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ORIGINAL PAPER

Shrinkage Limit Determination of Soils Using Hydrophobic Compounds

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Abstract Shrinkage limit is one of the important parameters among the Atterberg limits used to classify fine-grained soils and to express its volume stability. Conventional method to determine volume of wet and dry shrinkage pats to evaluate shrinkage limit involves the usage of mercury, which is highly toxic and hazardous to health. Recently various methods have been proposed by different researchers which do have certain shortcomings. The present study proposes a simple and alternate method to determine the volume of dry shrinkage pat with the use of hydrophobic compound and water submersion method, which is required to calculate the shrinkage limit of soils, thereby overcoming the various shortcomings of the alternate methods proposed by different researchers in the recent past. Three hydrophobic compounds, namely Vaseline, hydrophobic liquid, and hydrophobic glue were used to coat the dry shrinkage pats with a thin layer of these compounds and make them hydrophobic, and hence, determine their volume with water submersion. The results of shrinkage limits of soils used in this study obtained using all these three hydrophobic compounds are within the acceptable range as suggested by ASTM standard D427-04 (Test method for shrinkage factors of soils by the mercury method, ASTM International, West Conshohocken, 2004. https://doi.org/10.1520/d0427-04) and ASTM standard D4943-02 (Standard test method for shrinkage factors of soils by the wax method, ASTM International, West Conshohocken, 2008. https://doi.org/10.1520/d4943-08) for any two results obtained from two different tests of the same type, for multi-laboratory determinations. The proposed alternate method of determining the shrinkage limit is not only simple but also reliable. It can be verified by various researchers before it can find universal acceptance as a standard and alternate method of determining the shrinkage limit of soils.

Keywords Clays · Shrinkage limit · Mercury · Hazardous · Hydrophobic compound

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List of Symbols

w_L	Liquid limit
w_p	Plastic limit
w_s	Shrinkage limit
I_p	Plasticity index
M_{sha}	Mass of dry soil pat coated with hydrophobic compound in air
V_{sh}	Volume of dry soil pat coated with hydrophobic compound

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V_h	Volume of hydrophobic compound
M_h	Mass of hydrophobic compound
V_d	Volume of dry soil pat
$V_{d\ (Mer)}$	Volume of dry soil pat by mercury displacement method
$V_{d\ (V)}$	Volume of dry soil pat by Vaseline method
$V_{d\ (Hyp\ L)}$	Volume of dry soil pat by hydrophobic liquid method
$V_{d\ (Hyp\ G)}$	Volume of dry soil pat by hydrophobic glue method
$w_{s\ (Mer)}$	Shrinkage limit by conventional mercury method
$w_{s\ (V)}$	Shrinkage limit by Vaseline method
$w_{s\ (Hyp\ L)}$	Shrinkage limit by hydrophobic liquid method
$w_{s\ (Hyp\ G)}$	Shrinkage limit by hydrophobic glue method

1 Introduction

Shrinkage limit as one of the Atterberg limits of fine-grained soils is the lower limit of water content beyond which volume change ceases. It is an important index parameter used to represent the volume stability of soil mass in the field and also used to classify the cohesive soil based on swell potential (Holtz and Gibbs 1956; IS 1498-1970 1987). To determine the limit of shrinkage, methods were developed from time to time by various researchers, which later got standardized. Among them, a few of the methods became popular for some time, which are the linear shrinkage test (BS:1377-2 1990; IS: 2720 Part 20 1992), volumetric shrinkage, and shrinkage limit test (ASTM D427-04 2004; BS:1377-2 1990; IS: 2720 Part 6 1972- Reaffirmed in 1995). The above-listed methods have their relative merits and shortcomings. Linear shrinkage test measures the percentage decrease in length of a soil sample when it dries, starting from a moisture content equivalent to the liquid limit, and is used to assess the potential for cracking and instability in clayey soils rather than the true shrinkage limit. Volumetric shrinkage, on the other hand, measures the change in soil volume as it dries, providing insights into its potential for volume changes and cracking. This parameter gives only the percentage volume change from the initial condition and does not give the limiting water content at which the soil ceases to shrink. In contrast, the shrinkage limit test is the only test that gives the limiting water content when the soil ceases to shrink. Hence, it became the standard method and is routinely used to determine the shrinkage limit of soils. The laboratory test procedure for determining shrinkage limit has been standardized and outlined in various international codes (ASTM D427-04 2007; BS: 1377-2 1990; IS: 2720-2021). Though the method is standardized and routinely used in laboratories the world over, it has a serious concern as discussed here. To determine the shrinkage limit, it is essential to determine the volume of soil pat both in the wet and dry state. For accurate determination of the volume of dried shrinkage pats shrunk to a shape that is not to a specific geometry, the mercury displacement method has been used universally worldwide. With time, realizing the hazardous nature of mercury and having serious effects on human health, the use of mercury in the determination of shrinkage limit was banned in many international laboratories. Further, Cerato and Lutenegger (2006) stated that: “Mercury having higher specific gravity, one extra or missing drop of mercury in the displacement calculation can change the shrinkage limit results”. To avoid the use of mercury in the determination of shrinkage limit, ASTM c-02 (2008) has proposed the wax method. In continuation of this initiation, researchers tried to develop alternate methods to determine the volume of shrinkage pats (Prakash et al. 2009, 2011; Prakash and Sridharan 2012; Kayabali 2013; Hobbs et al.2014, 2019). However, there are shortcomings in each of the methods proposed by the researchers as presented in the following paragraphs.

The wax method, introduced by Prakash et al. (2009), is an alternative to the mercury displacement method (ASTM D 4943-02, 2008). This method involves coating a dry soil sample with wax and measuring the volume of the dry soil using water displacement. Prakash et al. (2009) have highlighted several shortcomings of the wax method. Notably, it can be challenging to collect all of the displaced water from the pan due to the adhesive properties of water, which is used to determine the volume. Additionally, this method requires heating the wax, which poses safety hazards, such as burns from hot wax or wax-melting equipment. There is also a risk of overheated wax catching fire. Moreover, the water displacement method employed to measure the dry volume of the wax-coated sample is susceptible to errors. Soltani et al. (2024) through statistical analysis explored the relationship between shrinkage limits from the wax method and the mercury method. They observed that the mercury method and wax method do not produce identical shrinkage limit values for a given fine-grained soil under similar testing conditions. To overcome this difficulty, they developed a conversion relationship between the SL obtained by the two methods. The approach undertaken has notable limitations. The relationship is derived from specific empirical data, which may not be universally applied to all soil types, particularly those absent from the original dataset. Soils with unique mineralogy, structure, or composition may also not align with the established relationship. Additionally, because of the limitations of the wax method as discussed above, the efforts done by Soltani et al. (2024) may not be much beneficial.

Prakash et al. (2011) proposed an alternative technique for determining the volume of dry soil pats to assess shrinkage limit. This method replaces both the mercury displacement and wax methods by calculating the volume of the dry soil sample through the calibrated density of sand and the volume of sand it displaces. This approach is straightforward and environmentally friendly, as it uses non-hazardous sand. However, the careful selection of sand type is crucial due to variations in grain size compositions. Prakash et al. (2011) indicated that well-graded sands are unsuitable for this method because they tend to sort grain sizes during the use. Instead, only uniformly graded sands are appropriate for accurate results. Additionally, coarser varieties of uniformly graded sands are less effective, as larger particles and void spaces can introduce potential errors. The angularity of the sand grains may also impact the results.

To further improve the method and address the challenges of obtaining uniformly graded sand, Prakash and Sridharan (2012) proposed using uniform and smaller inert beads as a replacement. They suggested using two types of beads: plastic and glass beads, to determine the shrinkage limit of fine-grained soils. However, it is important to note that the use of plastic beads has limitations due to variations in their properties with changes in temperature and humidity, which can affect the results. In contrast, glass beads should be spherical and made of inert material, with a uniform size, ideally less than 1 mm, to ensure reliable results. Otherwise, variations in size could impact the accuracy of the test. Further, Kamil Kayabali (2013) introduced two new methods, namely, the dimension & density method and the reverse extrusion method to the determine shrinkage limit of soils. Dimension and density method requires sophisticated instruments like a gas pycnometer to determine the volume of soil particles and a digital caliper for measurement of the dimensions of the soil pat. This method is time-consuming and prone to human error. Its main drawback is the potential for inaccuracies due to challenges in precisely measuring small volume changes and the assumption of uniform shrinkage. In contrast, the results obtained from the reverse extrusion method are often considered unreliable, because for remoulded samples the ratio of extrusion pressure to the undrained shear strength varies as reported by O'Kelly (2019). This variability impacts the reliability of the results from this method.

Hobbs et al., (2014) have independently developed a sophisticated laboratory apparatus, that houses a traveling microscope, a laser range-finder, and digital balance to manually measure three-dimensional shrinkage, and hence, to determine shrinkage limit and other parameters without the use of mercury. This initial version of the apparatus was later improvised and a fully automated version of the apparatus referred to as 'SHRINKiT' (Hobbs et al. 2019) was designed, and constructed to determine the shrinkage limit. Though this instrument is commercially available and is found to be a useful apparatus for determining shrinkage limit, it appears to be not affordable by all laboratories due to its high initial cost.

Recently, Vincent et al., (2021) introduced an indirect method for assessing the shrinkage limit of well-compacted soils using the electrical resistivity (ER) method. This method allows for quick estimation of shrinkage limit by obtaining the relationship between moisture contents and ER of well-compacted soils. Though the method is interesting, it has certain limitations, among which the main limitation is the need for an ER instrument and the expertise to use it. Additionally, the relationship between ER and moisture content is non-linear and highly influenced by saturation levels, which can complicate the determination of accurate shrinkage limit. ER measurements are also sensitive to temperature changes, potentially leading to errors if not controlled. Further, this method infers volume changes and water loss instead of measuring them directly, which can result in interpretation errors if proper correlations are not established.

In light of the above-discussed drawbacks and shortcomings of various alternate methods to determine the shrinkage limit, it was felt necessary to develop a simple, yet reliable method of estimating the shrinkage limit.

The growing interest in hydrophobic compounds for creating water-repellent soils presents promising opportunities for innovation in geotechnical engineering. Various types of hydrophobic materials including silicon-based, fluorinated, wax-based, and polymer-based compounds are being effectively utilized for practical applications. Additionally, the recent focus on enhancing saline soils through hydrophobic or superhydrophobic modifications could lead to significant advancements in the durability and performance of such soils. Huang et al. (2023) examine the effectiveness of different hydrophobic agents in modifying saline soils to decrease water affinity and address issues such as salt expansion and chloride-induced corrosion. Even the use of wax to determine SL as proposed by Prakash et al (2009) is also a hydrophobic material. This concept has been further utilized in the present study to coat the dry shrinkage pats to ascertain the dry volume which is necessary for determining the shrinkage limit. This paper presents an alternate and simple method to determine the shrinkage limit by using easily available hydrophobic compounds to coat the dry shrinkage pats, and thereby, determine its volume by water submersion method suggested by ASTM (D4943-18, 2024).

2 Materials and Methods

Nineteen natural soil samples procured from various geological locations and one commercially available kaolinite with a wide range of plasticity properties were used in the present study. The soils used in the present study were characterized for their physical and plasticity properties adopting standard procedures as specified by Bureau of Indian Standards (SP36 1987) and the same has been summarized in Table 1. The position of soils used in the present study on the plasticity chart has been presented in Fig. 1.

Two different hydrophobic compounds, namely hydrophobic liquid and hydrophobic glue which are commercially available for water proofing in construction activities, and one hydrophobic compound used for cosmetic purposes, namely Vaseline were used in this present experimental work. Hydrophobic liquid (Mr. Bond-MB-100(WB) is a water dilutable transparent water repellant system, whereas Hydrophobic glue (Onesta crack seal glue) is used as a roof sealing agent is made of durable materials, friendly to the human body and the environment, and also non-toxic. Vaseline used in this study is a refined product obtained from petroleum, which contains mineral oil sand polycyclic aromatic hydrocarbons (PAHs), and it is commercially available in all pharmacies. Table 2 gives the density of hydrophobic compounds used in the study.

A shrinkage limit test was conducted on all the soils used in the present study as per the conventional mercury displacement method (ASTM D 427-04 2007; BS: 1377-2 1990; IS: 2720, Part 6 1972). For repeatability of the determination of shrinkage limit, thirty shrinkage pats were prepared for each soil used in the present study. The average value of shrinkage limit obtained from the mercury displacement method for these thirty trials has been reported in Table 3. Then, ten pats were used for each of the three hydrophobic compounds selected for this study to determine the dry volume of the shrinkage pats by coating the pats with a thin layer of hydrophobic compounds. The volume of dry shrinkage pats was determined by the water submersion method. The wet volume or initial volume of soil pats, which is equal to the volume of the shrinkage dish used was determined with water instead of mercury. The details of the experimental procedure adopted to determine the volume of wet and dry soil pats are presented in the subsequent section.

2.1 Determination of Wet Volume of Shrinkage pat

Conventionally, the volume of wet soil pat, which is equivalent to the volume of the shrinkage dish used in the determination of shrinkage limit, was determined by the use of mercury. After the use of mercury was banned in most of the laboratories world over, researchers have explored alternate methods to the use of mercury to determine the volume of shrinkage dish. The suggested alternate procedures are the use of water instead of mercury (ASTM D4943-02, 2008, ASTM D4943-18, 2024; Byers, 1986) or use of digital calipers to measure the dimensions of the dish

Table 1 Physical properties of soils used in the present study

Sl No	Soil type and location	Atterberg limits			Grain size distribution			Free swell ratio	IS Classification
		Liquid limit, w_L (%)	Plastic limit, w_P (%)	Plasticity index I_p (%)	Sand (%)	Silt (%)	Clay (%)		
1	B C Soil, Chamarajanagar.	61.2	29.2	32.0	24	32	44	1.60	CH
2	B C Soil, Jharkand, Pithoria.	58.4	27.8	30.6	8	44	48	1.50	CH
3	Silty Soil, Kodagu.	57.6	NP	–	30	34	36	1.00	MH
4	B C Soil, Odisha.	55.8	27.4	28.4	21	37	42	1.33	CH
5	Silty soil, Kanasawadi.	53.1	NP	–	48	28	24	1.10	MH
6	Silty Soil, Mangalore.	49.4	NP	–	40	35	25	1.17	MI
7	Kaolinite (commercial)	44.2	NP	–	–	80	20	0.54	MI
8	Red Soil, Basavanagudi, Bangalore.	42.5	26.6	15.9	34	30	36	1.08	CI
9	B C Soil, Balangir, Odisha.	42.2	20.8	21.4	44	28	28	1.33	CI
10	Red Soil, Bellary.	40.4	23.7	16.7	56	19	25	1.20	CI
11	Red Soil, Kollegala	38.7	21.6	17.1	46	26	28	1.00	CI
12	Red Soil, Nelamangala.	36.0	15.5	20.5	46	25	29	1.00	CI
13	Red Soil, Yelahanka, Bangalore.	35.6	21.5	14.1	36	48	16	1.00	CI
14	Red Soil, Jharkand, Ranchi.	35.0	17.7	17.3	49	27	24	1.10	CI-CL
15	Red soil, T-Narasipura.	34.9	18.5	16.4	53	39	8	1.11	CI
16	Red Soil, Devanahalli.	34.7	17.9	16.8	53	30	17	1.00	CL
17	Red Soil, Doddaballapura.	33.4	17.8	15.6	54	22	24	1.00	CL
18	Red Soil, Sadashivanagar, Bangalore	33.0	15.8	17.2	74	20	6	1.00	CL
19	Red Soil, K.R. Circle, Bangalore.	31.6	17.7	13.9	89	9	2	1.00	CL
20	Red Soil, Ramanagara.	30.6	16.6	14.0	52	20	28	1.10	CL

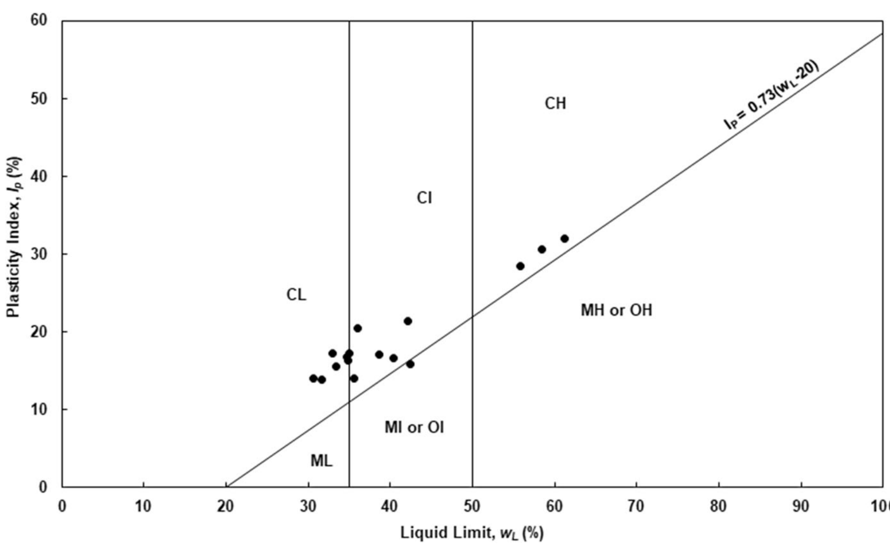

Fig. 1 Position on the plasticity chart of the soils used in the present study

Table 2 Density of hydrophobic compounds used in the study

Hydrophobic compounds	Density (Mg/m^3)
Vaseline	0.86
Hydrophobic liquid	0.97
Hydrophobic glue	1.27

(Prakash et al. 2009, 2011; Prakash and Sridharan 2012; Kayabali 2013). In the present paper, water was used to determine the volume of the shrinkage dish. The details of the procedure adopted in this study are presented in this section.

To determine the volume of wet soil mass, a thin layer of Vaseline (used as a water-tight seal) was smeared on the inner side of the shrinkage dish and one side of the plain glass plate. The mass of the dish along with the Vaseline coated glass plate was recorded to an accuracy of 0.01 g, M_1 (Fig. 2). The shrinkage dish was filled with water above its rim. The Vaseline coated side of the glass plate was slid over the top of the shrinkage dish to remove excess water. Care was taken to see that no air bubbles were entrapped. Water adhering to the shrinkage dish and plate was removed with tissue paper. The mass of the shrinkage dish filled with water and covered with the glass plate was recorded, M_2 (Fig. 3).

The difference between the two recordings M_1 and M_2 gave the mass of water required to fill the shrinkage dish. The temperature of water was recorded to obtain the absolute density of water for the corresponding temperature. The volume of the shrinkage dish was determined by dividing the mass of water filling the shrinkage dish by its absolute density.

Soil passing through 425 microns was mixed with distilled water to form a paste such that its initial water content was slightly above the liquid limit of the soil. To ensure complete saturation, this soil sample was transferred to a polythene cover and kept for 24 h in a desiccator filled with water. The subsequent day, the saturated soil specimen was transferred from the polythene cover onto a glass plate and mixed

Table 3 Shrinkage limit of soils obtained from the conventional mercury displacement method and from the three hydrophobic compounds used in the present study (Vaseline, hydrophobic liquid and hydrophobic glue)

Soil No.	Shrinkage limit, w_S (%)			
	Mercury method, $w_{S(Mer)}$	Vaseline method, $w_{S(V)}$	Hydrophobic liquid method, $w_{S(\mathrm{Hyp\ L})}$	Hydrophobic glue method, $w_{S(\mathrm{Hyp\ G})}$
1	8.0	10.5	NE*	8.5
2	9.9	8.5	NE*	9.6
3	29.4	29.2	26.6	30.1
4	7.8	6.9	6.5	9.6
5	21.2	20.4	20.2	22.8
6	23.9	23.1	NE*	24.1
7	27.8	28.0	NE*	27.1
8	15.0	14.0	14.1	15.9
9	8.0	7.3	6.5	9.6
10	12.8	11.4	10.7	12.3
11	12.5	11.5	11.0	14.8
12	12.4	12.2	10.7	12.0
13	13.0	11.6	11.5	15.7
14	13.1	12.2	12.5	12.3
15	16.3	15.5	15.5	16.1
16	18.1	15.5	15.4	16.6
17	14.2	11.4	12.5	14.5
18	17.2	15.8	13.8	16.2
19	18.5	17.0	14.8	17.7
20	14.4	13.8	12.4	14.1

*NE- Was not possible to evaluate shrinkage limit (The Reasons are brought out in the section on advantages and limitations)

Fig. 2 View showing recording the mass of empty shrinkage dish and glass plate coated with vaseline

Fig. 3 View showing recording the mass of shrinkage dish filled with water along with glass plate

thoroughly with a pair of spatulas to ensure uniformity of moisture content. The initial condition of the saturated soil samples as indicated by the initial water content was adjusted to have a cone penetration between 21 and 22 mm as checked using the Fall cone apparatus, which is used to determine the liquid limit of soils. Thereby, all the saturated samples had an almost uniform initial water content, being slightly above the liquid limit before it was transferred into the shrinkage dishes. The empty mass of the shrinkage dish, M_1 was recorded and then, the prepared soil sample was transferred into the shrinkage dish (whose inner surface was smeared with a thin layer of machine-grade oil) in a few layers. Care was taken to ensure no air bubbles were entrapped in the wet soil paste by tapping the shrinkage dish on a hard rubber base after placing each layer of the wet soil sample. After recording the mass of the shrinkage dish with the wet soil mass M_2, the dish was allowed to dry in air for one day to ensure gradual shrinkage of the sample. For all the soils used in the study, the shrinkage limit test was determined using 10 trials for each of the hydrophobic compounds used in this study. This was done to ensure repeatability. Then the shrinkage dish with the partially dried soil sample was kept in a thermostatically controlled oven maintained with a temperature of 105 °C for 24 h. The shrinkage dishes with oven-dried pats were removed from the oven and placed in a desiccator with calcium carbide, a desiccating agent, to bring it to laboratory temperature. Then, the mass of each of the shrinkage dishes with dry soil pat, M_3 was recorded. For soils having montmorillonite as a principal clay mineral, namely Black cotton soils (B.C. soils), which generally have difficulty getting an intact dry sample after shrinking due to the development of multiple cracks on drying, extra care was taken to air dry the sample in air for a week to a fortnight. Later, the shrinkage dish with the air-dried sample was placed on the top of the oven for a day, which was followed by placing it inside the oven for complete drying. The dry soil pats were labeled with a number of shrinkage dish for ease of identification (Fig. 4). Further, dry volume determination was carried out using mercury displacement for all 30 dry shrinkage pats. The pats were carefully transferred into polythene covers to

Fig. 4 A typical view showing dry shrinkage pats of few soils used in the study with numbering for identification

prevent the absorption of atmospheric moisture by the shrinkage pats. This was followed by coating 10 pats each with the three hydrophobic compounds. Later, after drying the surface-coated pats with hydrophobic compound at lab temperature, the dry volume of each of the pats was determined.

2.2 Procedure for Preparation Of dry Soil Pats with a Coat of Hydrophobic Compounds

2.2.1 Vaseline

In this method, dry shrinkage pats (ten in number) of each soil were uniformly coated with one thin layer of Vaseline using fingers such that it was sufficient to cover the surface of pats and then it was allowed to dry for a minimum of 24 h at laboratory temperature (22 ± 2 °C) (Fig. 5).

2.2.2 Hydrophobic Liquid

In this method, dry shrinkage pats (ten in number) of each soil were coated twice with hydrophobic liquid using a brush to ensure proper coating. After each coating of the pats, its surface was allowed to dry for 2 min at laboratory temperature (22 ± 2 °C) (Fig. 6).

2.2.3 Hydrophobic Glue

In this method, dry shrinkage pats (ten in number) of each soil were coated on the top side with hydrophobic glue using a brush and then allowed to dry at laboratory temperature (22 ± 2 °C). Since the hydrophobic glue was sticky, slightly more time (around 5 min) was needed to dry the coating on one side. Then, the same procedure was followed for the bottom side (Fig. 7).

Fig. 5 A typical view showing the shrinkage pats coated with vaseline

2.3 Determination of Volume of Dry Shrinkage Pat

In the first instance, for all the thirty sets of dry pats for each soil, the dry volume was determined conventionally by displacing mercury. Later, the volume of the dry shrinkage pats was determined by smearing the dry pat with a thin coat of the selected hydrophobic compounds as discussed in the previous paragraph (ten sets of dry pats for each of the selected hydrophobic compounds) to make the surface waterproof or hydrophobic. The details of the procedure adopted are explained below.

Fig. 6 A typical view showing the shrinkage pats coated with hydrophobic liquid

Fig. 7 A typical view showing the shrinkage pats coated with hydrophobic glue

i) A cradle was made with the help of 250–300 mm long sewing thread by tying loose ends together. The surface of the thread used was coated with Vaseline to prevent water absorption by the thread during water submersion test.
ii) The dry soil pat coated with the hydrophobic compound was placed on the loop of the thread.
iii) The tied end of the sewing thread was brought over the soil pat, and then, it was tightened by pulling the tied end through the looped end.
iv) The mass of the dry soil pat coated with a thin layer of hydrophobic compound along with the cradle was recorded in air using a balance of accuracy 0.01 g (Fig. 8). This is indicated as M_{sha}.
v) The dry soil pat coated with a thin layer of hydrophobic compound was submerged in a beaker containing water placed on a balance while being suspended from the suspension apparatus placed adjacent to the weighing balance (Fig. 9). The reading on the balance was recorded (Fig. 10), which indicates the apparent loss in mass when the pat is submerged in water (i.e., the numerical difference between M_{sha} and M_{shw}) or buoyant force acting on the dry soil pat coated with hydrophobic compound (Archimedes Principle). Hence, the recorded value of mass directly gives the volume of dry soil pat coated with hydrophobic compound, V_{sh}.
vi) Finally, the volume of dry soil pat, V_d was obtained by subtracting volume of hydropho-

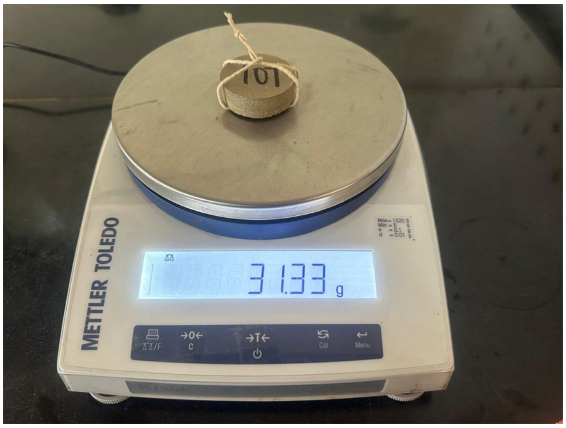

Fig. 8 A typical view showing recording the mass of dry soil pat coated with hydrophobic compound along with cradle before submersion

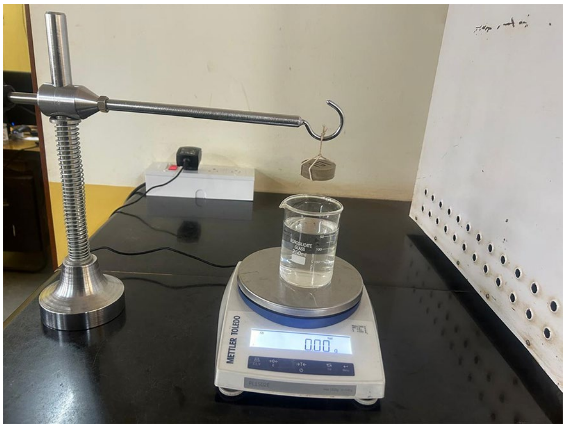

Fig. 9 A typical view showing the dry soil pat coated with hydrophobic compound and being suspended in air from suspension apparatus along with a beaker containing water placed on a weighing balance tared to zero before submersion

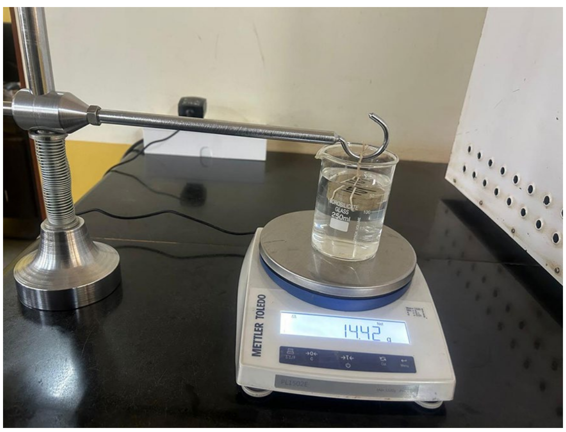

Fig. 10 A typical view showing recording the mass of the dry soil pat coated with hydrophobic compound when submerged in a beaker containing water through suspension apparatus

bic compound (V_h), from the volume of soil pat coated with hydrophobic compound (V_{sh}).

vii) Volume of hydrophobic compound, V_h used in the previous step was independently determined by knowing the mass of hydrophobic compound, M_h and the density of hydrophobic compound.

viii) The submerged soil pat was removed from beaker and the surface was wiped clean with a tissue paper to remove the surface water. Then the mass of the pat in air was again recorded (Fig. 11) to check the amount of water absorbed during immersion.

Fig. 11 A typical view showing recording the mass of dry soil pat coated with hydrophobic compound along with cradle after being removing from submersion

3 Results and discussion

For analysis purposes, the volume of dry shrinkage pat, V_d determined by conventional mercury method is denoted as $V_{d(Mer)}$ that by vaseline method as $V_{d(V)}$, that by hydrophobic liquid as $V_{d(\mathrm{Hyp\ L})}$ and that by hydrophobic glue as $V_{d(Hyp\ G)}$. Similarly w_s determined by the conventional mercury method is denoted by $w_{s(Mer)}$ and by the Vaseline method as $w_{s(V)}$, by the hydrophobic liquid method as $w_{s(Hyp\ L)}$, and by the hydrophobic glue method as $w_{s(Hyp\ G)}$. Figures 12, 13 and 14 compare the volume of dry soil pats obtained from the three hydrophobic compounds used in the present study, namely, Vaseline, hydrophobic liquid, and hydrophobic glue, respectively to the volume obtained from the conventional mercury displacement method. From the plots, it is very evident that there is a good relation between the volume of dry pat obtained by the mercury method and all three hydrophobic compounds. Table 3 shows the comparison of values of the shrinkage limit of soils

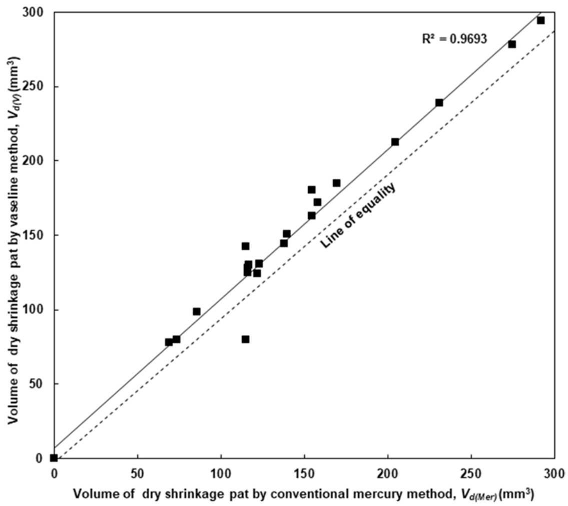

Fig. 12 Comparison of volume of dry shrinkage pats obtained from Vaseline method and conventional mercury displacement method

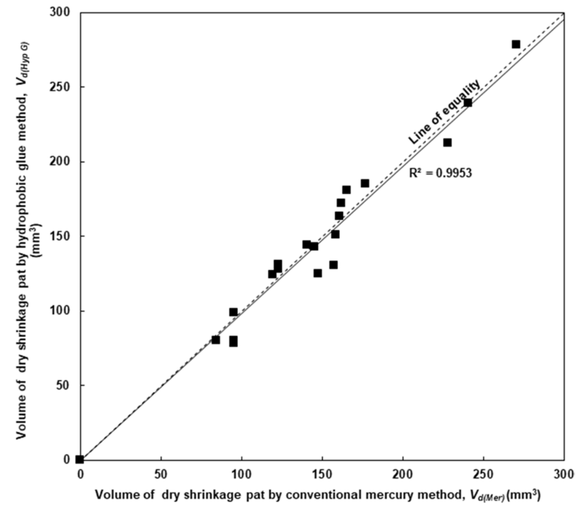

Fig. 14 Comparison of volume of dry shrinkage pats obtained from hydrophobic glue method and conventional mercury displacement method

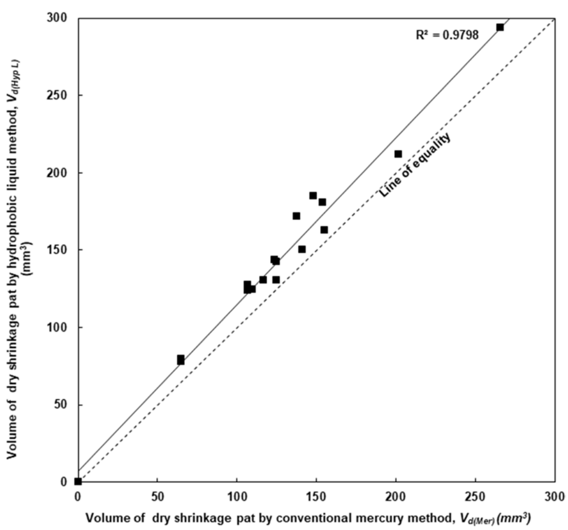

Fig. 13 Comparison of volume of dry shrinkage pats obtained from hydrophobic liquid method and conventional mercury displacement method

used in the study obtained from the mercury method and the proposed method using three hydrophobic compounds. Differences between the values of the shrinkage limit obtained for the soils used in the present study by the mercury method and the proposed method using three hydrophobic compounds, namely Vaseline, hydrophobic liquid and hydrophobic glue do not exceed ± 2.8%, ± 3.7%, and ± 2.7 respectively. The difference between the values obtained by the mercury method and the proposed method using hydrophobic compounds has been observed to be less than or equal to ± 3.7%. Since, ASTM standard D427-04(2007) and ASTM standard D4943-02 (2008) suggest the acceptable range of two results from two different tests of the same type, for multilaboratory determination, is + 4.8% for the mercury displacement method. Hence, the shrinkage limits of soils obtained by the proposed method using hydrophobic compounds are within the acceptable range. The same has also been presented graphically through Figures 15, 16 and 17. From the plots, it is very evident that there is a good relation between the shrinkage limit obtained by using the hydrophobic compounds and conventionally determined from the mercury displacement method. Further, Table 3 shows that the shrinkage limit could not be determined using a hydrophobic liquid for a few samples. During the determination of the dry volume of few black cotton and silty soils, it was discovered that the samples coated with hydrophobic liquid were absorbing water when determining it volume by water submersion method; hence the volume determination was discontinued. This was also observed for repeated trials. For such samples, it was indicated that the shrinkage limit was not evaluated (NE).

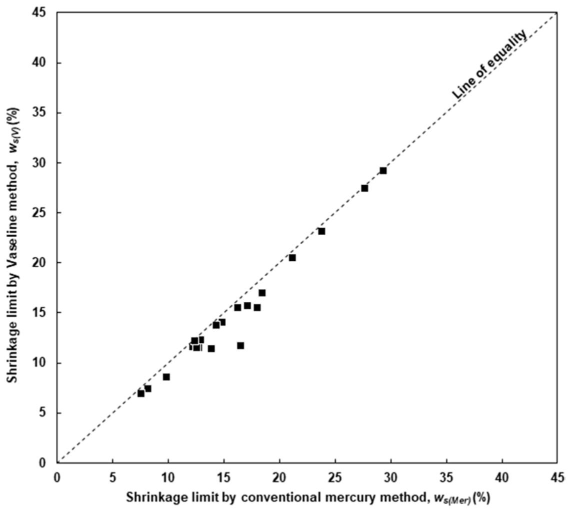

Fig. 15 Comparison of shrinkage limit of soils obtained from Vaseline method and conventional mercury displacement method

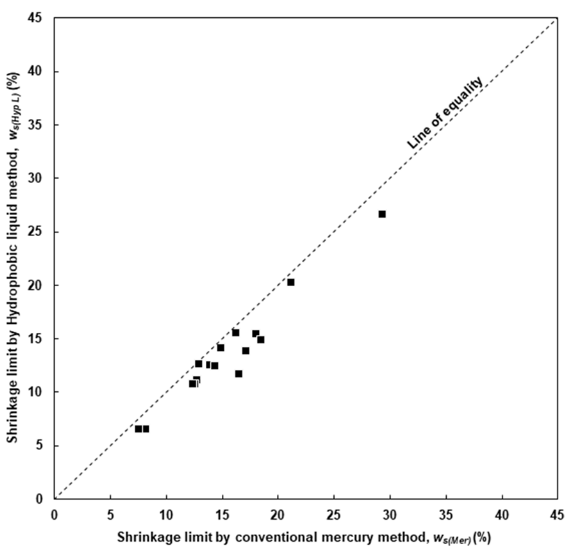

Fig. 16 Comparison of shrinkage limit of soils obtained from hydrophobic liquid method and conventional mercury displacement method

To ascertain the efficiency of hydrophobicity in the compounds used to coat the dry shrinkage pats and its influence on shrinkage limit determination, the average water absorption by pats coated with hydrophobic compounds was also examined, as shown in Table 4. The percentage

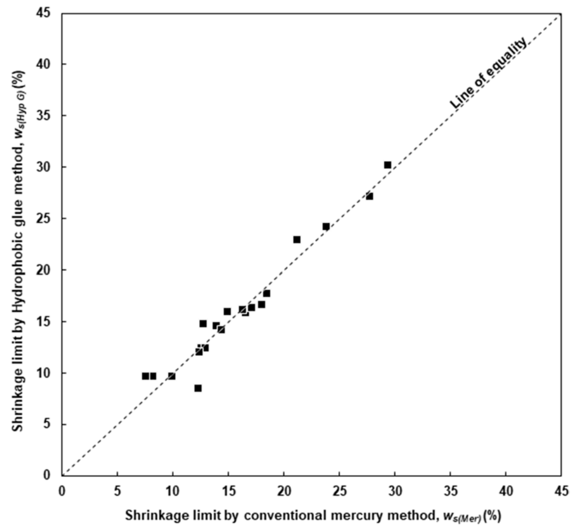

Fig. 17 Comparison of shrinkage limit of soils obtained from hydrophobic glue method and conventional mercury displacement method

Table 4 Percentage of water absorption of shrinkage pats after submerging in water

Percentage of water absorption (%)			
Soil	Vaseline method	Hydrophobic liquid method	Hydrophobic glue method
1	0.4	–	0.2
2	0.4	–	0.6
3	0.1	0.4	0.2
4	0.6	1.2	0.2
5	0.1	0.7	0.2
6	1.0	–	0.4
7	0.6	–	0.7
8	0.3	0.5	0.3
9	0.2	1.2	0.3
10	0.3	2.1	0.2
11	0.2	0.3	0.0
12	0.3	0.2	0.0
13	0.2	0.1	0.1
14	0.1	0.2	0.1
15	0.9	0.3	0.2
16	0.8	0.7	0.4
17	0.0	0.3	0.0
18	0.0	0.3	0.1
19	0.0	0.3	0.1
20	0.0	0.4	0.0

of water absorption observed with the Vaseline method varied from 0 to 1.0%, with the hydrophobic liquid method it varied from 0.1 to 2.1%, and with the hydrophobic glue method it varied from 0 to 0.7%. It is evident that the hydrophobic glue and Vaseline method absorb less than 1% of water. However, using the hydrophobic liquid approach, water absorption was slightly more than 1% for a few samples. Since the majority of the samples have water absorption values below 1%, it can be considered to have a marginal influence on the shrinkage limit determination.

3.1 Relative Advantages and Limitations of the Use of Hydrophobic Compound Method to Determine Shrinkage Limit

- The Vaseline method requires at least 24 h of drying at room temperature before evaluating the volume of dry shrinkage pat as compared to not more than 5 minutes for similar volume determination using hydrophobic glue and hydrophobic liquid.
- As previously discussed for black cotton soils (expansive soil) and silty soil, it is observed that the dry soil pat coated with hydrophobic liquid begins to absorb water while immersed in water for the determination of its dry volume, as indicated by the change in readings over time. This is a significant constraint in the case of such soils. However, this limitation was not noticed with Vaseline or hydrophobic glue, making it suitable for all types of soils.

4 Conclusions

The shrinkage limit of fine-grained soils is a significant parameter for assessing their volumetric stability. The standard method of evaluating shrinkage limit makes use of mercury to determine the exact volume of dry shrinkage pats. Identifying the hazardous nature of mercury, its use has been banned in many of the international laboratories. So, various alternative methods were proposed by researchers in the recent past. However, these alternate methods have their own limitations. To overcome these limitations, the present study was taken up to explore the possibility of using hydrophobic compounds in developing a simple and reliable alternate method of determining the volume of dry shrinkage pat and thereby evaluating its shrinkage limit.

In the present study, three hydrophobic compounds namely, Vaseline, hydrophobic liquid, and hydrophobic glue were selected and used to coat the dry shrinkage pats to determine their volume by water submersion method. In general, the values of shrinkage limit evaluated by the use of hydrophobic compounds match well with that determined by conventional mercury displacement.

Among the three hydrophobic compounds used in this study, Vaseline and hydrophobic glue are suitable for all types of soils. Hydrophobic glue dries quickly, allowing for faster volume determination, and hence, becomes the best option when quick results are desired. On the other hand, due to the easy availability of Vaseline in pharmacies, it can become a better option if, hydrophobic glue is not easily accessible. The use of hydrophobic liquid has certain limitations, specifically to expansive soils (e.g., B.C. soils) and kaolinitic silty soils, as these types of soils absorb the liquid compound, and thus make it difficult to evaluate the shrinkage limit. Based on the observations from this detailed experimental study, it is possible to use hydrophobic compounds to evaluate shrinkage limit of soils. Further, it is recommended to use hydrophobic glue or Vaseline to determine the dry volume of shrinkage pats using the water submersion method. The efficacy of the proposed hydrophobic method to evaluate shrinkage limit can be tried in various laboratories worldwide before finding acceptance and becoming a standard method instead of the conventional mercury displacement method.

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Data availability The datasets used and/or analysed during the current study are available from the corresponding author upon reasonable request.

Declarations

Conflict of interest The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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