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ORIGINAL ARTICLE

Xanthan gum biopolymer: an eco-friendly additive for stabilization of tropical organic peat

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Abstract Biogeotechnology is a recently established branch of geotechnical engineering, associated with the practical uses of microbiological techniques to improve the engineering properties of geomaterials. This study explores the utility of xanthan gum, an eco-friendly biopolymer obtained from microbial sources, for stabilization of tropical organic peat, using a series of macroscale and microscale test approaches. At the macroscale, the shear strength characteristics of both untreated and stabilized peat were evaluated using unconfined compression strength (UCS) and standard direct shear tests. Microscopic techniques, including field emission scanning electron microscopy (FESEM), Brunauer, Emmett, and Teller (N_2-BET) surface area analysis, and particle size analysis, were also utilized to examine changes in the microstructural characteristics of stabilized peat that are caused by the chemical reaction that occurs between the xanthan gum and peat particles. UCS test results showed that the xanthan gum stabilization significantly improved the shear strength of the peat in its natural condition, with the 28-day strength of the stabilized peat being six times higher than the strength of the untreated peat. Microstructural analysis showed that the morphological characteristics of the peat are changed due to the chemical reaction that occurs during the curing process, as indicated by the FESEM results. Over time, formation of cementitious products was clearly observed, which welded peat particles and filled the pores in the soil structure, yielding a denser soil fabric with less pore volume and stronger attractive forces. From the testing that was performed, xanthan gum stabilization is recommended for peat as an eco-friendly and sustainable alternative to traditional soil stabilization additives such as cement or lime.

Keywords Xanthan gum · Peat · Biopolymer · Soil stabilization · Shear strength · Field emission scanning electron microscopy (FESEM)

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Introduction

Construction site disposal of large volumes of surplus soils having unfavorable geotechnical properties is an important cost issue for earthwork companies (Blanck et al. 2014). Using traditional admixtures such as lime and cement to enhance the geotechnical properties of surplus soils, allowing for their use in some fashion on a given project site, is a preferable solution (Bozbey and Garaisayev 2010; Latifi et al. 2016b; Horpibulsuk et al. 2012; Kavak and Baykal 2012; Saadeldin and Siddiqua 2013; Kathirvel et al. 2013; Shen et al. 2013a, b; Gratchev et al. 2014; Wang et al. 2014; Ai-sharif and Attom 2014; Eisazadeh and Eisazadeh 2015; Met and Akgün 2015; Latifi et al. 2014, 2015a, b, d). Following sustainable development principles, it is desirable to minimize water consumption and the use of non-renewable resources; reducing or eliminating the production of harmful

chemical compounds or gases that contribute to global warming or ozone depletion is also beneficial. The use of traditional soil stabilizers such as lime or cement can change the pH of improved soil, with the potential to cause negative environmental impacts including retarded vegetation growth, reduction in groundwater quality, and even potentially human health problems (Chang et al. 2015). One of the most significant sources of CO_2 emissions in the world is cement production, with approximately one ton of CO_2 being produced during the production of one ton of cement; consequently, it is estimated that 5 % of the CO_2 that is produced in the world annually can be attributed to cement production (Chang et al. 2015). Particulate air emission in the form of cement dust is another environmental problem. Activities such as demolition and earthquakes may cause the cement dust that is present in concrete to escape into the atmosphere (Meo 2004). To address these issues, a number of researchers have more recently been focusing on the use of non-traditional soil improvement additives that may present a more sustainable solution for some applications (Sukmak et al. 2013, 2015; Kampala et al. 2013; Latifi et al. 2013; Marto et al. 2013, 2014; Phetchuay et al. 2014; Horpibulsuk et al. 2015; Suksiripattanapong et al. 2015a, b).

Biogeotechnology is a recently established branch of geotechnical engineering, associated with the practical uses of microbiological techniques to create additives that can improve the engineering properties of geomaterials. Biogeotechnologies generally have low up-front investment and maintenance costs, with greater benefits to the environment and aesthetics than their more traditional chemical stabilization counterparts (Ivanov and Chu 2008). As the scale of geotechnical construction is often large, particularly for land reclamation projects, adopting a biologically based soil stabilizing agent can in some cases yield significant cost savings, particularly if the stabilizing agent is ready available at a given location, or is the by-product of some other industrial process that must otherwise be dealt with (i.e., beneficial by-product reuse). It is well established that industrially produced water-insoluble gel-forming biopolymers of microbial origin such as xanthan gum, chitosan, polyglutamic acid, sodium alginate, and polyhydroxybutyrate can be used as additives for certain geotechnical applications such as soil erosion control, enclosing a bioremediation zone, and mitigating soil liquefaction (Ivanov and Chu 2008). More recently, Chang et al. (2015) have utilized xanthan gum to stabilize four inorganic soils. The results of their study, which utilized both unconfined compressive strength testing and scanning electron microscopy (SEM) tests, concluded that the xanthan gum was an effective and sustainable stabilizer that improved the compressive strength of the improved soils noticeably.

To date, the application of xanthan gum to stabilization of tropical organic peat has not been explored in detail; this is a potentially important application of this additive, as tropical organic peat generally has poor engineering characteristics. There are an estimated 30 million hectares of tropical land in the world, in which highly organic soil or peat is a common occurrence. Approximately 3 million hectares of this land are located in Malaysia (Moayedi et al. 2011), in which peat is commonplace; approximately 8 % of the total Malaysian land contains such soil (Edil 2001; Huat 2004; Moayedi et al. 2013). Peat is a natural soil formed mostly through combinations of bacteria, fungi, chemicals, and decomposed plants in an aerobic environment, which yields a matrix of soil solids that is highly organic in nature. Peat is particularly problematic from a geotechnical standpoint in that it tends to have a low shear strength and exhibits large settlements. Effective stabilization of tropical organic peats can be very difficult and is consequently an issue of interest to geotechnical researchers and engineers. The current study consequently uses a coupled macro- and microscale testing approach to study the use of xanthan gum for stabilization of tropical organic peat.

Microstructural analysis of stabilized soils is useful for understanding the underlying mechanisms of soil stabilization, as well as the chemical reactions that occur within the stabilized soil matrix. The previous research into the microstructure of chemically stabilized soils (e.g., Horpibulsuk et al. 2009, 2010; Chang et al. 2015; Suksiripattanapong et al. 2015a, b) has often used only a few microstructural tests to characterize a specific stabilized soil, with SEM testing being the most common approach. In the current study, a number of additional microstructural characterization tools were utilized, including: field emission scanning electron microscopy (FESEM), N_2-BET surface area analysis, and laser diffraction particle size analysis (PSA). The fairly common X-ray diffraction (XRD) test, which is commonly used to detect crystal mineral phases, was not carried out on stabilized soils as part of this study, as both xanthan gum and the cementitious products between xanthan gum and peat particles are amorphous materials (Bhattacharya et al. 2013; Pandey and Mishra 2011).

The effect of xanthan gum content and curing time on the stabilized soil shear strength was investigated using unconfined compression strength (UCS) tests and standard direct shear tests. In a concurrent fashion, microstructural characterization was performed using the aforementioned tests to analyze and understand the chemical reactions that occur between the xanthan gum and peat particles, as well as the underlying mechanisms of stabilization with respect to the soil microstructure and pore space.

Materials, sample preparation, and testing program

Materials

Tropical organic peat samples were obtained from various locations in Kampung Bahru, Pontian, West Johore, Malaysia, from a depth of about 1 m. The disturbed samples were stored in sealed containers in order to prevent changes in moisture content. Physicochemical characterization of the peat samples was performed in accordance with BS 1377-1 (1990), which includes moisture content, organic content, bulk density, fiber content, pH, and specific gravity. Table 1 shows the resulting physicochemical characteristics of the natural peat that was used in the current soil stabilization study. Figure 1 shows the corresponding EDAX spectra of the peat; from these results, it can be observed that the dominant elements present were Si, Al, Fe, K, and C. Figure 2 shows the XRD pattern of the natural peat, which indicates that the main minerals present were kaolinite ($2\theta = 12.5°$, 20°, 25°, 35°, 38°, 51°), quartz ($2\theta = 21°$, 27°, 37°, 39°, 50°), cristobalite ($2\theta = 21.5°$, 35.5°, 52°, 56°), and albite ($2\theta = 14°$, 26°, 28°, 30°) (JCPDS 1995).

Xanthan gum, a polysaccharide utilized mainly as a food additive and rheology modifier, is formed by carrying out the fermentation of glucose or sucrose using the Xanthomonas campestris bacterium (Davidson 1980; Rosalam and England 2006). The resulting anionic polysaccharide is composed of D-uronic acid, D-mannose, pyruvylated mannose, 6-O-acetyl D-mannose, and 1, 4-linked glucan (Cadmus et al. 1982). The simplified chemical structure of xanthan gum ($C_{35}H_{49}O_{29}$) is a linear linked b-D glucose backbone with a trisaccharide side chain attached to each glucose. The trisaccharide side chain and the backbone are in alignment, providing stability and conformity throughout the structure by the formation of hydrogen bonds.

Table 1 Physicochemical properties of natural peat

Physicochemical properties	Values
pH (L/S = 2.5)	5.30
Specific gravity	1.42
Bulk density (kN/m^3)	9.8
Organic content (%)	80
External surface area (m^2/g)	54
Fiber content (%)	60
Classification (von Post)	H_3
Void ratio	11
Natural moisture content (%)	150
Unconfined compressive strength (kPa)	13
SiO_2 (%)	25
Al_2O_3 (%)	21
CO_2 (%)	48
Fe_2O_3 (%)	3.5
K_2O (%)	2.5

A defining characteristic of xanthan gum is pseudoplasticity, where the viscosity degradation is dependent on the shear rate (Casas et al. 2000). In a static condition, a small amount of xanthan gum (in most foods, 0.5 %) induces a large increase in the viscosity of a liquid. Furthermore, xanthan gum, unlike other gums, displays higher stability over a broad range of temperatures and pH (Zohuriaan and Shokrolahi 2004). Its anionic and hydrophilic surface can react with cations and various polysaccharides, including glucose, mannose ($C_6H_{12}O_6$), potassium gluconate ($C_6H_{11}KO_7$), acetate (CH_3CO_2), and pyruvate ($CH_3COCOOH$), which can result in stronger gelation that what is observed with other gums (Laneuville et al. 2006; Bergmann et al. 2008). In practice, xanthan gum has long been utilized as a drilling mud thickener in the oil industry to provide consistent rheology throughout the drilling hole. It has also been used as an additive in concrete for enhancing viscosity and stopping washouts (Plank 2004; Comba and Sethi 2009). The molecular structure of xanthan gum is shown in Fig. 3 (Garcıa-Ochoa et al. 2000).

Sample preparation and testing program

The mixing procedure and curing system used in this study were adopted from the soft soil stabilization process outlined in the EuroSoilStab (2002) project. This project described various techniques for soft peat stabilization, the design methods that are commonly adopted given the context in which the research is implemented, the appropriate assessment methods to determine the most suitable binder, and eventually the pieces of equipment as well as the installation procedures which can be used for soil stabilization of peat on a given construction site. Following this guide, the air-dried peat was first passed through a 2-mm sieve, in order to remove coarse materials such as roots and large fibers, and then mixed with water to attain its natural water content. The xanthan gum was next mixed with the peat at 0.5, 1, 1.5, 2, and 2.5 % of the wet soil weight. In order to prepare a homogeneous mixture for creating uniform UCS test specimens, irregular hand mixing with palette knives was performed. The resulting peat–xanthan gum paste was transferred in batches to a steel cylindrical UCS mold and compressed by a hydraulic jack to form UCS test specimens, in accordance with 4.1.5 of BS 1924 (Part 2 1990b). Finally, the cylindrical specimens were extruded using a steel plunger, wrapped in several layers of cling film and cured for 3, 7, 28, and 90 days in a temperature-controlled room (20 °C) before performing UCS tests. To ensure the accuracy of the

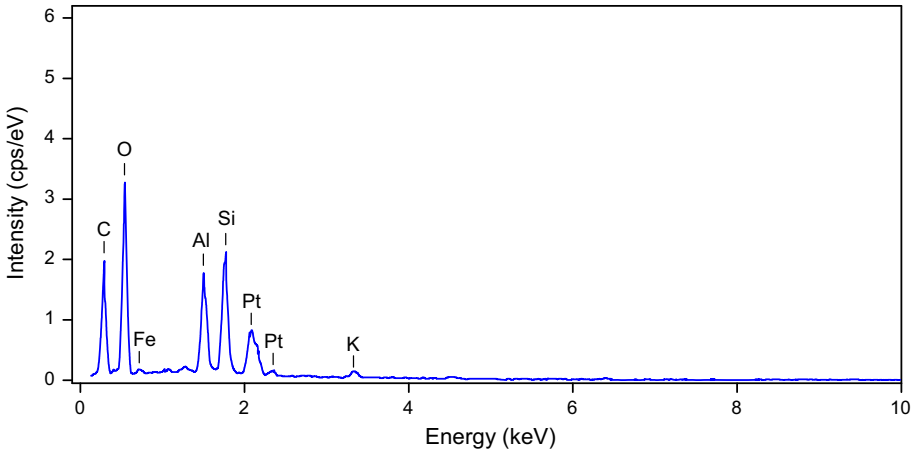

Fig. 1 EDAX spectrum of natural peat

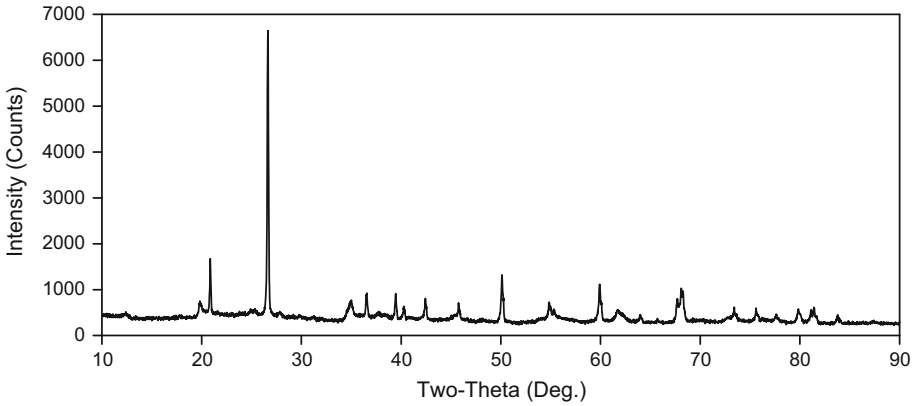

Fig. 2 XRD pattern of natural peat

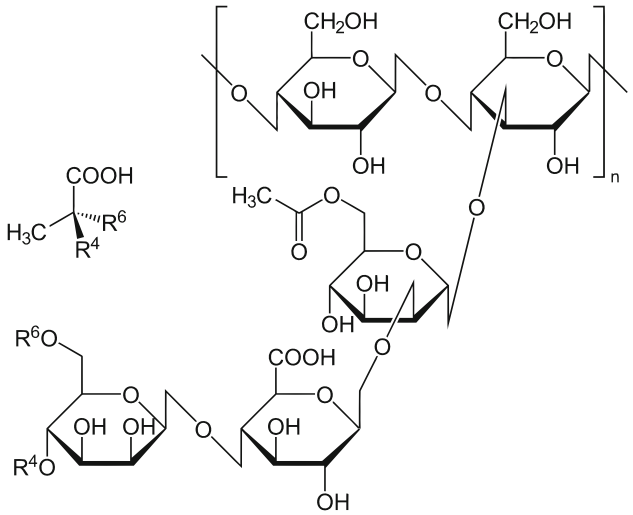

Fig. 3 Molecular structure of xanthan gum (Garcia-Ochoa et al. 2000)

results, four specimens for each soil mix design were prepared for each of the four curing periods that were tested.

The soil improvement index was determined by conducting a series of UCS tests (BS 1924: Part 2: 1990) on specimens at different curing time intervals. For the UCS tests, a constant strain rate of 1.25 mm/min was applied to all specimens. A data acquisition unit (DAQ) was used to record the applied load and axial deformation automatically. The failure of each specimen was defined by its peak axial stress. After testing, the failed specimens were dried and weighed to determine their moisture content.

Direct shear tests were performed on untreated specimens and stabilized specimens prepared at the optimum additive amount, based on the results from the UCS tests, to determine the shear strength properties. Direct shear tests were conducted in accordance with standard procedures (BS 1377: Part 7 1990) and ASTM D3080 (cited in Head 1990b). Direct shear specimens were prepared by compacting untreated or stabilized soil mixtures in a steel box with a 60 mm length × 60 mm width × 20 mm height, to achieve the target density at the optimum moisture content (which was previously obtained from compaction testing). The specimens were cured for 3, 7, 28, and 90 days, in a 20 °C temperature-controlled room.

Direct shear testing of specimens was performed by applying a constant strain rate of 0.6 mm/min to the specimens inside the shear boxes, until the soil failed or reached a maximum horizontal displacement of 10 mm. This total shear displacement was chosen based on the capability of the machine used. Tests were performed at three different normal stresses: 28, 56, and 112 kPa. Tests conducted on untreated and stabilized specimens under different normal stresses were used to plot relationships between failure normal stresses and shear stresses and to determine the associated Mohr–Coulomb shear strength parameters.

Scanning electron microscopy (SEM) and field emission scanning electron microscopy (FESEM) are both common methods for the analysis of the microstructural characteristics of soil fabric, providing data regarding the shape, size, and state of orientation and aggregation of soil particles. Both methods can also be implemented in soil stabilization studies, to determine the topographical features of stabilized soils and observe the formation of cementitious products. In the current study, FESEM was used as it provides clearer and higher-resolution micrographs than SEM. Specimen preparation for FESEM testing involves sputtering each sample with platinum for 120 s at 30 mA under a high vacuum, until the sample is covered completely; the specimen is then subjected to microscopic analysis.

For a given soil matrix, characterizing the particle surface area provides useful information for understanding both the physical and chemical changes that take place during the stabilization process, because chemical reactions occur on the surface of the soil particles. In this study, the nitrogen-based Brunauer–Emmett–Teller (N_2-BET) surface area method was used to determine the changes that occurred on the surface area and in the micropores of the stabilized specimens. The surface area was determined by assessing the physical adsorption of nitrogen gas by means of a micromeritics surface area analyzer. This device is microprocessor-controlled and interacts with a personal computer, which allows for a physisorption investigation. To run each test, a small sample was taken from a cured and dried UCS specimen and placed in the sample container, the sample was degassed for 1 h at 130 °C, nitrogen gas was then pumped into the sample, and the outer area value was estimated using the single-point BET technique (Quantachrome Corporation 2007).

Particle size analysis of untreated and stabilized specimens was performed using a CILAS particle size analyzer. The CILAS 1180 particle size analyzer is capable of measuring particle sizes ranging from 0.04 to 2500 μm, by utilizing a laser diffraction technique with a laser light wavelength λ of 635 nm. The Fraunhofer diffraction theory was used as a basis to determine the particle size distributions of the tested samples through the use of Particle Expert V5.12 software. All tests were performed with an approximate 0.2-g sample, and other test procedures followed were in accordance with BS ISO 13320:2009 (particle size analysis, laser diffraction methods).

All samples were identified using the following notation: P for peat, UNT for untreated/unstabilized soil, T for treated/stabilized soil, and D for days of curing period.

Results and discussion

Shear strength characteristics

Figure 4 shows UCS values of stabilized peat for various xanthan gum contents and curing times, relative to the UCS of untreated peat (13 kPa). As shown, the rate of UCS improvement was proportional to the xanthan gum content and curing time. The rate of strength development that occurred within the first 28 days was higher than the rate at longer curing times (i.e., from 28 to 90 days). The highest UCS values were observed at a xanthan gum stabilizer content of approximately 2.5 %. A xanthan gum content of

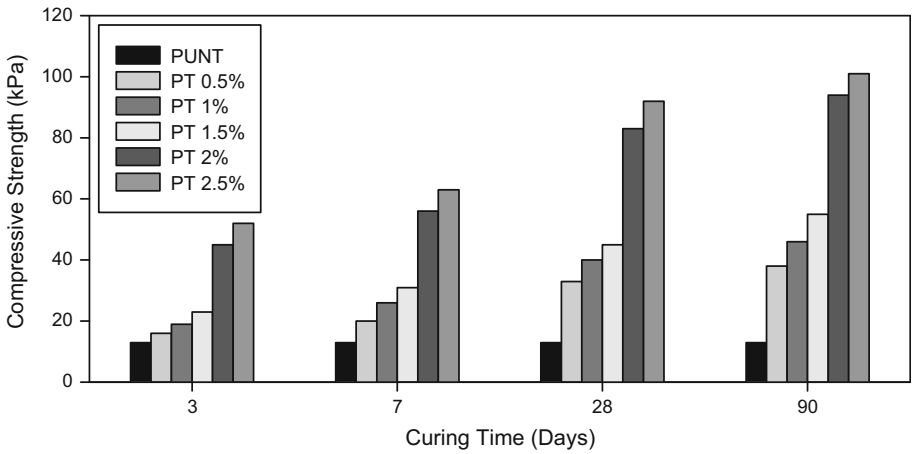

Fig. 4 Strength gained for xanthan gum-treated peat with different additive contents and curing times

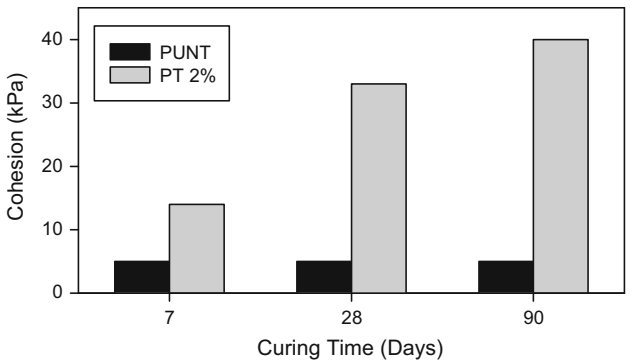

Fig. 5 Variation of cohesion of peat-treated samples at different time intervals

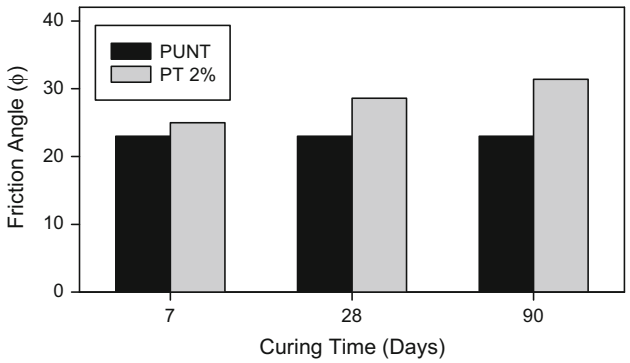

Fig. 6 Variation of friction angle of peat-treated samples at different time intervals

2 % was designated as the optimum stabilizer content, as increases in xanthan gum content beyond this point exhibited less significant strength gains. At a 2 % xanthan gum content, the 28-day UCS was 83 kPa, which is six times the unstabilized peat strength, a significant improvement.

Figures 5 and 6 show the average values of cohesion and internal friction angle of untreated and stabilized peat specimens at the optimum xanthan gum stabilization level (2 %), cured for 7, 28, and 90 days, respectively. As curing time increases, both the cohesion and friction angle of stabilized specimens increased significantly. The 28-day cohesion of stabilized peat (33 kPa) is approximately six times greater than the untreated soil cohesion (5 kPa). The additional increase in cohesion from 28 to 90 days of curing is less significant; i.e., the 90-day cohesion is just 40 kPa. Similarly, the internal friction angle varies from 23° to 29° and to 31° after 28 and 90 days of curing, respectively. It is implied from the UCS and direct shear test results that the most significant increases in shear strength occur in the first 28 days of curing.

It is preferable to explain the increase in shear strength that occurs over curing time (both UCS and direct shear) by understanding the changes that occur in the soil structure, which includes the fabric (arrangement of soil particles and pores) and interparticle forces (attractive/repulsive forces) (Mitchell 1993; Miura et al. 2001; Horpibulsuk et al. 2005). For xanthan gum stabilization of peat, strength development with time can be explained by the growth of cementitious products that weld the soil particles together and fill the pores in the xanthan gum–peat matrix. Due to the electrically charged nature of clay particles, direct interaction (e.g., hydrogen bonding) between kaolinite particles and xanthan gum occurs and increases the attractive forces. The UCS results of xanthan gum-stabilized peat in this study showed greater effectiveness when compared to previous results (Moayedi et al. 2013) where cement and lime were used to stabilize the same peat soil over the same curing time. From this observation, it can be concluded that xanthan gum stabilization of peat offers an environmentally friendly alternative to more traditional chemical additive stabilization. Changes in soil structure can be evaluated using FESEM testing to examine the morphology of cementation materials, N_2-BET surface area analysis to examine the porosity of treated particles, and particle size analysis to assess the resulting size of treated particles (i.e., formation of agglomerations).

Microstructural analysis

FESEM

FESEM results for untreated peat, xanthan gum, and stabilized peat after 7, 28, and 90 days of curing are shown in Fig. 7a–e, respectively. A dispersed, irregular, and discontinuous microstructure with noticeable voids and porosity was observed in the natural peat (Fig. 7a), while a regular and smooth surface having fiber matrices was observed in the xanthan gum (Fig. 7b). Elongated fibrous structures were common in the peat sample (Fig. 7c). This clearly detected fiber indicates that the chemical reaction was still developing, and hence, the fibers were not fully covered by the new cementitious products. In contrast, Fig. 7d, e (28 and 90 days, respectively) shows the formation of new gel-like white lumps indicating the chemical reaction between peat and xanthan gum, which were visible throughout the micrographs of the stabilized sample, particularly after 28 days of curing. After 90 days of curing, the peat soil particles and the fibers were coated strongly together by the new cementitious products, and the contact area among the soil particles increased noticeably. It should be noted that these new cementing products filled the porous areas within the soil particles almost completely, leading to a much more continuous soil structure with denser and stronger engineering properties.

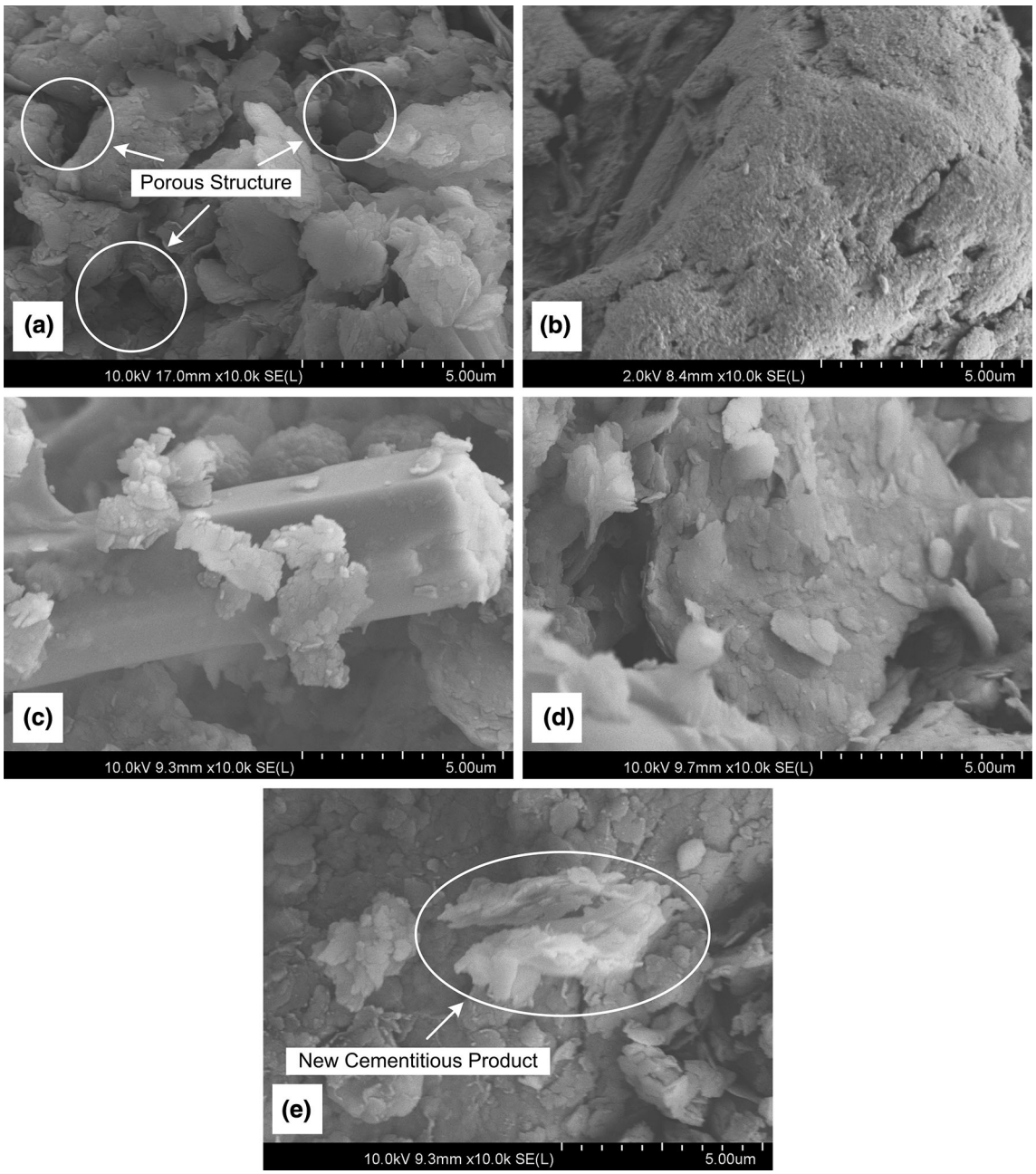

Fig. 7 FESEM results of **a** natural peat, **b** xanthan gum, **c** xanthan gum-treated peat after 7 days of curing, **d** xanthan gum-treated peat after 28 days of curing, and **e** xanthan gum-treated peat after 90 days of curing

N_2-BET surface area analysis

The specific surface area is an important characteristic to examine to assess changes in soil structure that occur from chemical stabilization (Latifi et al. 2015c). Figure 8 shows the N_2-BET results for natural (untreated) peat and stabilized peat at various time intervals. As shown, over time, a significant reduction in the surface area of the stabilized samples occurs. After 28 days of curing, the flocculation and growth of cementitious compounds among soil particles and in pores (as shown in the FESEM images) yield a reduction in pore volume over time, which corresponds to a smaller surface area (Latifi et al. 2016a; Eisazadeh and Eisazadeh 2015). Time-dependent changes in the surface area were fairly insignificant after 28 days of curing, as shown in Fig. 8. This indicates that the greatest part of the

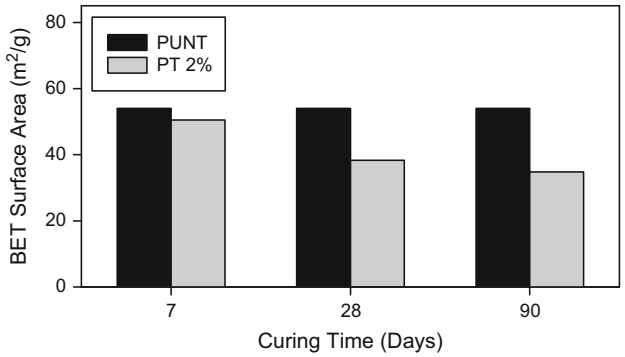

Fig. 8 N_2-BET results for untreated and xanthan gum-treated peat samples

peat–xanthan gum reactions happened within the first 28 days. Analysis of the surface area from N_2-BET tests confirmed that new cementitious products filled the porous structure and micropores of peat. Hence, it can be deduced that the new materials formed were the main reason for the improvement in soil strength.

Particle size analysis (PSA)

In addition to causing changes in surface area, the growth of cementing products causes the formation of particle agglomerations at the microlevel, which changes the behavior of the stabilized soil. Formation of these agglomerations can be observed by examining changes in the particle size distribution using a microlevel approach to PSA. In the current study, the particle size of untreated and stabilized peat samples was determined using the CILAS machine, Fraunhofer diffraction theory, and Particle Expert V5.12 software. The resulting PSA curves for untreated and stabilized samples containing 2 % xanthan gum are shown in Fig. 9. As shown, generally the particle size curve shifts to the right with longer curing times. Approximately 37 % of clay-sized (<0.002 mm) particles were observed for the untreated sample. The xanthan gum stabilization causes a reduction in the amount of clay-size particles; i.e., from 37 to 11 % after 28 days of curing, which was associated with an increase in silt-sized (0.075–0.002 mm) particles; i.e., from 63 to 89 % after 28 days of curing. The change in particle size is relatively low after 28 days of curing; i.e., from 28 to 90 days, the incremental change in the silt-sized particles was found to be only 4 %; this trend was similar to the reduction in surface area that occurred. This observation further supports the conclusion that most of the reaction between xanthan gum and peat took place in the first 28 days of curing. The results obtained from PSA were also consistent with the aggregation and cementation behavior observed in the various microstructure tests, particularly in the FESEM micrographs. These results imply that the cementitious products welded the peat particles upon interaction, leading to an increased particle size and shear strength enhancement over time.

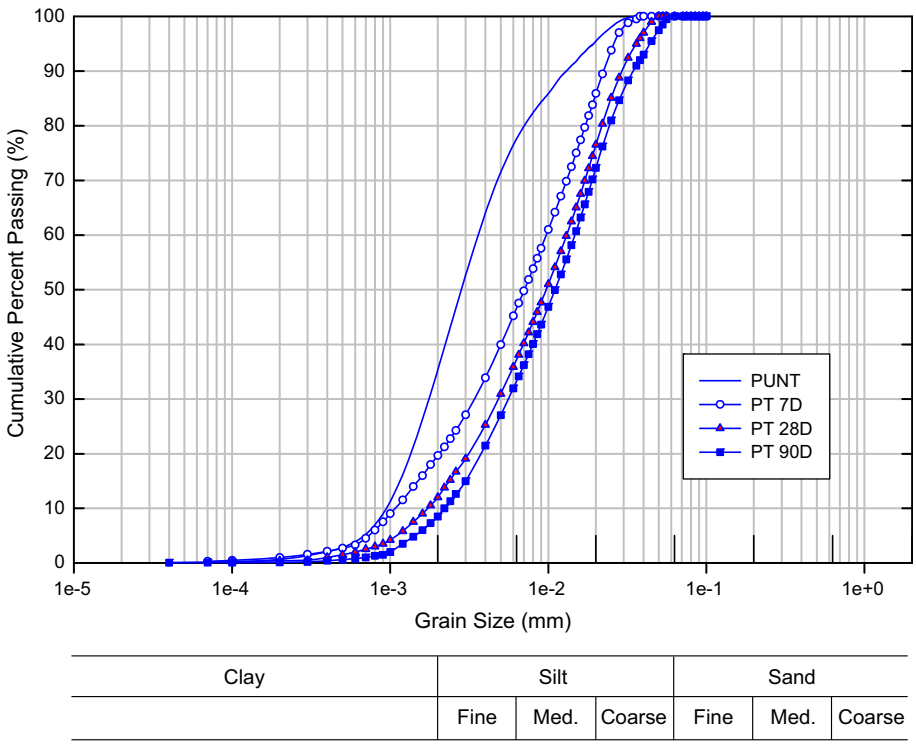

Fig. 9 Particle size analysis of untreated and xanthan gum-treated peat samples

Conclusions

Strength development in xanthan gum-stabilized peat occurs from development of a xanthan gum matrix (e.g., threads or textiles) having its own strength, hydrogen or electrostatic bonding between the xanthan gum and clay particles, and a change in the void-space characteristics of the stabilized peat matrix. These factors improve the overall soil fabric and enhance the attractive forces in the soil structure. Significant gains in shear strength were observed for stabilized peat, even with the addition of a small amount of xanthan gum. The optimum xanthan gum content was found to be 2 %. Beyond this content, shear strength improvement was observed, but in a decreasing amount as additional xanthan gum was added. Additionally, peat mixtures having xanthan gum contents greater than 2 % are generally not as economical and can have workability issues (e.g., high viscosity leading to poor mixing). For stabilized specimens, the most significant shear strength improvement occurs within the first 28 days of curing. Both UCS and shear strengths measured in the direct shear test increased with time, particularly in the first 28 days of curing. Gel-like cementitious products from xanthan gum–peat reactions were observed using FESEM imaging. Cementitious products that weld peat particles and fill pores in the soil structure were believed to be the main contributors to the strength development that was observed for stabilized specimens. The welding of peat particles and the filling of pores were also observed in the PSA results and the N_2-BET analysis results, respectively.

The xanthan gum-stabilized peat specimens examined in this study exhibited marked improvements in strength, even at relatively small additive amounts. Moreover, xanthan gum is considered to be environmentally friendly "all natural" emulsifier, which is even used as a food additive. Taken together, the macro- and microstructural analysis results from this research indicate that xanthan gum can be used as a sustainable additive for peat, making it a good alternative to more traditional additives such as cement and lime. These findings have significant implications for those that need to stabilize tropical organic peats and may also be useful for peat stabilization in other regions of the world as well; future research is warranted in this area.

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