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Amal Azad Sahib [a,b] and Retnamony G. Robinson [c]

ªAssociate Professor, TKM College of Engineering, Kollam, India; ᵇQIP Research Scholar, Department of Civil Engineering, IIT Madras, Chennai, India; ᶜProfessor, Geotechnical Engineering Division, Department of Civil Engineering, Indian Institute of Technology Madras, Chennai, India

ABSTRACT

Marine clays are reported to undergo an irreversible change in plasticity on drying. Various reasons such as the presence of halloysite and allophane minerals that change their structure, presence of sesqui-oxides or organic matter that undergo cementation and presence of salinity that generates strong attractive forces leading to aggregation on drying have been reported. In this study, the properties of Cochin marine clay, obtained from Cochin, India, in air-dried and oven-dried conditions were evaluated. The study shows that the plasticity characteristics get altered on drying, but can be reversed by proper dispersion using dispersion tools.

Abbreviations: CMC: Cochin marine clay; LL: liquid limit; PL: plastic limit; SL: shrinkage limit

1. Introduction

Atterberg limits such as liquid limit and plastic limit give significant insight about the engineering behaviour of fine-grained soils. The liquid limit and plasticity index are needed for soil classification. They are often used to predict the engineering properties of fine-grained soils. Hence, the determination of these limits in the laboratory warrants much precision.

Clays that exhibit an irreversible change in physical and mechanical properties on drying are found in many parts of the world. Central America, Japan, India, Indonesia, New Guinea, New Zealand, Kenya and Java are a few regions where this behaviour is reported (Pandian, Nagaraj, and Sivakumar Babu 1993). In India, Kuttanad clay and Cochin Marine Clay belong to this category (Ayyar 1966; Rao, Sridharan, and Chandrakaran 1989; Suganya and Sivapullaiah 2015). The compressibility and shear strength behaviour of these soils also undergo significant change on drying (Pandian, Nagaraj, and Sivakumar Babu 1991).

The postulated causes of irreversible change in properties on drying are many. Alteration of clay mineralogy on dehydration, particle aggregation, presence of gibbsite and allophane, presence of cementing agents such as iron oxides, carbonates and organic matter, prevalence of high pore water salinity, presence of divalent and trivalent cations in the pore fluid are some of the reasons attributed to this behaviour (Townsend 1985; Rao, Sridharan, and Chandrakaran 1989). Clays that exhibit changes in plasticity on drying present many limitations when brought from field to laboratory, especially when they undergo substantial drying. Many standard sample preparation procedures insist that the soils be dried for the sake of uniformity and sieving through 425 micron sieve, before performing Atterberg limits tests. While IS: 2720 (Part 5)-1985 recommends air drying before performing the

tests, ASTM D4318–17 highlights the importance of wet specimen preparation for soils that exhibit changes on drying and recommends the usage of wet preparation technique unless the dry preparation is specified by the requesting authority.

The plasticity characteristics, grain size distribution and subsequently compressibility behaviour of the dried soils obtained in the laboratory deviate largely from the soils in field condition. Stott and Theron (2015) report large number of failures in a subsidy housing project on clay soil in Botshabelo, South Africa; for which the predicted heave was half of that was actually observed. The heave prediction methods were based on the commercial laboratory values of liquid limit and plasticity index of dried soil. Compared to natural condition of soil, the plasticity characterisitics in the dried form were reduced and hence the heave predicted for natural soils were underestimated. With reference to the tropically weathered residual soils of Ethiopia that were employed in earthworks, Teklay et al. (2015) recommend using the soils in their natural state, without subjecting to drying for obtaining grain size distribution and plasticity data.

In most of the studies on the irreversibility of dried clays, the procedure of rewetting and remixing the dried samples is not clear. Ayyar (1966) conducted Atterberg limits tests on Kuttanad clay (from Kerala state, India) processed in various conditions namely natural, air dried, partially air-dried, oven-dried, treated with hydrogen peroxide (to remove organic matter) in dried and undried forms. Sodium hexametaphosphate solution was also used to disperse the dried clays. In all cases, the liquid limit of the soil got reduced from the natural conditions with the drying water content of the soil. Drying causes profound reduction in plasticity, which was attributed to the strong attractive forces generated at close contacts. Ayyar (1966) reported that no chemical changes occurred and that the formation of oriented aggregates of the small

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Table 1. Liquid limit values on drying (from the literature).

Reference	Soil		Liquid limit (%)		
			Natural state	Air dried	Oven dried
Ayyar (1966)	Kuttanad clay, India		154	75	53
Gidigasu (1974)	Laterite clay, Ghana		245	217	Non-plastic
Rao, Sridharan, and Chandrakaran (1989)	Cochin marine clay, India		116	77	68
Jose (1989)	Cochin marine clay, India	Munambam	117	-	57
		Nettoor	138	88	55
		Cheranelloor	119	97	57
		Parur	108	73	57
		Maradu	137	105	80
		Kumbalam	96	80	56
Pandian, Nagaraj, and Sivakumar Babu (1991)	Cochin marine clay		106	84	60
Pandian, Nagaraj, and Sivakumar Babu (1993)	Cochin marine clay (Parur), India		106	70	60
	Red soil, India		-	50	44
	Brown soil, India		-	62	55
	Black cotton soil, India		-	84	77
Abraham (1993)	Cochin marine clay, India		129	93	76
	Mangalore marine clay, India		104	88	73
Basma, A1-Homoud, and Al-Tabari (1994)	North Jordan, Jordan	Azraq	110	79	73
		Irbid 1	79	88	70
		Jerash 1	49	48	41
		Irbid 2	77	80	69
		Jerash 2	84	80	78
Sridharan, Shafei, and Miura (2000)	Ariake clay, Japan	Kawazoe	116	-	69
		Kubota	68	-	58
		Isahaya	170	-	90
		Fukudomi	134	-	87
Navaneethan and Sivakumar (2002)	Belfast Upper Boulder clay, UK		61	58	56
Suganya and Sivapullaiah (2015)	Kuttanad clay, India		137	78	66

clay fraction is the real cause of irreversible behaviour. It was reasoned that these soil particles do not swell back considerably due to the crossing of energy barrier.

Rao, Sridharan, and Chandrakaran (1989) investigated the factors responsible for change in the liquid limit behaviour of Cochin Marine Clay. The dried specimens were soaked for a period of 15 days before testing for Atterberg limits. They concluded that the presence of divalent/trivalent ions and a high pore salt concentration facilitates strong inter-particle attraction and small particle separation, whereas monovalent Na ions promote strong interparticle repulsion. Cochin marine clay consists of divalent and trivalent ions that permit an intimate contact of particles during drying, thereby reducing the available surface area for interaction with water and hence reduction in liquid limit. In the discussion following Rao, Sridharan, and Chandrakaran (1989), Ayyar et al. (1990) reiterated the role of organic matter in bonding the individual units, as cementing compounds.

Pandian, Nagaraj, and Sivakumar Babu (1991) also indicated the change in the index and engineering properties of Cochin Marine Clay (from Cochin, Kerala State, India) on air drying and that the test results of standard procedures which recommend air drying could be misleading. They had soaked the dried specimens in distilled water for different time periods up to 12 months and reported that the plasticity values were still not reversed back to the natural state. They also compared with other soils showing similar trend on air drying and concluded that liquid limit state can be used as a good reference for identifying the change in engineering behaviour with drying (Pandian, Nagaraj, and Sivakumar Babu 1993).

Basma, A1-Homoud, and AI-Tabari (1994) followed a pulverization procedure on dried soil specimens from north Jordan that were soaked subsequently in distilled water. Suganya and Sivapullaiah (2015) investigated the effects of drying on the chemical and geotechnical properties of Kuttanad clay. The dried specimens were pulverized in a ball mill before soaking in water for a month. Both studies reported no alteration in the reduced plasticity characteristics. A summary of the change in liquid limit that occurs on drying is given in Table 1.

The table clearly shows that the liquid limit values get significantly altered on drying. The reduction in liquid limit can be as high as 60%. However, the change in plastic limit is marginal (Rao, Sridharan, and Chandrakaran 1989). The above review suggests that the Atterberg limits of clays get affected due to drying. Attempts were also made in the literature to revert the liquid limit by different means, with limited success. This study focuses on disintegrating the aggregated structure through a high-energy mechanized dispersion technique in an attempt to reverse the plasticity of Cochin marine clay (which is known for change in properties on drying).

2. Basic properties and preliminary drying studies

In this study, Cochin marine clay (CMC) from Cochin region, situated in the south western coast of India, is used to study its behaviour on drying. The clay is normally consolidated young clay with high compressibility and low shear strength. These clays are well known for their drying-induced changes in plasticity behaviour. The sample was procured from Vallarpadam, Cochin, India, at a depth of 8–10 m. The samples, brought from the field, were stored air tight, in sealed bags and laid in water-filled desiccators to avoid loss of moisture. The natural moisture content of Cochin marine clay was 116%. A pH of 8.5 suggests alkaline nature whereas electrical

conductivity is 1.11 mS/cm indicating salinity. The organic matter determined using Loss on Ignition method as per AASHTO T 267–86 (2013), is 6%. The X-Ray diffraction patterns indicated that CMC comprised of the minerals Illite, Montmorillonite, and Kaolinite; illite being the predominant clay mineral. The grain size distribution curve was obtained by wet sieve analysis. In natural condition, CMC had 37% silt and 63% clay size particles.

The liquid limit and plastic limit values were determined using Casagrande's method as per Indian Standard Specifications (IS: 2720-Part 5 1985). Tests were conducted in three conditions such as in natural state, air-dried state and oven-dried state. In order to determine the liquid limit in the natural state, water was added to the soil in its natural state and mixed thoroughly using a spatula. The specimen was spread in a glass plate and allowed for moisture loss. At intermittent times, the soil was thoroughly mixed again and liquid limit test was carried out.

For air drying, the sample was spread uniformly on a glass plate and exposed to room temperature of 35–40°C for several days. Oven drying of the samples was carried out at 110°C in a temperature-controlled hot-air oven. After drying, the specimens were soaked in distilled water to sufficiently rewet and further thoroughly hand mixed to determine the liquid limit and grain size distribution.

The results of Atterberg limit tests and grain size analysis for all the three conditions are listed in Table 2. The liquid limit values, after air drying and oven drying for CMC are 77% and 66%, which are 25% and 36% lower than those of soils in natural condition. The reduction in plastic limits is marginal. The shrinkage limits are practically the same for all the conditions.

It can be seen that the clay fraction got reduced, probably as a result of aggregation, on drying. The reduction in plasticity and reduction in clay content are consistent with previous studies (Ayyar 1966; Rao, Sridharan, and Chandrakaran 1989; Jose 1989; Pandian, Nagaraj, and Sivakumar Babu 1993, 1991; Abraham 1993; Suganya and Sivapullaiah 2015).

Liquid limit versus drying water content

From the previous section, the change in liquid limit with drying is evident in Cochin marine clay. It became pertinent to find out at which stage of drying, the irreversible change sets in. To observe this closely, different specimens of CMC were dried at room temperature to various water contents from slurry state. The specimens were placed in flat trays and dried from the slurry state with water content of 130% in ambient room conditions. As the specimens attained different water contents after drying, they were subsequently tested for liquid limits. The results are plotted in Figure 1. The liquid limit, plastic limit and shrinkage limit of the soil in their field condition (without drying) are also indicated on the plot. The liquid limit values after drying to various initial moisture contents get reduced. The reduction in liquid limit is predominant in the region where the specimens were dried at water contents below plastic limit. In other words, when the natural clay gets dried to water content below plastic limit, the liquid limit gets reduced. Beyond shrinkage limit, the change is most predominant. One of the reasons why this happens may be aggregation due to shrinkage stresses.

To verify the role of shrinkage stresses upon drying, suction values were measured in Cochin marine clay specimens using Dew point Potentiameter (WP4) that employs the chilled mirror hygrometer. The device is used as a rapid means of determining the total suction of unsaturated soils (Leong, Tripathy, and Rahardjo 2003; Vikas and Singh 2005). The specimens were filled in slurry state (about 1.5 times liquid limit) in a mould of 35 mm diameter and 7 mm height. The soil sample was placed in a sealed chamber containing a mirror with a detector of condensation. The dew-point is the
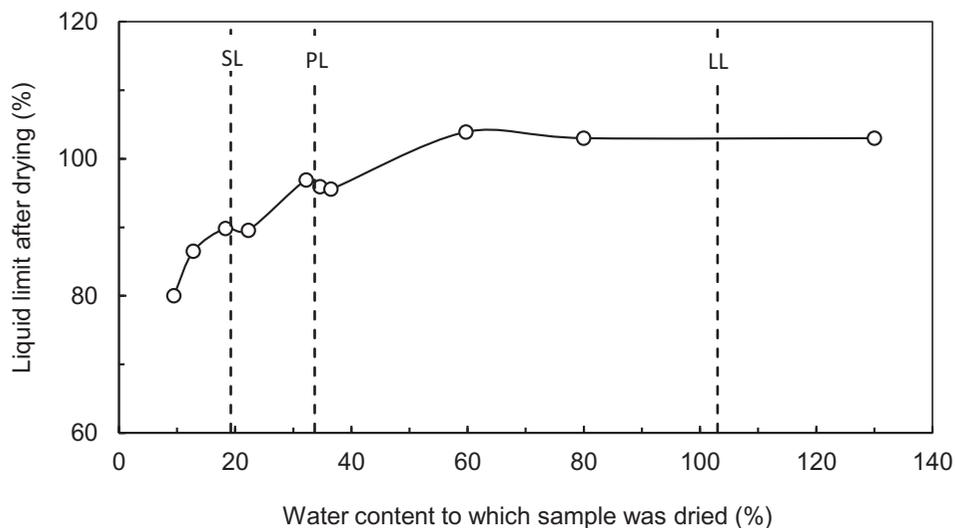
Table 2. Properties of Cochin marine clay at three conditions.

	Liquid limit (%)	Plastic limit (%)	Shrinkage limit (%)	% silt size	% clay size
Natural state	103	34	19	37	63
After air drying	77	33	19	43	57
After oven drying	66	35	20	43	57

Figure 1. Liquid limit variation for Cochin marine clay with change in initial moisture content of the sample.
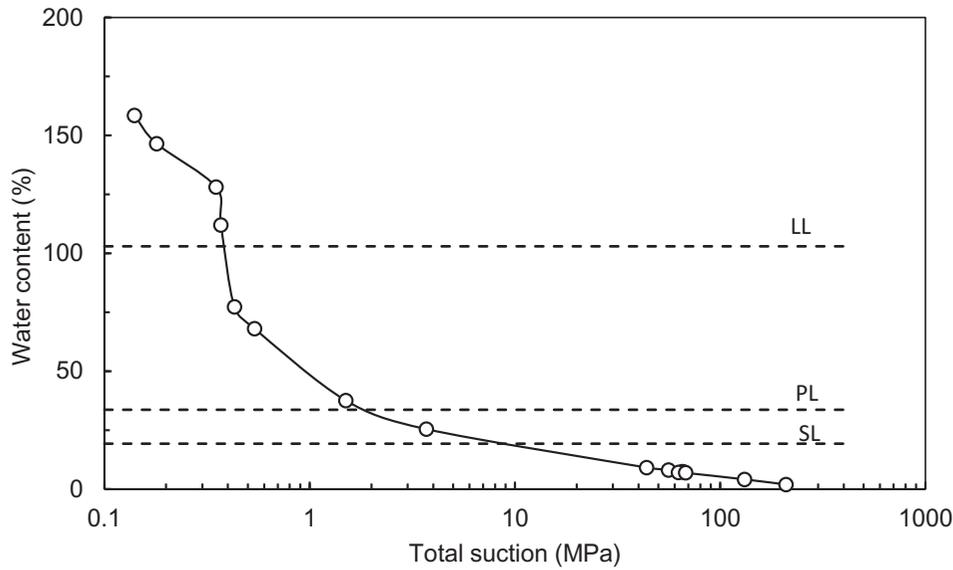
Figure 2. Gravimetric water content versus total suction for Cochin marine clay.

temperature to which the air must be cooled so that the water vapour in the air condenses to liquid water. At the dew-point, the water vapour present in the air is just sufficient to saturate it. When equilibrium prevails, the relative humidity of the air in the chamber is equal to the relative humidity of the soil sample. Relative humidity is calculated as the ratio of the saturated vapour pressure of water at the dew-point to the saturated vapour pressure of water at the air temperature. This ratio can be substituted in the following thermodynamic equation to calculate the total suction pressure.

$$\psi = \frac{-RT}{v_{wo}w_v}\ln\left(\frac{u_v}{u_{vo}}\right) \qquad (1)$$

where ψ is the soil suction or total suction (kPa), R is the universal (molar) gas constant (8.31432 J/(molK)), T is the absolute temperature ($T = (273.16 + t^0C)$ K), t is the temperature in ^{0}C, v_{w0} is the specific volume of water, w_v is the molecular mass of water vapour (i.e. 18.016 kg/kmol), u_v is the partial pressure of pore water vapour (kPa), and u_{v0} is the saturation pressure of water vapour over a flat surface of pure water at the same temperature (kPa). The term u_v/u_{v0} is called the relative humidity (R_h) and can be substituted in equation (1). With the help of a built-in software on the instrument, the suction of the soil sample (in MPa) is displayed on the LCD panel along with its temperature.

The suction measurements of Cochin marine clay are plotted in Figure 2. The total suction is the sum of osmotic and matric suction. At initial water contents, the total suction is about 0.2 MPa or 200 kPa. Considering the matric suction in the initial stages to be zero, as the soil is saturated, this value refers to the osmotic pressure. This high initial value is indicative of the high salinity of the soils. Osmotic suction is negligible in the absence of salts (Thakur and Singh, 2005). It can also be observed that below the water contents corresponding to plastic limit, the suction values increase rapidly which has led to the strong aggregation of colloidal fraction. Assisted by the attractions generated by the osmotic ions, this aggregation may be strong enough not to be disintegrated on further rewetting. Osmotic

suction is also known to increase with reduction in water content (Krahn and Fredlund 1972). This could be a reason for the consistent reduction in the liquid limit values of the soils, dried below plastic limits as observed from the previous section.

3. Reversing the Atterberg limits of the dried samples

Previous attempts in the literature to reverse the Atterberg limit of dried samples were not successful (Ayyar 1966; Pandian, Nagaraj, and Sivakumar Babu 1991; Basma, A1-Homoud, and AI-Tabari 1994; Suganya and Sivapullaiah 2015). In the present study, a heavy duty dispersion tool (IKA-T 50 Digital Ultra Turrax Disperser with S50N-G45F dispersing element of 45 mm stator diameter and 40 mm rotor diameter), as shown in Figure 3, was used to disperse the dried soil. The dispersion is effected by a forced vortex action that allows an inward-out motion of the particles in suspension. Soil suspensions constituting 200 g of soil solids in 800 ml suspension were used for every dispersion trial. Some initial trials were conducted to fix the speed and time of mixing. Subsequently, a speed of 9000 rpm and dispersion time of 30 min was fixed. The dispersed specimens were subjected to liquid limit determinations as per standard procedures.

4. Results and discussions

Figure 4 shows the flow curves obtained for soils in various conditions in the Casagrande's liquid limit test. The results obtained for the dispersed air-dried and oven-dried specimens are compared with the natural, air-dried and oven-dried specimens not subjected to the dispersion technique. These results indicate that the dispersion technique has effectively dispersed the clay particles aggregated on drying and increased the liquid limit values closer to the original values in natural state. Table 3 gives the results of liquid limit tests on the dispersed soil specimens along with that of the undispersed soil specimens.

Cochin marine clay has a liquid limit of 103% in its natural state, 77% in air-dried condition and 66% in oven-dried

(a)

(b)

Figure 3. (a) IKA-T 50 digital ultraturrax disperser and (b) dispersing element.

condition. Dispersion has increased the values to 96% and 98% for air-dried and oven-dried specimens, which is 93% to 95% close to the liquid limits in natural condition. This technique has reversed the properties attained on drying close to that in its natural state. From the values of plasticity indices, it is clear that the natural plasticity is restored well.

Physically, the change can be observed in Figure 5(a,b) wherein before dispersion, the dried soil particles in solution settle due to floc formation. After dispersion, the clay particles are deflocculated and hence remain in suspended form. In order to check whether the grain size of the dispersed soils is different from the natural conditions, hydrometer analysis was carried out and the results are compared in Figure 6. The primary observation of clay aggregation that leads to an increase in the silt size fraction and reduction of clay fraction was seen in the grain size distribution of air-dried and oven-dried soils (which were not subjected to dispersion).

Reduction of surface area and consequent loss in water-holding capacity led to the reduction in plasticity and hence lower liquid limit values. On dispersion, the grain size distributions indicate that the agglomerated clay particles were efficiently separated and the clay fractions returned to the values close to the natural state. The percentage clay fraction of Cochin marine clay is 63% in natural state; 57% in the undispersed air-dried condition
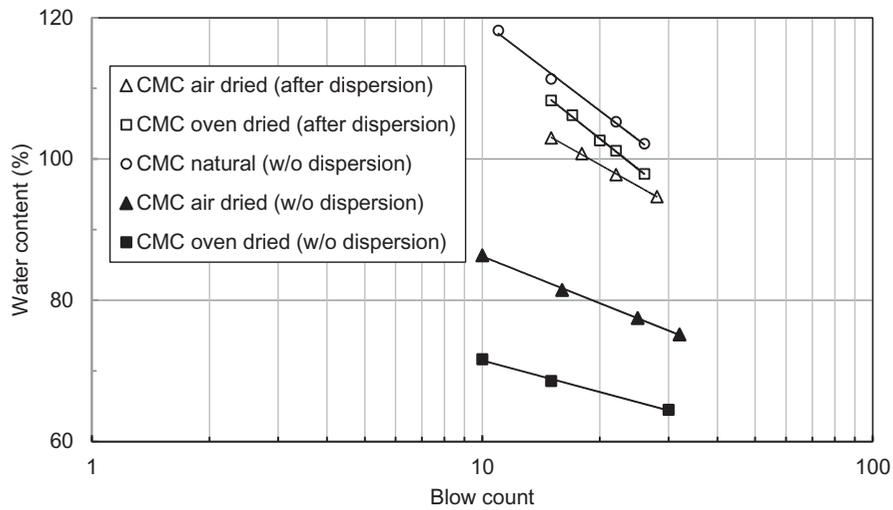
Figure 4. Comparison of flow curves for Cochin marine clay.

Table 3. Comparison of liquid limit and plastic limit for Cochin marine clay.

	Without Dispersion			With dispersion		
	Liquid limit (%)	Plastic limit (%)	Plasticity Index (%)	Liquid limit (%)	Plastic limit (%)	Plasticity Index
Air dried	77	33	44	96	33	63
Oven dried	66	35	31	98	38	60

and undispersed oven-dried condition. Dispersion increased the clay fraction to 63% for air-dried and 62% for oven-dried specimens, respectively; these values being the same as that of natural soil (Table 4). However, some amount of grinding of the sand and

silt-size fraction could not be ruled out in this technique, but nevertheless, the clay fraction obtained is representative of the natural soil. From the above results, it can be summarized that the dispersion technique is capable of reversing the properties acquired on drying.

5. Conclusions

The liquid limit of Cochin marine clays significantly get reduced due to particle aggregation. The usual practice of soaking in water and mixing using spatula is not able to disperse the clay particles significantly. Therefore, it was generally concluded in the literature that drying cause irreversible change in liquid limit. In the
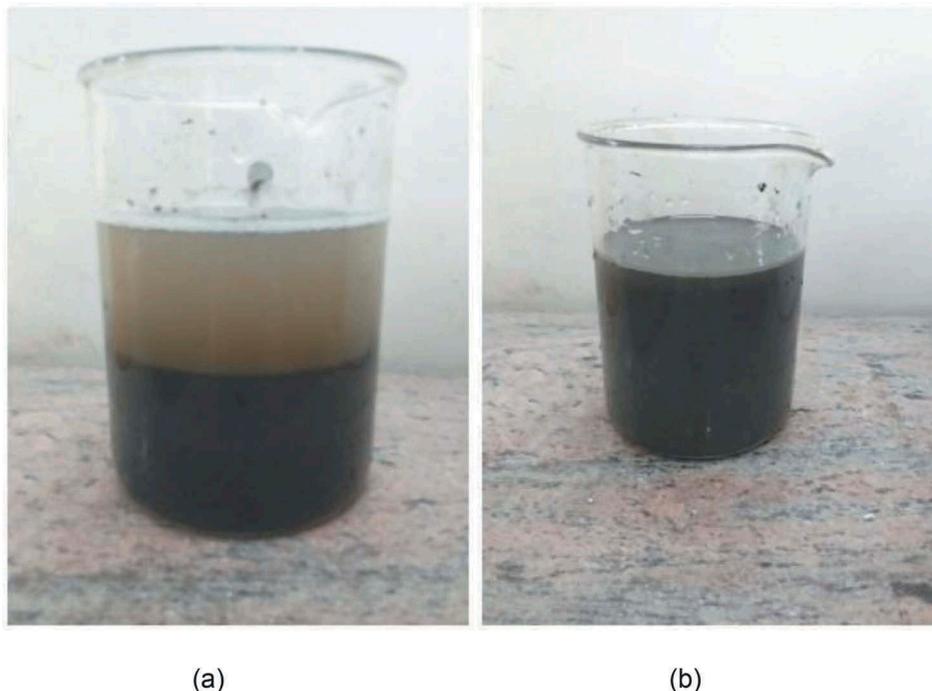

(a)   (b)

Figure 5. Images of oven-dried specimen in suspension (a) before dispersion (b) after dispersion.
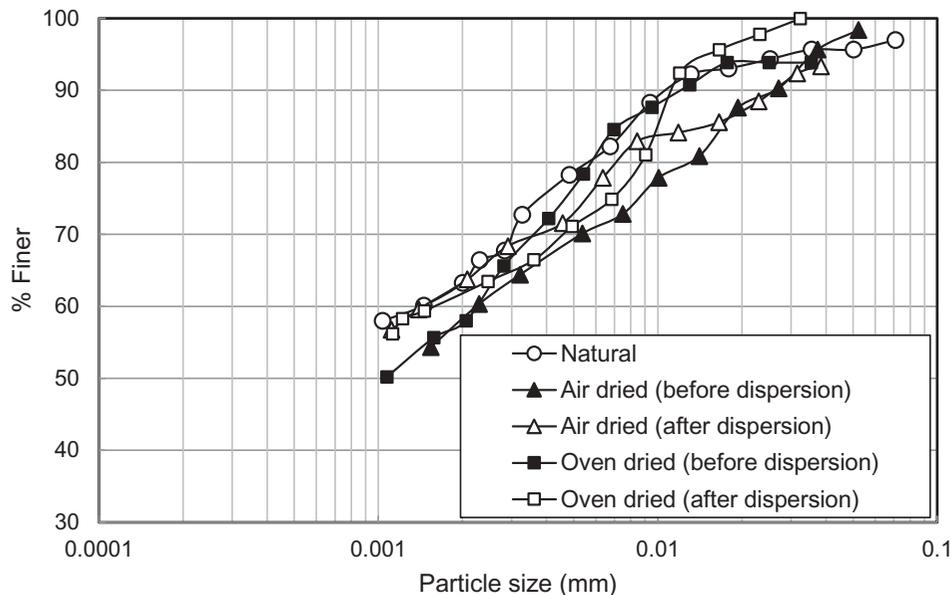
Figure 6. Grain size distribution by hydrometer analysis for Cochin marine clay in different conditions.

Table 4. Comparison of grain size distribution by hydrometer analysis for Cochin marine clay.

	Without Dispersion		With dispersion	
	Silt size (%)	Clay size (%)	Silt size (%)	Clay size (%)
Air dried	43	57	37	63
Oven dried	43	57	38	62

present study, a dispersion technique was attempted. The dispersion technique has significantly reversed the properties of Cochin marine clays, acquired on air drying and oven drying.

It is recommended that clays that undergo changes in plasticity characteristics on drying may be subjected to this dispersion technique when they were dried substantially from field conditions, in order to capture the plasticity and clay fraction of the original soils in reliable estimates.

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Disclosure statement

No potential conflict of interest was reported by the authors.

Notes on contributors

Amal Azad completed her B. Tech (Bachelor of Technology) degree in 2006 and M.Tech (Master of Technology) degree in Geotechnical Engineering in 2008 from College of Engineering, Trivandrum, India. She joined as faculty in Civil engineering at TKM College of Engineering, Kollam, India, in August 2007. Currently she is doing her PhD at Indian Institute of Technology Madras, Chennai, India.

Robinson R G is a Professor at Indian Institute of Technology Madras, Chennai, India. He works in the area of Soft clay engineering and Ground Improvement.

ORCID

Amal Azad Sahib   http://orcid.org/0000-0002-1408-3471
Retnamony G. Robinson   http://orcid.org/0000-0001-6114-7637

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