. The average of the last three test results was taken the representative measurement for the 3rd test procedure. The process described above was repeated for each GWC target until the completion of the wetting phase.

Tests performed along the drying path

A similar approach to the wetting path tests was used for the drying path tests. Again, two specimens were used for each GCL type. The 1st test used the first specimen while the 2nd and 3rd tests shared the second specimen. A common initial step for the three procedures was to hydrate the GCL specimens to an initial gravimetric water content which corresponded to 90% of the saturation values (see Table 1 for saturation values). A hydration of 90% saturation was chosen to represent the maximum hydration before initiating the drying path. This procedure was used to avoid irreversible bentonite loss from the GCL and excessive pull-out of GCL fibres.

The specimens were conditioned for two weeks in sealed containers under 1 kPa confining stress. After two weeks of conditioning, the specimens were then cut to the WP4C sample container size and left in a desiccator with silica gels to dry in drying steps of approximately 15-20% gravimetric water contents. A vacuum pump was connected to the desiccator to expedite the drying process. The thickness of the specimens was monitored during vacuum drying. For the first suction measurements, the specimens were kept in the desiccator until the specimen thickness was reduced such that it could fit into the WP4C measurement chamber.

After the suction measurement corresponding to the highest hydration state was made for the specimen in the 1st test, it was placed in the desiccator containing silica gels. A vacuum pump was run until the gravimetric water content of the specimen dropped by ≈15-20% to correspond to the next target gravimetric water content step. The specimen was sealed and allowed to condition for ≈2 h. The steps used for the 1st test were repeated until the suction value reached 100,000 kPa which corresponded to the gravimetric water contents (10-12%) of virgin GCLs under laboratory storage conditions.

For the 2nd and 3rd test procedures, the specimen was dried as described for the 1st test procedure to the target gravimetric water contents using the vacuum pump procedure. The specimen was then sealed and left to condition for 2 days after each target gravimetric water contents. The suction measurement for the 2nd test was taken and, similar to the wetting phase, the specimen was left within the measurement chamber for further conditioning for 1 day. For the 3rd test, three consecutive suction measurements were taken without disturbing the specimen, and the average value was recorded.

3. *Results and Discussion*

The chilled mirror test gave total suction measurements that were assessed for time effects (i.e., conditioning and testing time) as well as the effects of the GCL characteristics (namely; bentonite form, structure, geotextile types). The fitting equation (eq. 1) developed by Fredlund and Xing (1994) was used to obtain the WRCs corresponding to the three test procedures used for the wetting and drying paths. The selected fitting equation characterises water retention water content as a function of total suction:

$$w(\psi) = w_s\left[1-\frac{ln\left(1+\frac{\psi}{h_r}\right)}{ln\left(1+\frac{10^6}{h_r}\right)}\right]\left[\frac{1}{\left[ln\left[exp(1)+\left(\frac{\psi}{a_f}\right)^{n_f}\right]\right]^{m_f}}\right] \quad [1]$$

where, ψ is suction (kPa); w is gravimetric water content (kg/kg or m^3/m^3), w_s is saturation gravimetric water content; and a_f (kPa), n_f, m_f, and h_r (kPa) are curve fitting parameters.

The experimental results and the WRCs for GCL wetting and drying paths are shown in Figures 3 and 4, respectively. The fitting coefficients of Fredlund and Xing (1994) for both the wetting and drying paths are given in Tables 3 and 4, respectively. The coefficients of determination (R^2) for the best fitting curves ranged between 0.78 and 0.99 on the wetting path and between 0.89 and 0.98 on the drying path. The procedures associated with the 3rd test gave the best fitting results on both wetting and drying paths with min R^2 values of 0.96 for the wetting path and 0.97 for the drying path.

Figure 3 indicates that no significant conditioning time effect occurred between 2 h (1st test) and 6 days (2nd test) on the suction values for the wetting path of powder bentonite based GCLs (i.e., GCL1 and GCL2). However, for the granular bentonite based GCL (GCL3), there was a significant decrease in the suction values corresponding to the longer conditioning time (6 days as in the 2nd test) compared to the shortest conditioning time (2 h as in the 1st test).

Vangpaisal and Bouazza (2004) investigated the effect of the bentonite particle type (i.e., powdered versus granular) on the hydration mechanisms of similar GCLs. While differences in the wetting rates and pathways were observed, in both cases, the bentonite gradually absorbed the water deeper into the core until the water potential fully equilibrated. The observed difference was that in the granular bentonites the suction built up inside granules. This behaviour may have inhibited the overall water distribution until either the granules separated or air was absorbed by the wetting front which tends to slow down the homogenization

The GCL wetting path test results showed that suction values of the granular bentonite based GCL (GCL3) were more sensitive to the conditioning time. The decrease in suction values for GCL3 related to conditioning time suggests that the water is taken up by the granular bentonite based GCL re-distributed faster throughout the entire thickness of the GCL. Reference to faster water intake throughout the GCL thickness should not be taken to apply to the overall distribution or homogenization of the water potentials; the distribution in this first stage happens only in the macro-pores. Homogenization of water distribution throughout the micro-pore scale will be slower in GCL3 due to its granular bentonite structure. Thus, the sensitivity observed can be interpreted as being due to the presence of macro (inter-aggregate) pores present within the granular bentonite. However, while a quasi-equilibrium state existed and enabled an accurate measurement of total suction, homogenisation was incomplete. Complete homogenisation appears only to happen when water completely re-distributes throughout the macro-micro pores.

While the conditioning and testing time responses were similar for both the wetting and drying paths of GCL1 and GCL3; GCL2 showed different time dependency for the wetting and drying paths. GCL1 had a scrim-reinforced woven, and nonwoven carrier geotextile which provided greater bonding between the carrier and cover geotextile layers as a result of the needle punched fibres. While GCL1 had the same bentonite and cover geotextile as GCL2, its structure maintained a greater resistance against swelling in comparison to GCL2. Also, GCL2 had a greater bentonite mass per unit area than GCL1, and not having scrim-reinforced nonwoven carrier geotextile meant that it had a greater swell capacity. Table 5 shows free swell hydration test results for GCL1 and GCL2. GCL2 had greater volume change (52%) at reference GWC compared to GCL1, which had only 36% of volume changes at the reference GWC. In addition the reference GWCs of GCL1 and GCL2 were highly comparable (Table 1).

Gatabin et al. (2016) investigated the suction responses of confined and unconfined bentonites on the wetting path and reported that unconfined bentonite reached suction equilibrium faster than confined bentonite. Similarly, the level of confinement is higher in GCL1 due to the scrim-reinforcement. GCL2 has less confinement due to its geotextile configuration and tends to have higher swelling pressure due to higher bentonite mass per unit area. Thus, higher sensitivity to testing time can be expected from GCL1 in comparison to GCL2 within the selected testing and conditioning time

Similar to the response for the wetting path, the conditioning time dependency for the drying path of the GCL1 was not significant. However, testing time dependency was observed. While no significant time-dependent behaviour was observed during the wetting path of GCL2, dependency was observed for both the conditioning and testing time on the drying path. More volume change is expected in GCL2 in comparison to GCL1 due to higher level of swelling (Table 5) during hydration prior to the drying path. The bonding of the GCL, as well as the bentonite mass per unit area, affected the total suction time dependencies (i.e., both conditioning and testing) of the GCLs. The higher freedom in swelling probably resulted in GCL2 being able to physically respond to drying (i.e., wetting-drying hysteresis) and therefore provided greater time-dependent behaviour during drying because of the greater bulk swelling in the system after initial hydration.

Both wetting and drying path test results indicated that all GCLs showed similar responses to the testing times (between 2nd and 3rd tests) (Figures 3 and 4). Before the cross-over point of the water retention curves, the measured total suction results decreased in the case of longer measurement times. The trend was opposite for total suction values higher than the cross-over point. The observed behaviour is believed to be due to the measurement and re-conditioning times and is further discussed in the following section.

4. Conceptual Model

Bentonite is composed of aggregates, which are made up of smaller particles and ultimately quasi-crystals of smectite. Smectite quasi-crystals are composed of crystallite layers (Bergaya and Lagaly 2013) as shown in Figure 1. GCLs, on the other hand, consist of bentonite confined within geotextiles, which imparts an additional type of pore structure. The triple pore structure model of GCLs consists of the micro-, meso- and macro-pores associated with the bentonite and the geotextiles. At low gravimetric water contents, the first stage of swelling (crystalline), the swelling behaviour of GCL is expected to be mostly associated with micro-pores associated with the bentonite component, while during the osmotic swelling (a.k.a. bulk swelling) the GCL meso and macro-pores gradually become involved. Due to the different locations of the water (i.e. interlayer, inter/intra-particle and inter/intra-aggregate), unsaturated bentonite can be defined as having a dual (macro and micro) pore structure.

As a result of hydration at different pore levels, the swelling of bentonite occurs in two main stages (Norrish 1954; Saiyouri et al. 2000; Saiyouri et al. 2004; Laird 2006): crystalline and osmotic (or bulk) swelling. Crystalline swelling can be defined as the insertion of organised layers of water between crystallites making up the quasi-crystals (within the interlayer space) at lower gravimetric water contents. The second stage, osmotic swelling is described as the division of the particles (i.e., reducing the number of quasi-crystals making up a particle) at higher gravimetric water contents above saturation.

Saiyouri et al. (2000) reported the existence of four constant interlayer distances in montmorillonite with three transition steps between these distances as well as two significant transition suctions at ≈7,000 kPa and ≈60 kPa. After progressing through the first stage of swelling (crystalline swelling), the separation between crystallite layers became much greater than ~2.2 nm (Saiyouri et al. 2000; Saiyouri et al. 2004). Saiyouri et al. (2004) further discussed the probability of water distribution in the montmorillonite fraction of a sodium bentonite (MX80) at 0- to 4-layer hydration states (corresponding to d-values of 1.0 to 2.2 nm, with a mean interlayer distance of 1.9 nm) as a function of suction. The distribution of the d-values indicated a hydration state predominantly between 1- to 3-layers at suction values between 6,900 kPa and 33,000 kPa. This behaviour changed to predominantly 2- to 4-layers (1.6-2.2 nm) within a suction range of 2,800 kPa and 6,900 kPa.

Saiyouri et al. (2004) also quantified the average number of smectite quasi-crystals making up particles as a function of hydration. At the lowest gravimetric water contents, an average of 350 crystallites were observed, but the number of crystallites decreased dramatically over the suction range 2,800 kPa to 6,900 kPa, indicating a breakdown of particles into swollen quasi-crystals. The re-arrangement of the fabric responded directly to homogenisation and also caused changes in the water potential. It would appear that these changes likely accounted for the observations associated with the cross-over points.

4.1. Cross-over points

The cross-over points shown in Figure 4 correspond to suction values ~2,700 kPa, 2,800 kPa and 5,000 kPa (for GCL1, 2 and 3, respectively). These suction values are generally within the suction range (2,800-6,900 kPa) reported by Saiyouri et al. (2000, 2004) as corresponding to suctions within the crystalline swelling stages of smectite. The cross-over points highlighted in Figure 4 probably show the behavioural change in bentonite due to prevailing water locations. The cross-over points in Figure 3 are not as clearly observed and perhaps represent multiple cross-over points along the wetting path. The multiple cross-over points (Figure 3) are likely the result of the impact of the geotextile bonding structure in the GCL. Some of these cross-over points might also be as a result of the accuracy of the WRC fitting. In summary, the cross-over points in Figure 3 appear to correspond to the suction values at breakdown of the smectite particles into the fundamental crystallites. The impact of the conditioning and testing time is further discussed in this subsection.

A conceptual model based on the chilled mirror test results is presented to explain the time dependency and the different time-dependent behaviours observed for GCL wetting and drying paths after the cross-over points. A conceptual model is also proposed to explain the testing time dependency and the impact of the test procedure on the suction measurement accuracy of bentonite based materials such as GCLs.

The homogenisation time in a granular medium can be described as the conditioning time needed for potential equilibrium after the water content appears to stabilise. The total suction through the granular medium is expected to be macroscopically constant when the system is homogenised under constant temperature and loading conditions. After the gravimetric water content has been stabilised at a new level, the suction value is expected to decrease along the wetting path (or to increase along the drying path) (i.e., it is path independent). The suction values should asymptotically reach an equilibrium (homogenization) suction value. However, in accordance with the measured chilled mirror test result (Figures 3 and 4), some results follow a suction increase trend along the drying path and a suction decrease trend along a wetting path. Also, the results reverse after the observed cross-over points.

4.2. Conditioning Time Dependency

Figures 5 and 6 introduce the proposed conceptual model for conditioning time dependency. The model consists of three axes, namely: gravimetric water content; suction; and time. The left sides of both figures are typical water retention graphs with axes for gravimetric water content and suction (w-ψ). Two WRCs; namely, "t_0" and "t_{Eq}"; are defined on the left sides of the figures. Curve "t_0" shows the WRC which consists of the suction values just after the final gravimetric water content of the GCL is reached. Curve "t_{Eq}" displays the suction values when the water homogenization is completed.

Figure 5 shows a conceptual model for the time dependency of a GCL on the wetting path. The cross-over point between the water retention curves "t_0" and "t_{Eq}" has a corresponding gravimetric water content and suction value as shown on the left side of the figure. The right side of the graph models the time dependency of the suction values during conditioning and present hypothetical suction values against time (ψ-t). Similar to the left side, there are two curves on the right side of the figure; namely, "w_L" and "w_H"; which represent two different gravimetric water content zones for the two stages of swelling as described earlier. The curve "w_L" is a final gravimetric water content (i.e., after water content change is completed), which corresponds to gravimetric water contents lower than the cross-over point, where the interlayer pores could potentially reach a hydration limit of 4-layers of water. Curve "w_H" depicts a gravimetric water content that is greater than w_{CP} on curves showing "t_0" and "t_{Eq}", and this zone corresponds to water entering larger meso- and macro-pores in the GCL structure.

While the impact of the geotextile fibres is not explicitly considered in this conceptual model, two types of time-dependent behaviour related to two gravimetric water content ranges (i.e., curves w_L and w_H, Figures 5 and 6) of the GCL are presented. It is worth nothing that the geotextile fibres are expected to contribute to suction only within zone w_H since suction values in zone w_L mostly depends on the micro-pore scale (i.e., that of the bentonite component). The suction value "ψ_i" is the initial suction in equilibrium before the hydration step. The suction value "ψ_0" is the suction value that occurs when the wetting step has been completed at the gravimetric water content w_L or w_H. The suction value "ψ_{Eq}" refers to stabilised conditions when the suction has stabilised (i.e., water homogenisation after wetting) at the final gravimetric water content (either w_L or w_H). The time when hydration starts, t_0, defines when the final gravimetric water content (i.e., either w_L or w_H) has been reached. Time t_{Eq} defines when homogenisation (suction equilibrium) has finished, and the final gravimetric water contents are established within either zone w_L or w_H. After wetting in the low gravimetric water content zone (w_L), added water is expected to distribute predominantly within interlayers. Water distribution in the higher gravimetric water content zone (w_H) will occur within the inter-particle or inter-aggregate pores once crystallites interlayers attain a 4-layer hydration state. These two different mechanisms of water distribution are the likely cause of the various conditioning time behaviours observed in the gravimetric water content zones. While the suction value undergoes a continuous decrease and reaches a quasi-equilibrium state in zone w_L, it then increases and eventually reaches the true equilibrium asymptotically. In the high gravimetric water content zone w_H, the decreasing trend of suction is continuous but the rate decreases on approach to the final or true equilibrium suction.

A conceptual model for GCL drying path time dependency is shown in Figure 6. Similar to the wetting path, two different time-dependent behaviours are proposed in the conceptual model. Opposite to the wetting path, the water re-distribution occurs initially within pores having the lowest potential (from the macro pores), which leads to a shrinkage in the macro scale. In the higher gravimetric water content range of the drying path, initially, the water potential increases with water loss but reaches an unstable quasi-equilibrium. Volume change due to the collapse of macro-pores is expected to lead to further water redistribution and ultimately a further decrease in the water potential. After this decrease, the water potential will eventually reach the true equilibrium. Within the lower gravimetric water content zone (w_L) drying happens within the same pore size level (interlayer) and results in increased suction values, the rate of which slows as true equilibrium is approached.

4.3. Testing Time and Duration Dependencies

The conceptual models for the wetting and drying paths were adapted to represent the chilled mirror test wetting path (Figure 7) and drying path (Figure 8) results to model the testing time and duration dependencies. In Figure 7 and 8, the suction values ψ_1, ψ_2 and ψ_3 are the measurement performed at 1st, 2nd, and 3rd tests, respectively. The time-dependent behaviours shown in Figure 7 and 8 (for wetting and drying paths, respectively) represents the case if there is no measurement effect, and the dashed curves show the measurement impact. T_1, T_2 and T_3 are the measurement time of 1st, 2nd, and 3rd tests, respectively.

Some drying was observed for all specimens regardless of whether tested on the wetting or drying paths. While the amount of water loss during the test was considered insignificant in terms of total gravimetric water content change, water loss did affect the measurement accuracy adversely, because even a small amount of water loss requires time for re-distribution and suction equilibrium. The drying impacts of the longer tests (i.e., between T_2 and T_3) are also modelled as parallel to the different drying path behaviours for the two gravimetric water content zones (w_L and w_H) which are shown in Figure 6.

Based on the chilled mirror test results, the conceptual model shows that measured suction results may significantly depend on the amount of time between changing the gravimetric water content of the specimen and when the suction measurement is started. The results are further impacted by the duration of the measurement. Moreover, the change in the measured value is also dependent on the GCL gravimetric water content.

5. *Conclusions*

Three different chilled mirror test procedures were used to investigate the time-dependent unsaturated behaviour of three different needle-punched GCLs on the wetting and drying paths. The procedures were chosen to investigate the time effects due to water redistribution during conditioning after wetting or drying steps as well as on total suction measurement to obtain WRCs.

The tests showed that both conditioning and suction equilibrium (testing) times impacted total suction values. Changes in time-dependent behaviour from increasing trends to decreasing trends in the suction values over time at certain points on WRCs (e.g., before and after cross-over points) was one of the critical observations of the study. A conceptual model was proposed to explain the time-dependent behaviour of the WRCs of GCLs. According to the proposed model, cross-over points correspond to bentonite particle separation initiated when crystalline swelling reaches the 4-layer hydrate state of smectite (i.e., all micro-pores (interlayer spaces) are filled with water). The conceptual model also suggests that suction values are highly dependent on when the measurement is performed and how long it takes to perform the measurement. In other words, the accuracy of the measurement will be different depending on when and how long the measurement is carried out. Bentonite can have different suction values due to the conditioning time and also due to the testing (measurement) time and duration.

Deeper understanding of conditioning and testing time dependencies on total suction measurements by chilled-mirror hygrometer technique leads to improved understanding of GCL hydration and dehydration behaviour in field applications which are critically important for their barrier performance. As a future study, the numerical implementation of suggested conceptual model will result in more accuracy in predicting of GCL behavior in practice. The implementation needs to be in a time dependent suction range for given gravimetric water contents instead of considering only the suction values at the homogenized condition since in the field application GCLs will not be always in homogenized condition. During the implementation the estimated numerical impact of the measurement time and duration needs to be taken into account to estimate the most accurate suction value during any time in conditioning period.

The objective of the study was to observe the time dependency of the unsaturated behaviour and explaining it in a conceptual model. A suggestion for an optimum procedure is avoided since there is not enough numerical data in the current study to determine the optimum procedure. However, based on the current results among three test procedures, 3rd test procedure can be recommended as the results obtained with this method were the closest to the homogenized results with minimal testing time dependency.

Other key findings of the study are:

- The comparison of GCL1 and GCL2 (which has the same powder bentonite but different geotextile configurations) showed that the degree of confinement due to geotextile fibres and bentonite particle size resulted in different time-dependent effects (conditioning and suction equilibrium) on the measured total suction values. Higher confinement owing to scrim-reinforcement and thermal treatment significantly minimised the conditioning time effects.
- Granular bentonite based GCL with woven carrier geotextile (GCL3) had the highest conditioning time effects for both wetting and drying paths of the WRC.

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Figure 8 Conceptual model for time dependent behaviour of GCL drying path adapted to chilled mirror test results

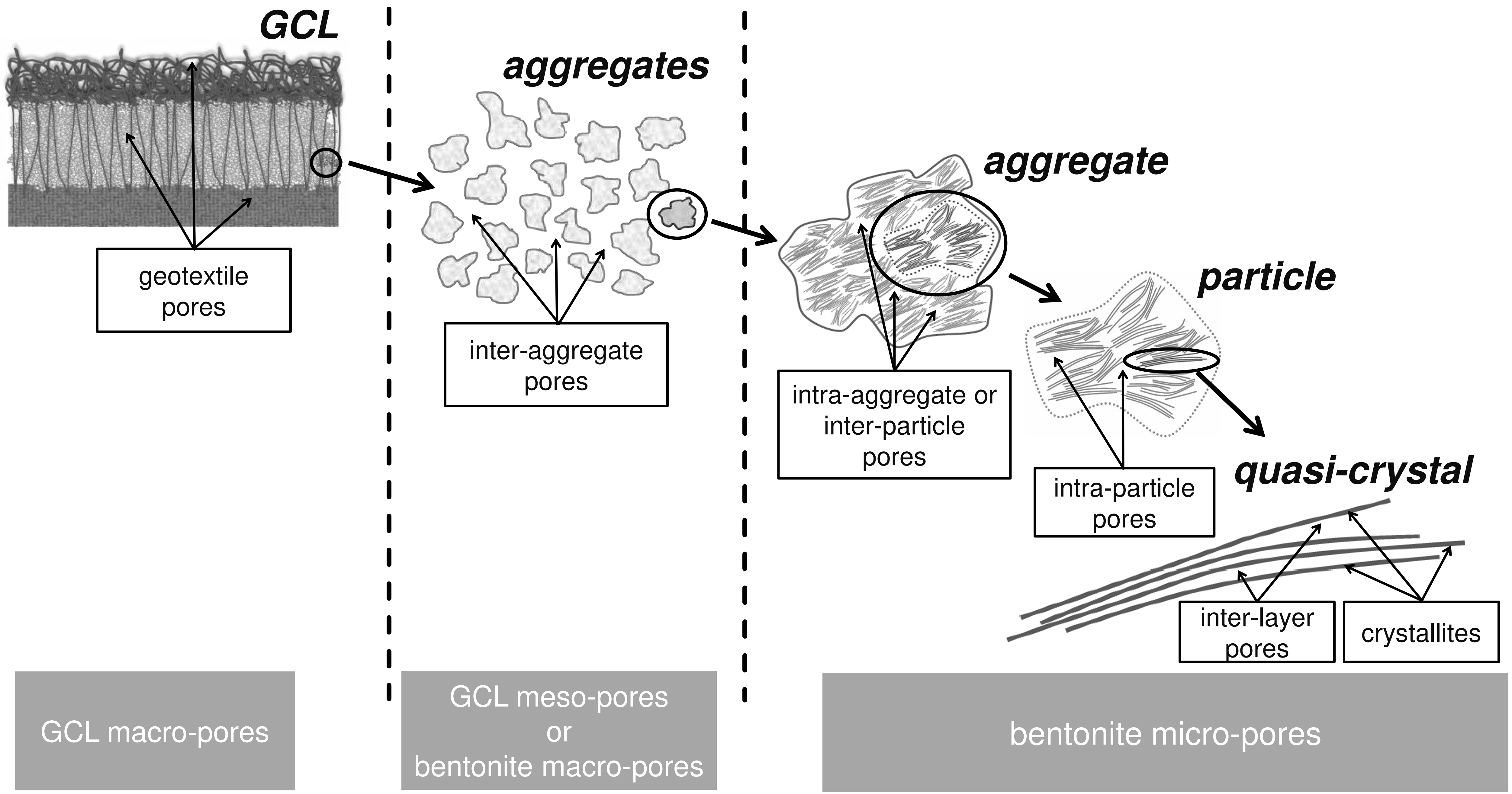

Figure 1 Representative micro- and macro-structural units and pores of bentonite and GCL tri-modal structure with macro, meso and micro pores (modified from Acikel et al 2018)

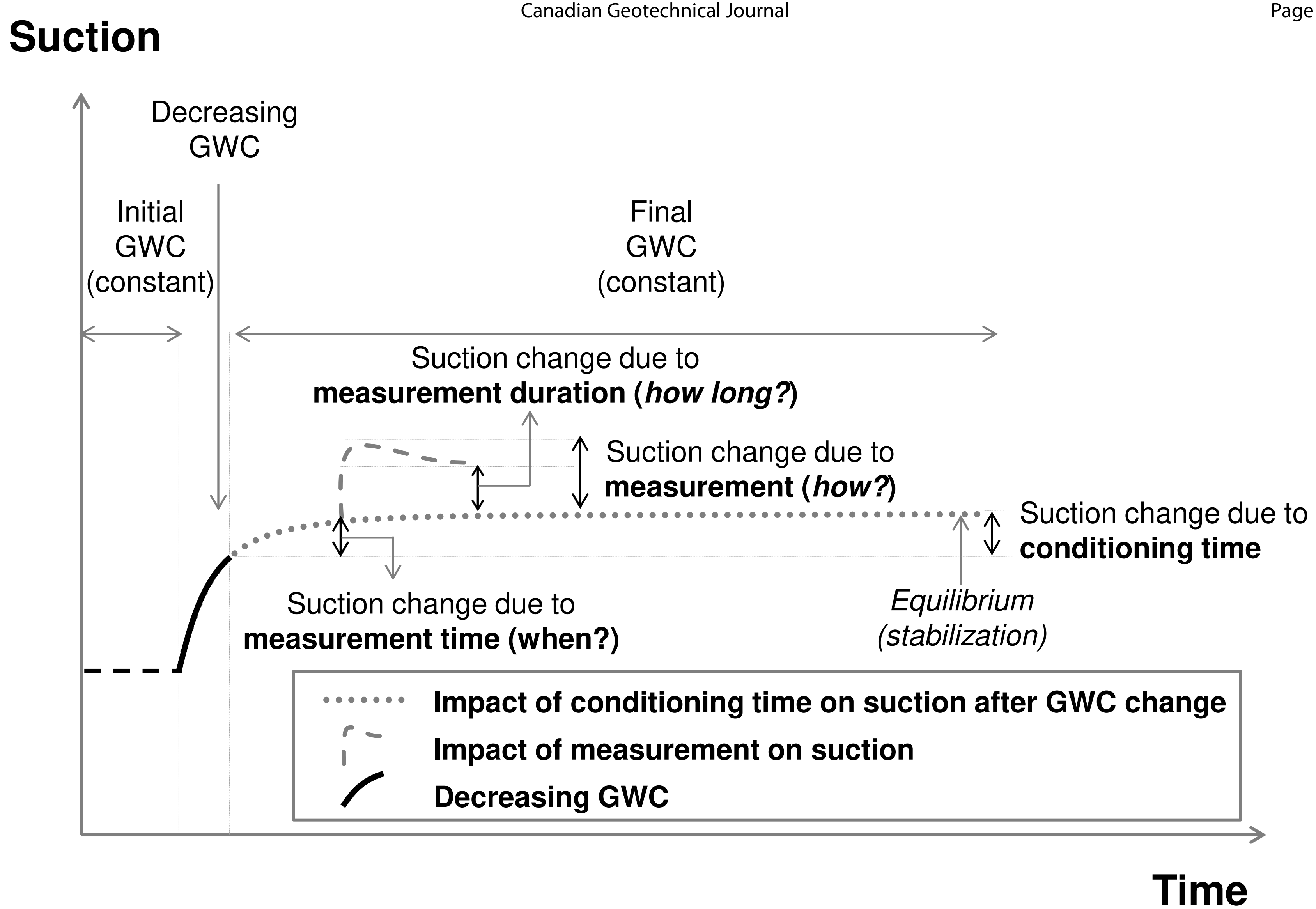

Figure 2 A hypothetical representations of conditioning and measurement time as well as measurement duration dependencies on GCL suction measurement on the drying path

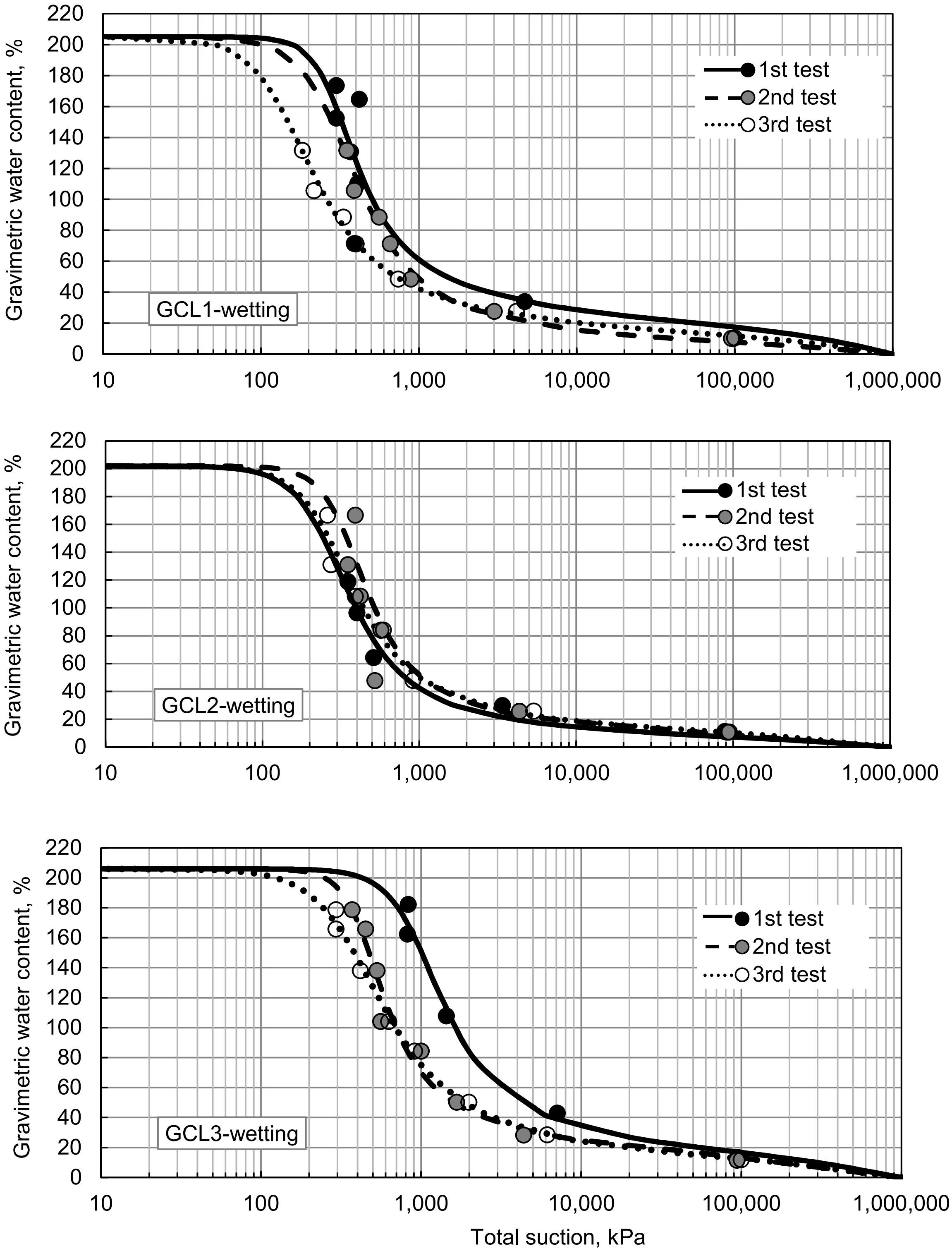

Figure 3 Experimental results from chilled mirror tests and the water retention curves (WRCs) as calculated using Fredlund and Xing (1994) model for the wetting path.

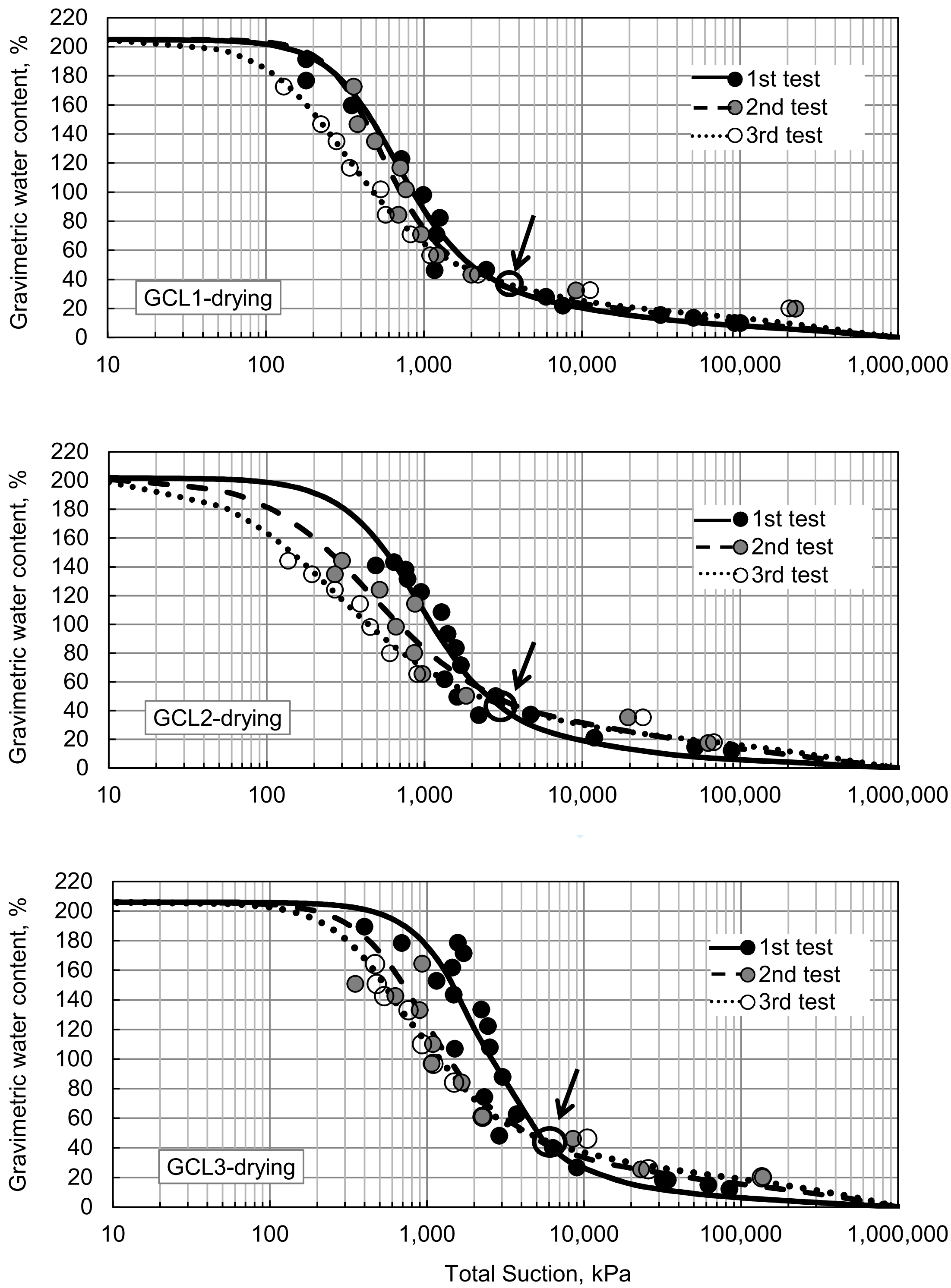

Figure 4 Experimental results from chilled mirror tests and the water retention curves (WRCs) as calculated using Fredlund and Xing (1994) model for the drying path. The cross-over points of the curves are indicated with circles and arrows.

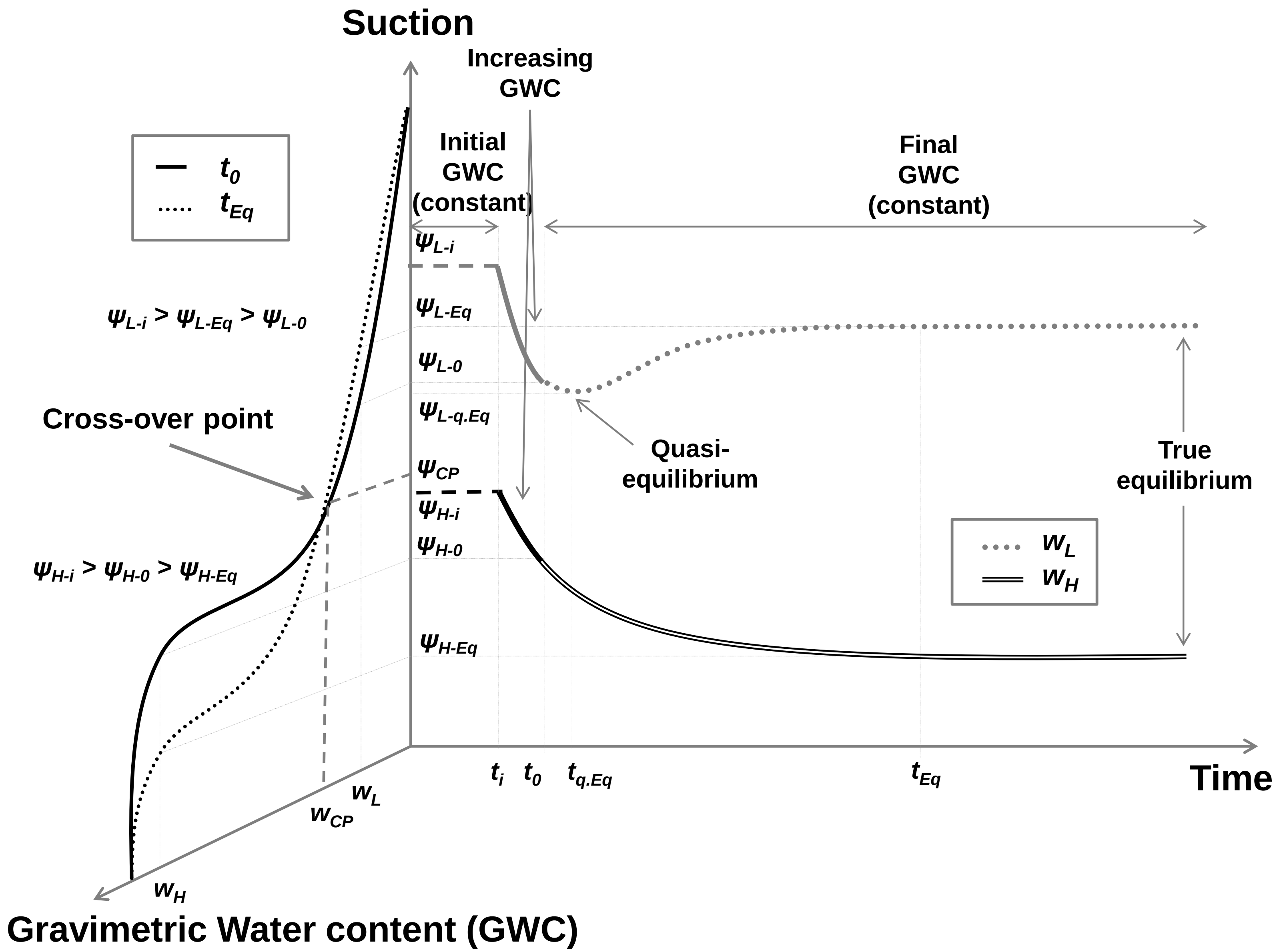

Figure 5 Conceptual model for time dependent behaviour of GCL on wetting path. On the left are the water retention curves for t_0 (when gravimetric water content stabilised) and t_{Eq} (when suction stabilised), and on the right are the time-dependent suction changes related to conditioning time for w_L (low gravimetric water content) and w_H (high gravimetric water content) zones.

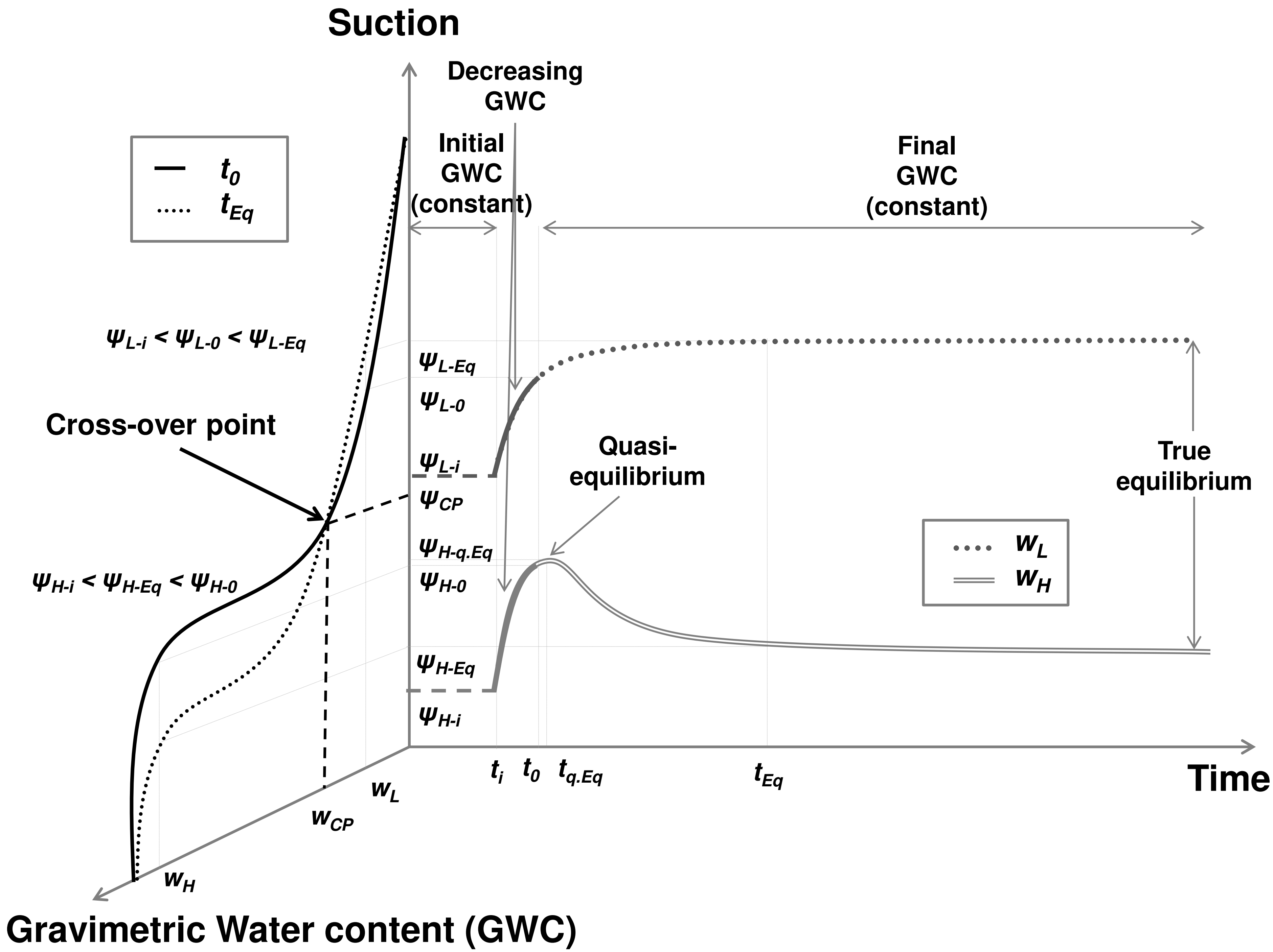

Figure 6 Conceptual model for time dependent behaviour of GCL on drying path. On the left are the water retention curves for t_0 (when gravimetric water content stabilised) and t_{Eq} (when suction stabilised), and on the right are the time-dependent suction changes related to conditioning time for w_L (low gravimetric water content) and w_H (high gravimetric water content) zones.

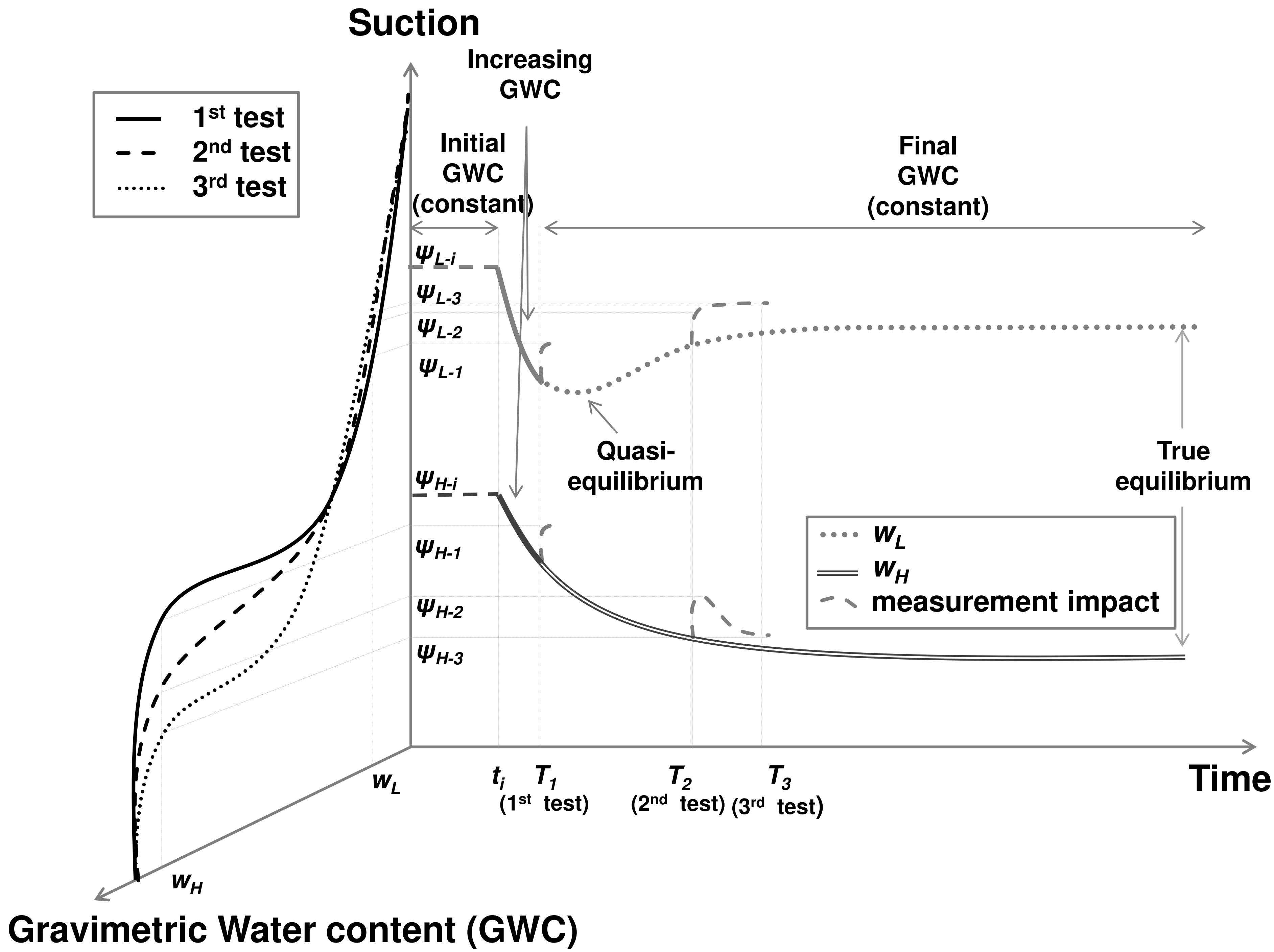

Figure 7 Conceptual model for time dependent behaviour of GCL on wetting path adapted to chilled mirror test results

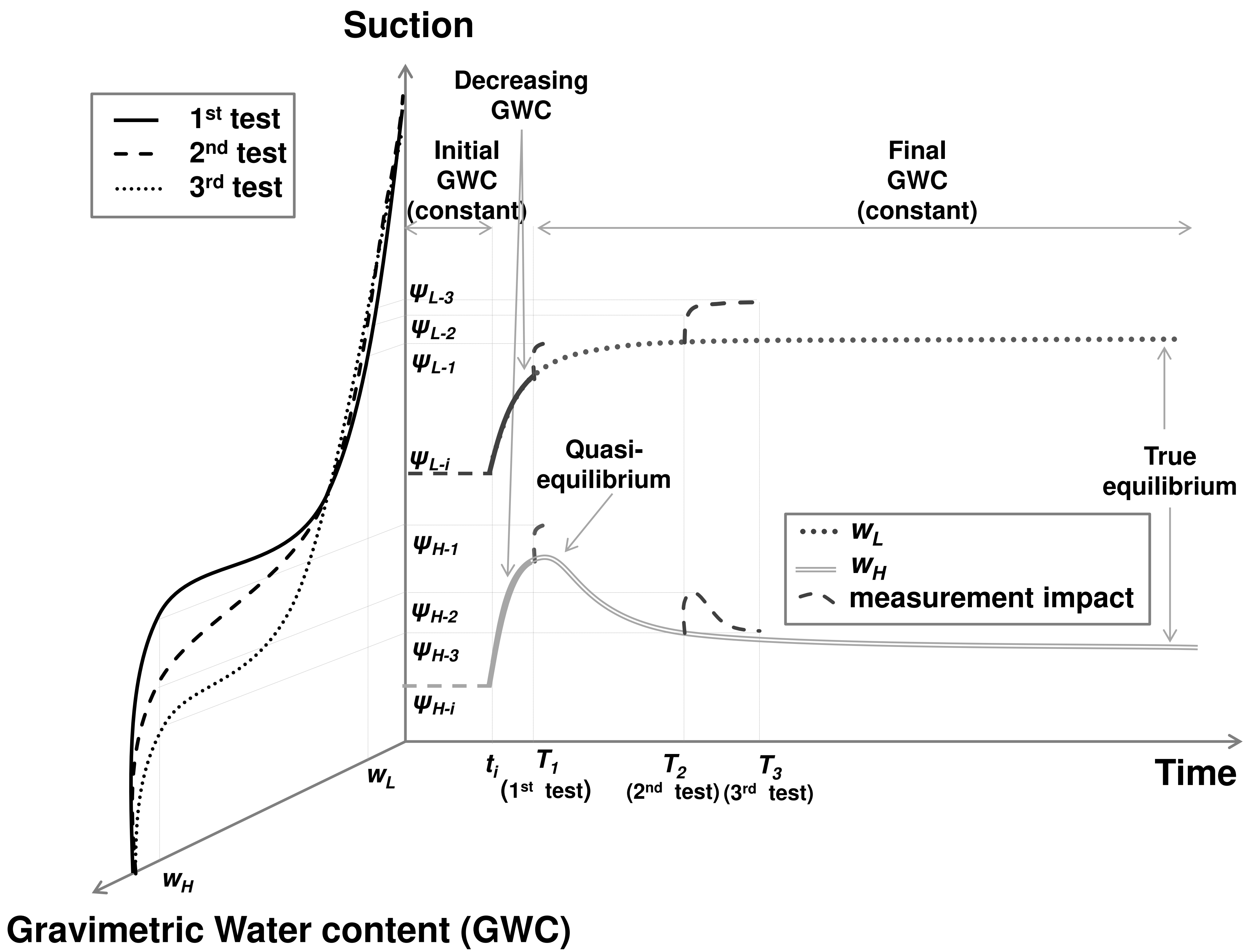

Figure 8 Conceptual model for time dependent behaviour of GCL on drying path adapted to chilled mirror test results

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Table 1 GCLs characteristic properties

			GCL1	GCL2	GCL3
Mass per unit area (g/m^2)					
GCL		Measured	4885	5266	4698
		MARV*	4350	4380	4000
Bentonite		Calculated	4159	4745	4273
		MARV*	3700	4000	3600
Carrier geotextile		Measured	438	135	126
Cover geotextile		Measured	288	386	299
Saturation (Reference) gravimetric water contents (GWC_{sat}) under 1 kPa vertical static loading			205%	202%	206%
Bentonite	Particle type		powder (B1)	powder (B1)	granular (B2)
	Smectite (XRD test results) †		80%	79%	82%
	Initial (off-roll) gravimetric water content		10%	11%	12%
	Liquid limit (ASTM-D4318, 2000)		755%	772%	370%
	Plastic limit (ASTM-D4318, 2000)		46%	46%	36%
	Swell Index‡ (ml/2g) (ASTM-D5890, 2011)		22	22	22
	Hydraulic conductivity § (m/s) (ASTM-D5887, 2009)		$3x10^{-11}$	$3x10^{-11}$	$5x10^{-11}$
Structure	Configuration (Carrier / Cover)		SRW / NW	W / NW	W / NW
	Bonding		NP	NP	NP
	Peel Strength (N/m) (ASTM -D6496, 2009) ǀ		1855	750	610
	Thermally treated		yes	yes	no

**MARV: minimum average roll value (from producer), SRW: scrim-reinforced woven, W: woven, NW: nonwoven, NP: needle-punched*
† *XRD tests conducted at CSIRO Land and Water, Mineralogical Services, Adelaide laboratory*
§ *Max values as provided by the manufacturers*
ǀ *Obtained from manufacturers*

Table 2 Chilled-mirror test procedures used for wetting and drying paths of GCLs

	Wetting Path			Drying Path		
	1st test	2nd test	3rd test	1st test	2nd test	3rd test
Conditioning time	2 h	6 days	7 days	2 h	2 days	2 days
Suction Equilibrium/Testing Time	20 min -2 h*	20 min*	1 day	20 min -2 h*	20 min*	1 day

** Testing times in 1st and 2nd tests in both wetting and drying paths are not selected values, the listed times are the average point measurement times of the test device obtained during the tests*

Table 3 Calculated gravimetric retention curves(WRC) using the Fredlund and Xing (1994) model fitting parameters for the wetting path chilled mirror tests

GCL	Test	a_f	n_f	m_f	h_r	R^2	Residual GWC
GCL1	**1st test**	276.3	4.091	0.726	943,600	0.78	27%
	2nd test	278.8	2.745	1.120	845,300	0.98	29%
	3rd test	134.4	2.632	0.945	897,500	0.98	30%
GCL2	**1st test**	246.6	2.878	1.110	836,900	0.96	28%
	2nd test	328.9	3.667	0.959	871,000	0.90	29%
	3rd test	255.2	2.867	1.002	888,600	0.96	28%
GCL3	**1st test**	892.1	3.219	0.870	943,600	0.98	27%
	2nd test	418.6	4.348	0.7988	924,900	0.97	30%
	3rd test	356.0	2.371	1.024	915,700	0.99	28%

a_f, n_f, m_f, h_r : fitting parameters for Fredlund and Xing (1994) WRC model

20 Table 4 Calculated gravimetric retention curves (WRC) using the Fredlund and Xing
21 (1994) model fitting parameters for the drying path chilled mirror tests

GCL	Test	a_f	n_f	m_f	h_r	R^2	Residual GWC
GCL1	1st test	496.2	2.084	1.266	862,300	0.97	29%
	2nd test	395.1	2.699	1.009	906,600	0.94	28%
	3rd test	209.7	1.698	1.103	915,700	0.98	27%
GCL2	1st test	732.9	1.793	1.540	804,000	0.91	29%
	2nd test	283.5	1.301	1.224	924,900	0.90	30%
	3rd test	156.5	1.188	1.179	934,200	0.97	28%
GCL3	1st test	1745.0	2.146	1.542	828,500	0.89	30%
	2nd test	660.8	2.088	1.039	934,200	0.89	29%
	3rd test	468.6	1.964	0.952	953,000	0.98	26%

a_f, n_f, m_f, h_r : fitting parameters of Fredlund and Xing (1994) WRC model

Table 5 Swelling of GCL1 and GCL2 under free water hydration (only 1 kPa contact pressure was applied) condition when they were hydrated to saturation (reference) gravimetric water contents (GWC_{sat}) (from Acikel 2016)

	GCL1	GCL2
Bentonite mass per unit area (g/m^2)	4159	4745
Initial average thickness of the specimens (mm)	8.9	6.4
Final average thickness of the specimens (mm)	11.1	8.8
Final vertical strain	25%	37%
Final volume change	36%	52%