International Journal of Environmental Science and Technology (2024) 21:2121–2128
https://doi.org/10.1007/s13762-023-05022-4

SHORT COMMUNICATION

Effect of chitin, chitosan and NaCMC biopolymers on the consistency limits of organic silt

G. Kannan[1] · B. C. O'Kelly[2] · E. R. Sujatha[1]

Received: 29 September 2022 / Revised: 3 March 2023 / Accepted: 20 May 2023 / Published online: 9 June 2023

Abstract

With global concerns on carbon emissions, there is increased need for various eco-friendly alternatives to conventional (cement and lime) additives used in ground improvement applications. This paper investigates the effect of three biopolymers—chitin, chitosan, and sodium carboxymethyl cellulose (NaCMC)—on the consistency limits of low plasticity organic silt soil. The theory of soil mechanics suggests that understanding the soil's plasticity nature is helpful in predicting strength and compressibility characteristics. The variation in the Casagrande liquid limit (LL), plastic limit (PL), plasticity index, and flow index of the soil are presented for 0.5–4% chitin and chitosan dosages, and 0.25–1% NaCMC dosage. For all three biopolymers, the consistency limits (more-so for LL than PL) increased with increasing dosage, with NaCMC addition producing a more dramatic effect compared to chitin and chitosan additions. The biopolymer additives formed 'bridge' connections between the soil particles, helping to improve strength properties. This bridging effect explains the significant increases in LL (being essentially a strength-based parameter) for biopolymer addition. A greater LL increase was observed for NaCMC addition since it is a gellable biopolymer with moisture retaining property.

Keywords Organic soil · Liquid limit · Plasticity index · Chitin · Chitosan · Cellulose

Introduction

Biopolymers are eco-friendly substitutes for conventional chemical additives (i.e., cement and lime) typically used in ground improvement applications (Sujatha and O'Kelly 2023). These polymeric substances are produced either by the chemical synthesis of biomaterials, or through bio-synthesising actions of organisms, like bacteria. Polysaccharides are a class of biopolymer that contains long chains of carbohydrates. Several researches have investigated the role of polysaccharides in improving the geotechnical engineering behaviour of soils. Some examples include xanthan gum, alginate, guar gum and β-glucan, gellan gum, and gum Arabic. The soil is treated with these polysaccharides by techniques like grouting and dry mixing (e.g., Khatami and O'Kelly 2013, 2018, 2022; Chang et al. 2016; Soldo et al. 2020). Cellulose and chitin are the most abundant polysaccharides on Earth; cellulose being found in the stems of plants and the cell walls of certain microorganisms and plants, while chitin (and its derivative chitosan) are crustacean biopolymers. Chitin and chitosan have beneficial applications in various fields; e.g., as hemostatic agents in biomedical engineering, as coagulant in water treatment, and biological active agents in the cosmetic industry (Casadidio et al. 2019; Zhao et al. 2018). Sodium carboxymethyl cellulose (NaCMC) is a cellulose derivative commonly used as a flocculating agent, water-retaining agent, and food additive (Chen 2015). To date, the feasibility of employing these biocompatible materials (i.e., chitin, chitosan and NaCMC) for geotechnical engineering applications has received relatively

Editorial responsibility: Parveen Fatemeh Rupani.

✉ E. R. Sujatha
r.evangelin@gmail.com; sujatha@civil.sastra.edu

G. Kannan
rajkannan93@gmail.com

B. C. O'Kelly
bokelly@tcd.ie

[1] Centre for Advanced Research on Environment, School of Civil Engineering, SASTRA Deemed to be University, Thanjavur 613401, India

[2] Department of Civil, Structural and Environmental Engineering, Trinity College Dublin, Dublin D02 PN40, Ireland

little attention, with more research needed to fully utilize their benefits. Additionally, all three chosen biopolymers have different ionic natures and interaction behaviours. Research has shown that chitosan (Hataf et al. 2018; Shariatmadari et al. 2020; Soldo et al. 2020) and NaCMC (Ma and Ma 2019) have a good tendency to improve the soil strength, with their affect increasing for higher dosage and longer curing period. However, understanding the soil plasticity characteristics would be helpful in knowing the behaviour of the amended soil in varied moisture conditions. This study takes the initiative of exploring the consistency limits (i.e., liquid limit, LL, and plastic limit, PL) of an intermediate plasticity organic silt treated with chitin, chitosan and NaCMC additives. An understanding of the nature of the unamended and treated soil is obtained based on consistency limit test results, from which the effect on mobilised strength for biopolymer treatment at various dosages can be inferred.

Materials and methods

Test soil

The sampled soil was obtained from Ariyalur, Tamil Nadu, India. The soil was found to have an organic matter content of 13.6%, which explained its relatively low particle density of 2.33 Mg/m^3, and a moderate tendency to swell, with a differential free-swell index of 35%. Particle-size distribution analysis indicated that the soil was comprised of 1.6% gravel, 24.7% sand, 52.6% silt and 21.1% clay. Chemical analysis via X-ray fluorescence (XRF) showed that the dry soil consisted of 58.1% SiO_2, 16.4% Al_2O_3, 15.1% Fe_2O_3, 4.3% CaO, 2.1% TiO_2, 1.8% MgO, along with traces of other compounds, like K_2O, P_2O_5, etc. The experimental testing programme reported in this paper investigated the fine fraction of the sampled soil (i.e., material passing the 425-µm sieve size), which had a predominance of *silt*, as reflected by the grading curve shown in Fig. 1.

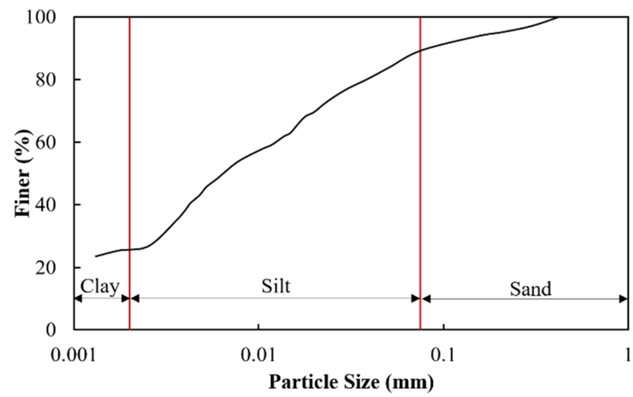


Fig. 1 Particle-size distribution plot of the tested soil

Chitin

Chitin is a non-ionic polysaccharide obtained by demineralising and deproteinising crustaceans, like crabs, prawns, and lobsters (Ohno 2007). Chitin has two different monomer structures of N-acetyl-glucosamine and N-glucosamine linked throughout the polymer chain (Min et al. 2015). It is insoluble in water and other organic solvents owing to its strong inter- and intra-molecular hydrogen bonding (Shirvan et al. 2018). Chitin-soluble substances exhibit toxic and corrosive nature (Shirvan et al 2018).

Chitosan

Chitosan is a cationic polysaccharide produced by deacetylation of chitin with alkali solutions (Cheung et al. 2015). It is insoluble in water, but dissolves in dilute organic acids, like acetic, formic and tartaric acids (Niaounakis 2015). However, Usmani et al. (2018) stated that this dissolution reduces the physical and chemical stability of chitosan. Hence, chitosan powder, with a degree of deacetylation of 85%, was used in the present study.

NaCMC

NaCMC is anionic and hydrophilic and is obtained as an ether derivative of cellulose prepared by alkalisation and carboxymethylation of natural cellulose (Chen 2015; Fink 2015). Natural cellulose does not readily dissolve in water due to the complex hydrogen bonding (Manoukian et al. 2019), whereas NaCMC readily dissolves in water owing to its hygroscopic and rapid hydration natures (Ergun et al. 2015). The present study used a NaCMC with a viscosity of 222 cP for 1% solution. Some physicochemical properties of the chosen biopolymers are listed in Table 1.

Experimental testing programme

Test-specimen preparation

In preparing homogeneous biopolymer-treated soil for testing, the powdered chitin, chitosan and NaCMC materials were added in required amounts to batches of the dry disaggregated fine soil, and then thoroughly blended together. Requisite amounts of water were then added

Table 1 Properties of the investigated biopolymers

	Chitin	Chitosan	NaCMC
Source	Crustacean	Crustacean	Plant
Molecular formula	$(C_8H_{13}O_5N)_n$	$(C_6H_{11}NO_5)_n$	$(C_8H_{15}NaO_8)_n$
Colour	Brownish-white	Brownish-white	White
Average particle diameter (μm)	105.92 ± 12.57	139.63 ± 21.14	397.90 ± 17.62
Density (kg/m^3)	247.21	286.90	700.00
Molecular weight of one monomer (g/mol)	203.22	161.00	262.19
Water solubility	Nil	Nil	Soluble
pH (1% suspension/solution)	5.56	5.97	6.82
Ionic nature	Non-ionic	Cationic	Anionic

to achieve targeted water contents, followed by further thorough mixing, after which the prepared mixtures were stored in moisture-lock containers prior to their testing.

Determining biopolymer dosage range for investigation

The biopolymer dosage range for investigation was fixed based on the trial-and-error approach. Trial studies on chitin- and chitosan-treated fine soil started from an initial 0.25% dosage, and investigating 0.25% increments up to 1%. However, the LL and PL results did not show any credible variations over this range (0.25–1%). Hence, the dosage range was adjusted to start from an initial 0.5% dosage, investigating 0.5% increments up to 2.5%, and a maximum 4% dosage. Beyond 4%, the chitin and chitosan additives started to dominate the clay fraction during the PL tests owing to their lightweight nature.

Initial studies of NaCMC-treated soil investigated dosage increments of 0.25%. It was quickly found that compared to chitin and chitosan, the treated soil absorbed more water, with enormous amounts of water absorbed for > 1% NaCMC, causing problems in performing the consistency limits testing. For instance, soil test-specimen with > 1% NaCMC did not adhere to the smooth metal surface of the Casagrande percussion-cup during the LL testing. In other words, under repeated impacts of the falling cup onto the apparatus base, the test-specimen slided over the cup surface, rather than a shear/slope failure of the soil itself occurring, whereas for PL testing with > 1% NaCMC, water oozed out of the soil cylinders (threads) as they were manually rolled out on a glass plate to reduce their diameters from initially approx. 6-mm down to approx. 3-mm. Based on these findings, the investigated NaCMC dosage was fixed in the range of 0.25–1%.

Experimental methods

The LL and PL testing was performed in accordance with ASTM D4318 (ASTM 2017b), employing the Casagrande-cup and thread-rolling methods, respectively. Tests were repeated for five trials to ensure uniformity in the obtained values. X-ray diffraction (XRD) analysis for a 2θ range of 20°–80° was performed using a Bruker XRD instrument to investigate the possible formation of new chemical compounds with the additive treatment. Scanning electron microscopy (SEM) images were obtained using a TESCAN Vega-3 SEM instrument to investigate microstructural variations between the unamended and biopolymer-treated soil.

Results and discussion

Preliminary

This section explains the physical significance of the consistency limits parameters investigated for the unamended and biopolymer-treated soil. The LL is the threshold water content at which saturated remoulded fine-grained soil possesses a small (measurable) undrained shear strength s_u, behaving as a viscous liquid (slurry) for higher water contents. In other words, the LL is an indication of the soil's water-holding capacity. In the present investigation, the LL was determined using the Casagrande-cup method, with the associated LL water content corresponding to a small specific strength, defined as the ratio of s_u to bulk density of the saturated test-specimen (Haigh 2012). According to O'Kelly (2019), the overwhelming body of experimental evidence indicates that the value of s_u at the Casagrande-LL water content can range between 1 and 3 kPa, and typically decreases from approx. 2.5–1.6 kPa for low to very high plasticity soils (i.e., LL increasing from 20 to 70%). Below the PL water content, the soil exhibits brittle mechanical behaviour. The plastic range (i.e., between the PL and LL) identifies the water content range for which the soil exhibits plastic mechanical behaviour, deforming without rupture upon (re)moulding. More detailed information on the above points can be found in the papers by O'Kelly et al. (2018) and O'Kelly (2021).

The unamended test soil had an LL of 48.8%, a PL of 26.3%, and a plasticity index PI (= LL – PL) of 22.5%. The rate of reduction in the s_u value with increasing water content is termed as the flow index (FI). The FI can be computed from the gradient of the Casagrande-LL flow curve of water content versus number of blows (N) plotted in a semi-log chart. In other words, higher FI indicates the s_u value reduces at a faster rate with increasing water content.

Effects of biopolymer additives on the consistency limits

Figure 2 shows the effect of increased biopolymer dosage on the LL, PL and PI. From Fig. 2a, LL steadily increases by different amounts for the three additives. Compared to the ~ 8–10 percentage points increase for 4% chitin or chitosan dosage, a 50 percentage points increase in LL occurred for 1% NaCMC dosage, with only 0.25% NaCMC needed to increase the LL (from initially 48.8%) to 68.0%.

From Fig. 2b, the overall trend shows relatively modest PL increases for increasing biopolymer addition. Similar to LL, chitin and chitosan both produced relatively marginal changes in PL (from initially 26.3%) to ~ 30% for 4% dosage, compared to the slightly bigger PL increase to 32.7% for 1% NaCMC dosage. Note that the 0.5, 1.0 and 1.5% chitosan dosages produced small reductions in the PL relative to the unamended soil.

Since the margin of LL increase was greater than that of PL, the PI also increased (from initially 22.5%) for increasing biopolymer dosage (Fig. 2c). Because of substantial increases in LL and only minor variation in PL with increasing NaCMC, tremendous PI increases of 16.5 percentage points and up to 42 percentage points occurred for 0.25 and 1% NaCMC dosages, respectively, whereas more modest PI increases of 5–7 and ~ 5 percentage points occurred for up to 4% chitin and chitosan, respectively. It is noted that PI increased by ~ 5 percentage points for 0.5% chitin and 1.0% chitosan dosages, i.e., higher dosages of between 1 and 4% produced only marginal additional changes in PI.

Effect of biopolymer additives on soil classification

The unamended and biopolymer-treated soil were classified using the Casagrande-style PI–LL plasticity chart (Fig. 3).

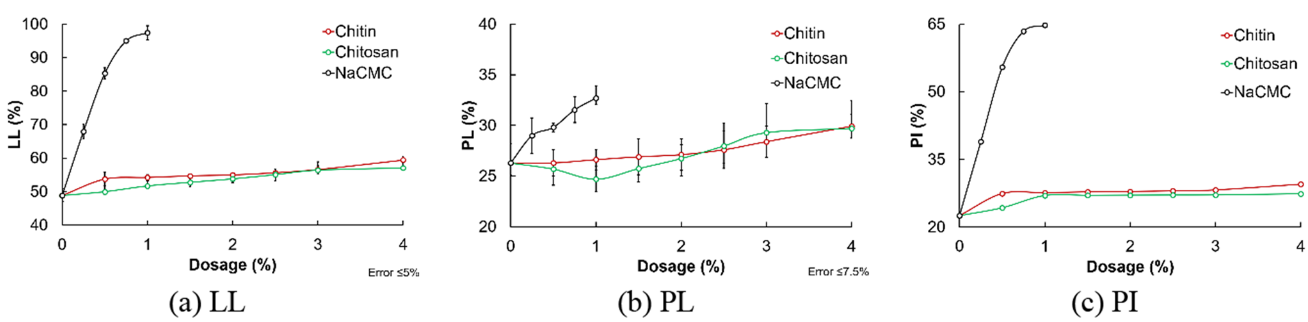


Fig. 2 Consistency limits of biopolymer-treated soil

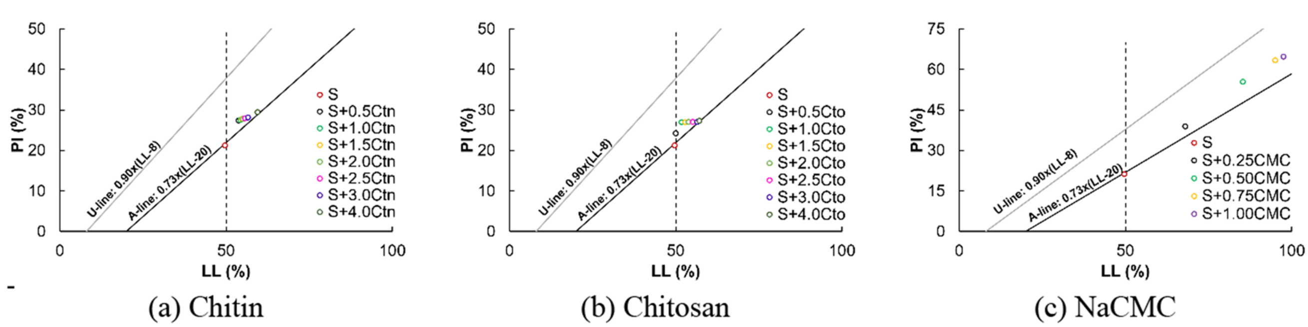


Fig. 3 Consistency limits of biopolymer-treated soil

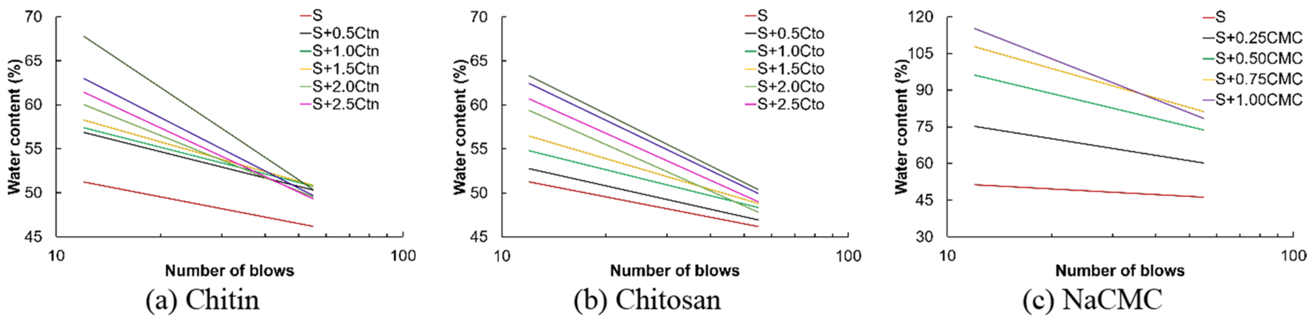


Fig. 4 Flow curves for biopolymer-treated soil

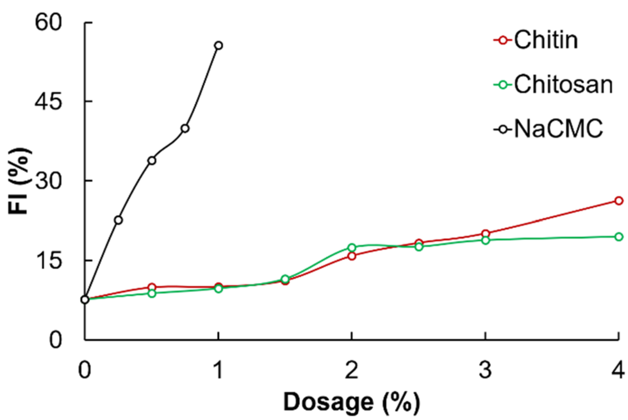


Fig. 5 Flow index of biopolymer-treated soil

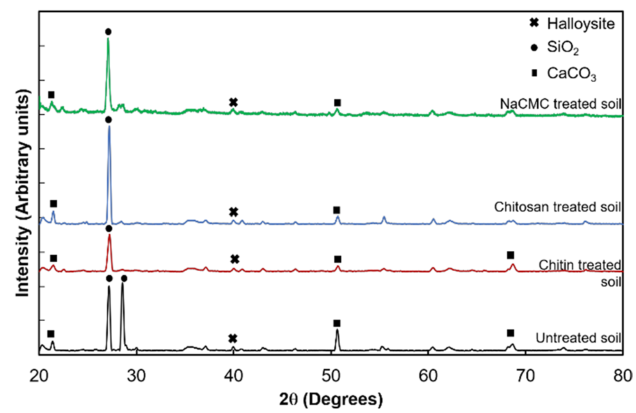


Fig. 6 XRD spectrum of untreated and biopolymer-treated soil

The A-line and U-line boundaries in the chart are designated as per ASTM D2487 (ASTM 2017a). Unamended soil plotted on the A-Line, which is the delineating boundary between *clay* and *silt* (plotting above and below, respectively). Whereas all biopolymer-treated soil plotted above the A-Line (i.e., as *clay*), but well below the upper limit of PI given by the U-Line boundary. Note the mere addition of small quantities of each biopolymer changed the soil plasticity class from one of intermediate plasticity (i.e., LL = 35–50%) for untreated soil, to high plasticity for chitin- and chitosan-treated soil, and very high to extremely high plasticity for NaCMC-treated soil.

Effect of biopolymer addition on strength reduction with increasing water content

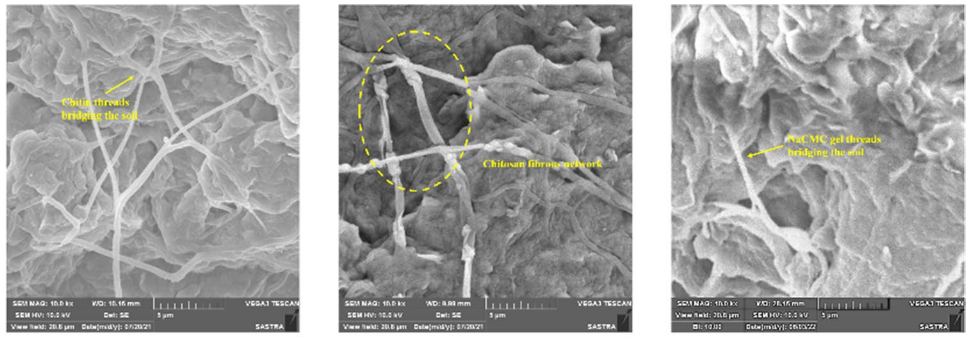
Figure 4 shows the Casagrande-LL flow curves for the unamended and biopolymer-treated soil, while Fig. 5 presents the variation of the computed FI with biopolymer dosage. From Fig. 5, FI increases (from initially 7.7%) with increasing biopolymer dosage. Compared to approx. similar behaviour/results for chitin and chitosan (with 4% dosages giving FI of 26.3% and 19.6%, respectively), the NaCMC-treated soil produced substantially greater FI of 55.6% for 1% dosage.

Chemical and microstructural analyses

Figure 6 presents the XRD results for the unamended and biopolymer-treated soil, with similar peaks in the XRD spectra implying that the biopolymer additives did not result in the formation of any new chemical compounds in the amended soil. This finding rules out the possibility of chemical reactions that could have binded the individual soil particles together.

The SEM images presented in Fig. 7 indicate that all three biopolymers resulted in forming 'bridge' connections between the soil particles, thereby improving the strength properties. Chitin and chitosan, being non-viscous in nature, tended to form a fibrous network (see Fig. 7a, b), typical of certain biopolymers. For instance, Kumar and Sujatha (2021) reported similar formation that helped in

Fig. 7 SEM images of biopolymer-treated soil (obtained at magnification of 10 k)



(a) Chitin (b) Chitosan (c) NaCMC

the improvement of β-glucan (non-gellable, non-viscous) biopolymer-treated soil. Whereas being viscous in nature, NaCMC evidently formed threads, as observed in Fig. 7c.

Discussion

It is clear from the presented results that NaCMC-treated soil behaved substantially different from chitin- and chitosan-treated soil. This was most apparent for the LL; i.e., 1% NaCMC dosage produced a 50 percentage points LL increase (from initially 48.8%) compared to ~ 8–10 percentage points increase for 4% chitin or chitosan dosages. Much smaller LL increases for chitin and chitosan could be explained as follows. Being non-ionic and having an average particle size similar to the soil particles, chitin acted like a filler material upon mixing with the test soil. The cationic chitosan interacts with the OH^- group of water, while the H^+ of water interacts with the negatively charged clay particles of the soil producing electrical neutrality (Hataf et al. 2018; Kannan and Sujatha 2023). However, addition of excess water over that required might disturb this electrical neutrality, and thereby cause the soil to lose shear strength. Whereas being a gellable biopolymer, addition of water molecules in NaCMC-treated soil increases its water imbibing capacity (without expelling it). Therefore, with increased NaCMC dosage, more water is needed for the soil to lose shear strength. For 1% NaCMC addition, the soil imbibed water was almost twice the measured LL of the unamended soil. This is the reason for using NaCMC as a water-retaining agent. The entire mechanism of the biopolymer additive reaction with water and soil is shown in Fig. 8.

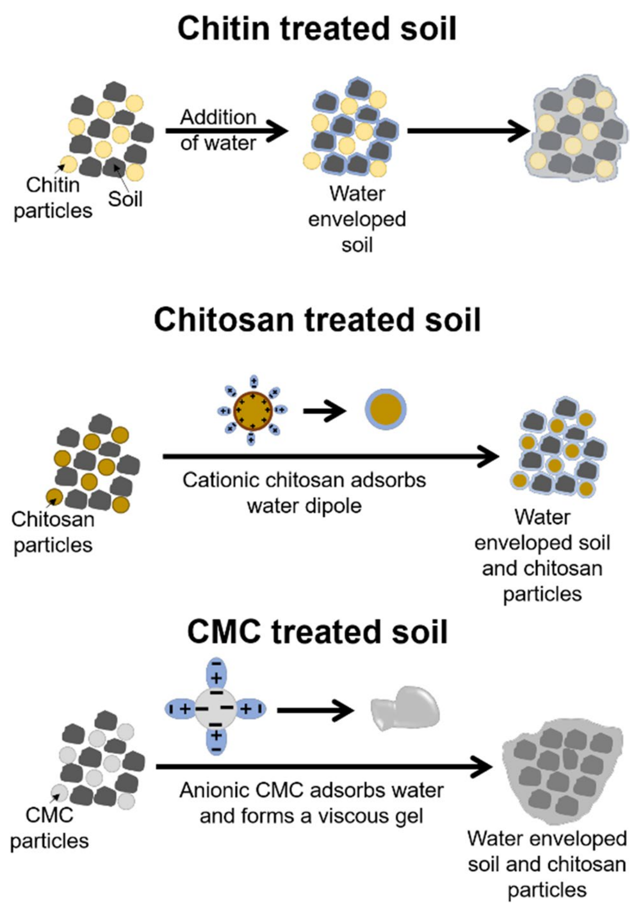


Fig. 8 Mechanism of water reaction with soil and biopolymer additive

Conclusion

This paper presented the results of consistency limits testing for an intermediate-plasticity organic silt amended with 0.5–4% chitin and chitosan, and 0.25–1% NaCMC dosages. An increase in dosage of all three biopolymers produced increases in the consistency limits, but more-so for LL than PL, thereby explaining the steady increase in PI for increasing dosage. The effects were more dramatic for addition of NaCMC (an anionic, hydrophilic, ether-derivative of cellulose) compared to the chitin and chitosan (non-ionic and cationic polysaccharides, respectively). The changes in consistency limits resulted in the soil classification changing, from '*silt* with intermediate plasticity' for the unamended soil, to '*clay* with high plasticity' for chitin and chitosan additions, and to '*clay* with very high to extremely high plasticity' for NaCMC addition. SEM examination confirmed that all three biopolymers formed 'bridge' connections between the soil particles, thereby improving the strength properties. This explained significant increases in LL (essentially a s_u-based parameter) for biopolymer addition, with greater LL increases produced for NaCMC addition, since it is a gellable biopolymer.

Acknowledgements The authors thank the Vice-Chancellor, SASTRA Deemed to be University, India, for providing the laboratory facilities to conduct the experiments.

Author contributions ERS planned the experiments and GK conducted the experiments. The results were critically interpreted by ERS. The first draft of the manuscript was prepared by GK, while ERS and BCOK improved it and approved the final draft. BCOK provided critical feedback and helped in improving the analysis and manuscript.

Declarations

Conflict of interest The authors declare that they have no conflict of interest.

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