

Recycling biopolymer stabilised earthen construction materials using β -mannanase

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ARTICLE INFO

Keywords:

Recycling
Earthen materials
 β -mannanase
Reuse
Xanthan
Guar

ABSTRACT

Biopolymers like guar and xanthan gums are considered to be sustainable soil stabilisers that can improve engineering properties of earthen construction materials. Recent studies indicate that water-based soil washing has achieved partial success in recycling these materials. In this study, β -mannanase was used in the washing process for recycling. An engineered soil mixture was stabilised using these biopolymers and then recycled in two stages, initially with water and then with β -mannanase. The impact of recycling was ascertained in terms of particle size distribution and Atterberg limits. The recycled soil was subsequently used to produce earthen construction materials, and their mechanical performance was evaluated. Two recycling cycles were conducted to explore the potential of recycling after repeated cycles of biopolymer amendment. Using β -mannanase proved effective in recovering more original material than washing with water and notably more effective for guar gum. Mechanical tests indicated that the recycled material was suitable for earthen construction.

1. Introduction

It is a common acceptance amongst earthen building practitioners and researchers that soil-based building materials like rammed earth and compressed earth blocks are sustainable due to their inherent attributes of low embodied energy and carbon emissions, along with a high potential for recyclability [1–3]. However, the sustainability of earthen construction materials remains valid as long as they are not amended by energy-intensive and carbon-emitting conventional chemical stabilisers like cement, which now is a widely used method in practice to improve the durability of these materials against water ingress. It is well reported in the literature that the carbon emissions of cement-stabilised earthen construction materials in practice are comparable to that of a fired brick or a low-grade concrete [4–6]. The addition of the cement adds further complication to the recycling process, as the cementitious gels formed during the stabilisation process hinder the separation of the different soil fractions originally used to manufacture the earthen construction material. As a result, recycling cement-stabilised earthen construction material becomes a tedious, energy-intensive and uneconomical process [2]. Recycling in actual sense means recovering the original raw components from the manufactured material in their initial forms and quantities, enabling their application for the same purpose again, while reuse implies using the

material for a similar application, employing little or no processing to alter its properties or separate the raw materials [1,7]. Given the significant challenges associated with the recycling process of the cement stabilised earthen construction material, many of the past studies interested in recycling earthen construction material have in actuality restrained themselves to ‘reuse’ rather than ‘recycling’ of the material [8–10].

Given the drastic influence of chemical stabilisation on the sustainability attributes of earthen construction materials, there has been a significant surge in research to explore alternative methods or processes that can provide comparable stabilisation outcomes as that of cement or lime. Out of the many potential alternatives, biopolymer stabilisation seems to be a possible frontrunner in addressing the sustainability issues raised by conventional chemical stabilisation without hindering any stabilising performance. Biopolymers, especially in the form of polysaccharides, have demonstrated similar stabilising ability to that of cement, in terms of strength and durability of the stabilised material. Although the use of polysaccharides for soil improvement is not a new concept, with ancient Chinese builders incorporating sticky rice starch in earthen construction [11], the application of industrial biopolymers to enhance the sustainability of earthen materials is an emerging area of interest among researchers. Out of various biopolymers available in the industry, guar gum and xanthan gum have been widely utilised as

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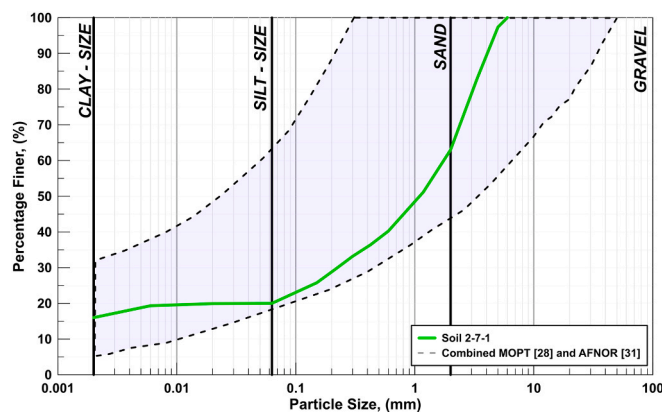
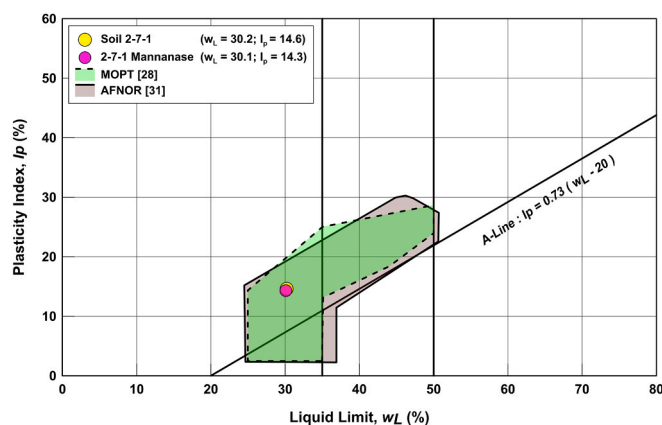
Table 1

Physical and compaction properties of the un stabilised soil mixture used in this study as per British Standard [32,33].

Soil	Clay (%)	Silt (%)	Sand (%)	Gravel (%)	Liquid Limit (%)	Plastic Limit (%)	OWC (%)	$\gamma_{d,max}$ (kN/m ³)
2-7-1	16	04	70	10	30.2	15.6	9.8	19.62

potential stabilisers for manufacturing earthen construction material. Guar gum is derived from the seed of a bean plant, *Cyamopsis tetragonoloba* which belongs to the Leguminosae family. Chemically guar gum is a neutral polysaccharide and comprises of monosaccharides namely mannose and galactose [12,13]. On the other hand, xanthan gum is a *Xanthomonas campestris* bacteria-derived anionic polysaccharide which consists of glucose, mannose and glucuronic acid monosaccharide units [14,15]. Both biopolymers are hydrocolloids, which are polysaccharides that disperse or dissolve in water and increase the viscosity of the mixture. They both contain a large number of hydroxyl (OH) groups which attract water molecules due to the polar nature of oxygen, present in both the hydroxyl groups and the water molecules [16]. Xanthan and guar gums are therefore hydrophilic and their strong affinity to water means they form a viscous dispersion in the medium in which they are placed; they become a coagulant and create a thickening effect [12,14]. These biopolymers form 'hydrogels' when combined with water, a property that enables them to stabilise various materials. Due to this versatility, they are widely available and used across multiple industries, including food, pharmaceuticals, cosmetics, textiles, and oil well drilling [12,15]. When mixed in soil along with water, the hydrogels formed by the biopolymer interlink different soil particles leading to soil agglomerations [17,18]. Hydrogels formed by guar and xanthan gums have versatility in stabilising different soil types and the ability to remain stable against a wide range of pH and temperature variations [12,15]. Several researchers have also reported that the soil stabilised with these biopolymers exhibit comparable mechanical performance to that of cement-stabilised soils in terms of their compressive and tensile strengths [17,19–22]. In relation to earthen construction materials, Muguda et al. [18] noted that the addition of biopolymer of about 1.5–2.0 % to dry soil mass achieved comparable or greater compressive strength than that of the material amended with cement at 10 % by soil mass. Further, the performance of guar and xanthan stabilised earthen construction material was satisfactory against standard durability tests against water ingress [23]. Notably, the tensile strength of both guar and xanthan gum stabilised material generally increases the tensile strength of the soil, while the strength gain achieved through xanthan gum is higher than that of cement-stabilised soil [18,20]. Considering, that the tensile strength is one of the key indicators of durability of the earthen construction material, improvement in the tensile strength of earthen construction material is an important outcome of the biopolymer stabilisation. The above list of positive attributes of both guar and xanthan gums highlights their potential to be sustainable soil stabilisers especially aimed at manufacturing earthen construction materials.

Apart from imparting required stabilisation, the nature of the stabilisation from guar and xanthan gums potentially provides an opportunity to fully recycle the stabilised earthen construction material. Muguda et al. [24] performed a preliminary study to understand the impact of soil washing on recycling guar and xanthan-stabilised earthen construction material. Though this was a preliminary study, it revealed that the hydrogels formed by biopolymers could be broken with a lesser energy-intensive process and potentially retrieve the original soil components used for the preparing the earthen construction material. Bruno et al. [25] performed an interesting study in the re-use of the guar gum stabilised earthen bricks. The study showed that the earthen brick stabilised with 4 % guar gum retained its ability to regain desirable compressive strength after being crushed and remanufactured as a brick even after three cycles of recycling. This study highlights the regenerative ability of guar gum to re-interact with soil particles after undergoing the process of re-manufacturing. Very recently, Soldo and Miletic

**Fig. 1.** Particle size distribution curve for the unamended soil mixture used in this study.**Fig. 2.** Plasticity properties of the unamended soil mixture used in this study.

[26] noted the regenerative ability of xanthan-stabilised soil i.e. the ability to re-interact with soil particles even after undergoing wetting and drying cycles and imparting strength and mending cracks formed on drying. The few above studies highlight the preliminary understanding of the recycling and regenerative abilities of both guar and xanthan gums which becomes the motivation for this study. Given the limitations of cement in recycling and the promising potential of biopolymers to be both recyclable and regenerative, this study focuses on recycling biopolymer-stabilised earthen construction materials using a bio-additive. The recycling process is evaluated by comparing the particle size distribution and soil plasticity of the recycled material with the original soil mixture. Additionally, considering the aimed application of the soil used is for manufacturing earthen construction materials, the mechanical performance, specifically unconfined compressive and tensile strengths, is assessed for samples prepared with the recycled material.

2. Materials and methodology

2.1. Materials

For this experimental programme, an engineered soil mixture denoted as Soil 2-7-1 consisting of 20 % kaolin, 70 % sand and 10 % gravel by mass was used. This soil mixture conforms with the recommendations given for earthen construction materials as suggested in the earlier publications [27–31]. This engineered soil mixture has also been extensively used in previous studies for characterising biopolymer-stabilised earthen construction material for their mechanical, hygroscopic and durability properties [18,23]. The physical properties and the compaction characteristics of the unamended soil mixture determined as per British standards [32,33] are presented in Table 1. The particle size distribution (PSD) and plasticity properties of the unamended soil mixture along with the recommended MOPT [28] and AFNOR [31] guidelines are presented in Figs. 1 and 2 respectively. The biopolymers (guar and xanthan gums) used in the study were procured from M/s Merck Life Science, United Kingdom. To keep the stabilisation process consistent with the previous research conducted [18,23,34], the biopolymer content in this study was maintained at 2.0 % of the dry soil mass.

In this study, a bio-additive namely β -mannanase was considered for recycling biopolymer-stabilised earthen construction material. β -mannanase can break down mannose monosaccharides, which are typically found in guar and xanthan gums, into smaller carbohydrate molecules that be easily washed away with water [35]. β -mannanase is a biologically processed enzyme using Bacillus bacteria species, and fungi from the genus Aspergillus [36]. Further, β -mannanase is reported to be stable over a wide range of pH (5.5–9.5) and temperatures (10 °C - 70 °C) [37, 38]. Considering these promising properties of β -mannanase, it was considered a suitable soil washing agent for recycling biopolymer-stabilised earthen materials in this study. The recycling agent, β -mannanase, in solution form of Mannanase 50K was procured from M/S Enzyme Supplies Limited, United Kingdom. A quick assessment was undertaken to understand the interaction of β -mannanase with the chosen soil mixture by conducting plasticity tests for unamended soil mixture washed with only β -mannanase and results are presented in Fig. 2. The plasticity properties remain similar to that of the original unamended soil mixture indicating relatively less or no interaction of β -mannanase solution with the soil particles.

2.2. Methodology

In this study, two cycles of recycling were undertaken for biopolymer-stabilised earthen construction materials and the recycling capabilities of β -mannanase were understood by comparing the changes in the particle size distribution and plasticity properties of the unamended soil mixture with the recycled material. Further, the impact of recycling on re-manufactured biopolymer stabilised earthen construction material between recycling cycles was evaluated by analysing the mechanical performance in terms of compressive and tensile strengths.

2.2.1. Sample preparation and testing

The initial step in this study involved preparing samples for unconfined compressive (UC) and tensile testing. For unconfined compression testing, cylindrical specimens (38 mm diameter \times 76 mm height) were prepared, while 'bowtie' specimens as specified by Stirling et al. [39] were prepared to determine tensile strength. For the sample preparation, initially, all ingredients (the engineered soil mixture and the chosen biopolymer) were dry mixed thoroughly. The biopolymer content in this study was maintained at 2.0 % of the dry soil mass to maintain consistency with the previous studies conducted by the authors. Following the dry mixing, water equivalent to the optimum water content of the unamended soil mixture and an additional 2 % water for guar gum samples and 1 % water for xanthan gum samples was added to the dry

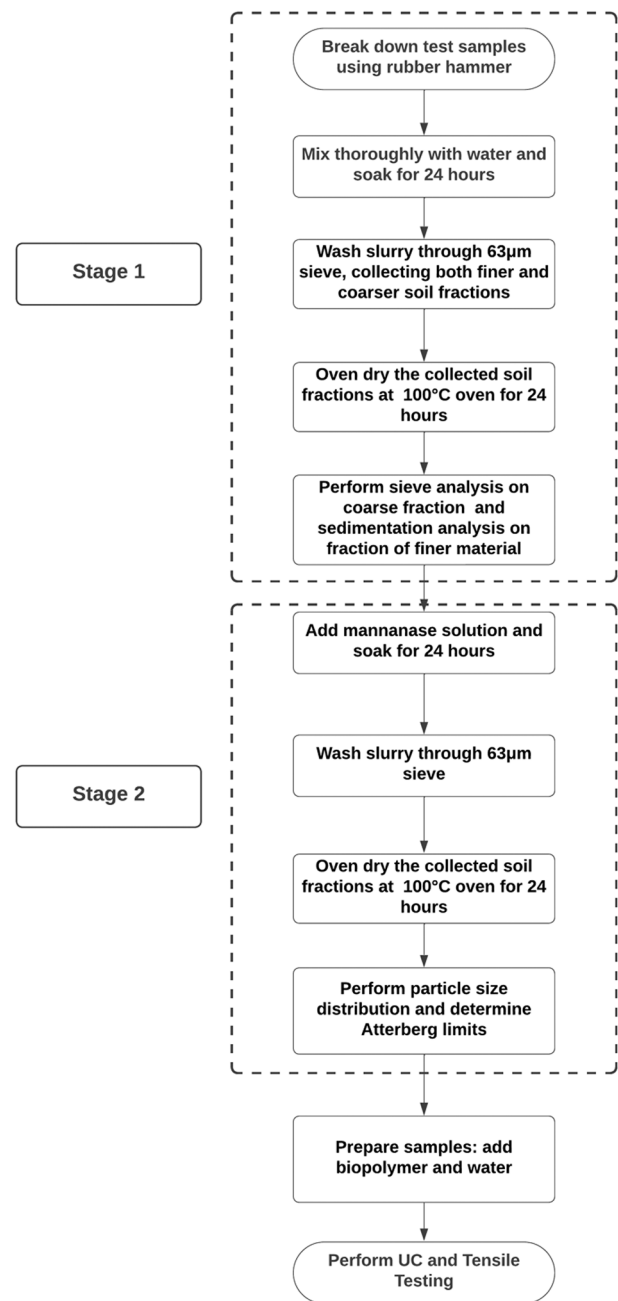


Fig. 3. Schematic diagram of the recycling procedure undertaken.

ingredients [18]. This additional water was necessary to hydrate the biopolymer, and the different quantities are due to the varying chemical and hydrophilic properties of guar and xanthan gums [40]. The wet mixture was then thoroughly mixed until a homogeneous wet mix was obtained. After this mixing, the required bulk mixture was placed into respective sample moulds and statically compressed in three layers to achieve the maximum dry density of the unamended soil mix (i.e. 19.62 kN/m³). The compacted test specimen was then extruded from the sample mould and left to air-dry on a laboratory bench at a temperature of 21 °C and a relative humidity of 50 % to gain strength. UC and tensile testing were conducted on two different curing periods i.e., 7 and 28 days. For each curing period, three identical samples were prepared for testing. On the day of testing, UC and tensile test specimens were tested as per the procedures outlined in BS 1377-7 [41] and Stirling et al. [39] respectively. Same procedures were followed for preparing samples and undertaking mechanical tests after each cycle of recycling.

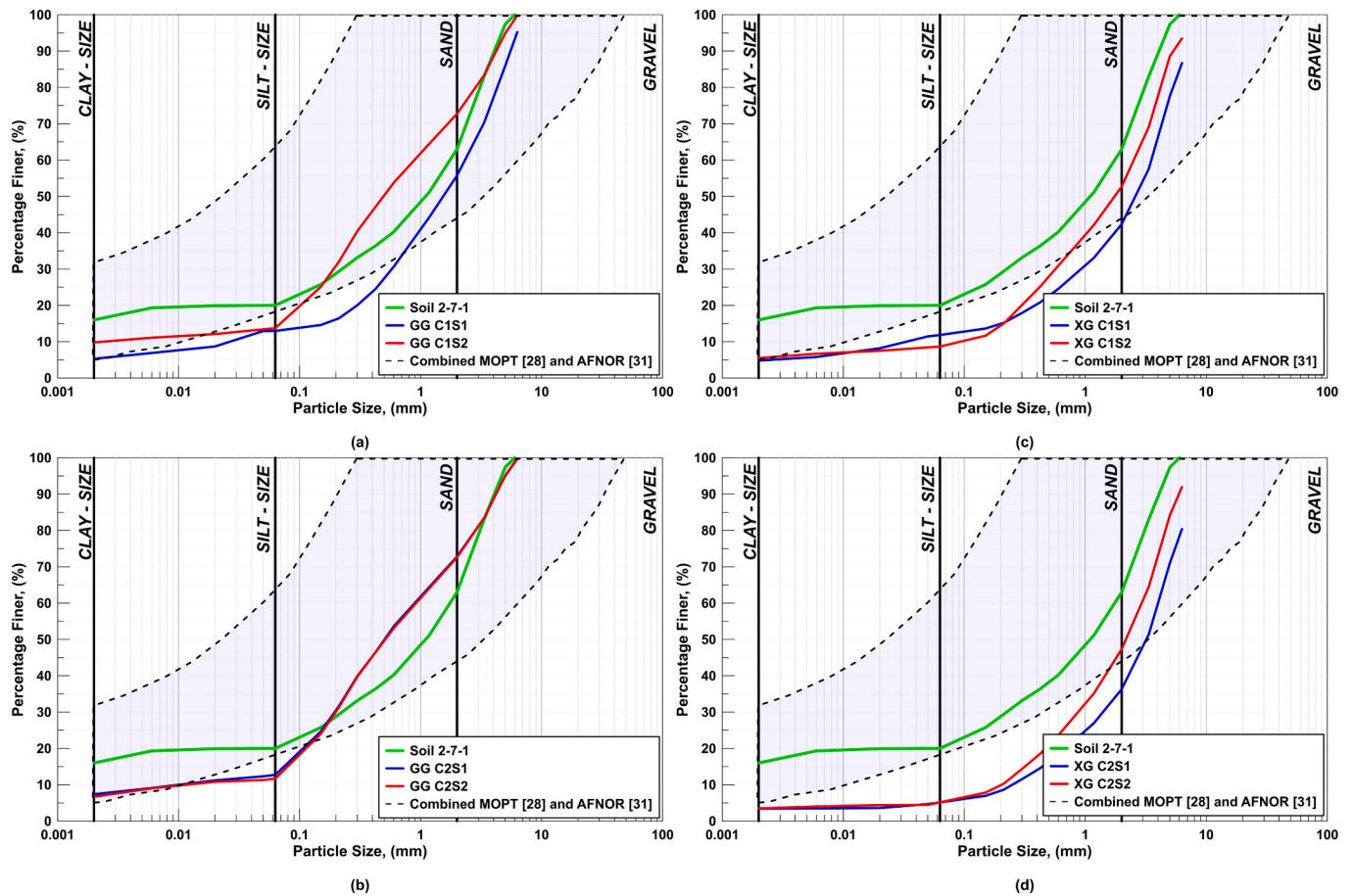


Fig. 4. Particle size distribution curves of the unamended and recycled soil samples: (a) guar gum cycle 1, (b) guar gum cycle 2, (c) xanthan gum cycle 1, (d) xanthan gum Cycle 2.

2.2.2. Recycling procedure

Fig. 3 presents a flowchart which outlines the recycling stages and testing protocols undertaken in this study. To initiate the experimental programme, biopolymer stabilised earthen samples were prepared to evaluate their unconfined compressive and tensile strengths cured for 7 and 28 days. After the set curing time, the samples were tested for their mechanical strengths and the crushed test specimens were used for recycling. To understand the differences between conventional soil washing with water and washing with β -mannanase, each recycling cycle was split up into two stages of washing, first with water and later with β -mannanase. In the first stage, the crushed test specimens obtained from mechanical testing were broken down gently using a rubber hammer until all the material passed through the 6.3 mm sieve. To commence Stage 1 of the recycling process, the crushed material was left to soak in distilled water for at least 24 h. A major portion of the soaked material was then manually washed with water through a 63 μm sieve until the water ran clear, to separate the fine fraction from the coarse material. The suspension of fine material in water collected from the washing was oven-dried at $100 \pm 5^\circ\text{C}$ until all the water had evaporated off, and sedimentation analysis using the pipette method in accordance with BS1377-2 [32] was performed on this material. The coarser fraction retained on the 63 μm sieve was also dried at $100 \pm 5^\circ\text{C}$ for 24 hours, and then dry sieve analysis was undertaken in accordance with BS1377-2 [32]. The results of the dry sieve and sedimentation analyses were combined to obtain the particle size distribution of the water-washed material.

In Stage 2 of the recycling cycle, a bulk solution of the chosen bio-additive β -mannanase was prepared in advance to ensure it remained consistent throughout the study. By the recommended dosages for

washing purposes from the supplier, the solution prepared for this study was 1% w/w in distilled water [42]. To commence Stage 2 of the recycling process, the material from Stage 1 was mixed thoroughly with β -mannanase solution and allowed to soak for at least 24 h. Soaking of the material was undertaken to provide sufficient time for the additive to interact with hydrogels formed by the individual biopolymer. The ratio of the β -mannanase solution to soil material used was 1:1.5. After 24 h, the soaked material in the form of slurry was then washed through the 63 μm sieve, and the fractions of fine and coarser material were collected and completely dried in an oven at $100 \pm 5^\circ\text{C}$. Dry sieve and sedimentation analyses were performed for coarse and finer fractions respectively as per BS1377-2 [32] to obtain the particle size distribution for the Stage 2 material. A portion of the soaked material after 24 h, was also used to determine the Atterberg limits of the material. These two stages of washing the material using water and then with β -mannanase would constitute one full cycle of recycling. All the washed soil material obtained from particle size distribution tests was gathered again to prepare samples for mechanical testing and the next cycle of recycling. In this study, two full cycles of recycling were undertaken for comparison of properties after multiple recycling efforts.

3. Results and discussion

3.1. Particle size distribution

Fig. 4 presents the particle size distribution curves of the unamended soil mixture along with the recycled soil mixture for the two cycles of recycling for both guar and xanthan gum stabilised samples. Within the figure, 'C' and 'S' refer to the recycling cycle and stage, e.g. C1S2 – Cycle

1, Stage 2. Referring to guar gum stabilised samples in Fig. 4a, after the first stage of recycling, the amount of the clay particles accounts for about 5 %, whilst in the unamended soil mixture clay accounts for about 16 %. At the same time, the coarse fraction retained on the 63 μ m sieve after recycling increased to 87 % from the original 78 %. This reduction of fine fraction (clay and silt-sized particles) and increased coarse fraction after the first stage of recycling indicates the inability of water washing to effectively separate the soil agglomerations formed by the guar gum. After the second stage of washing with β -mannanase, more agglomerations seem to have been broken down leading to the recovery of the finer fraction of the soil mixture. This can be observed in Fig. 4a in which the C1S2 curve is closer to the Soil 2-7-1 curve compared to C1S1. Though slightly more material was able to be recovered using β -mannanase, there was about 5% to 7% 5–7 % of particles not recovered within the clay to silt-sized range, indicating the resistance of soil agglomerations formed by guar gum even after washing using β -mannanase. Fig. 4b shows the particle size distribution curves for Cycle 2 for guar gum-amended samples. It is interesting to note that, both stages of washing resulted in obtaining material with effectively similar particle distribution. Further, it is very similar to particle size distribution from Stage 2 of Cycle 1 (C1S2). This similarity in particle size distribution between recycling cycles indicates that the samples prepared after the first recycling cycle with added guar gum essentially could not establish a strong soil matrix, and hence, water alone was sufficient for easy recycling. This hypothesis goes well in conjunction with the mechanical testing results discussed in the following sections, where the compressive strength for guar gum stabilised samples was reduced with recycling cycles. Alternatively, the good recovery of soil material at Stage 1 may have occurred because some portions of additive was able to remain in the material from the C1S2 wash, which was then activated by the water during the C2S1 wash and able to aid the breakdown of biopolymer. After two cycles of the recycling, the resulting particle size distribution curve is different from the original soil gradation. At first glance, it might seem the original soil components may not have been recovered during recycling process. However, coarser portions of the soil mixture would have undergone physical changes during sample crushing and preparation processes. The coarser particles likely altered in size and shape due to compaction pressure during sample preparation. Additionally, this change could also result from crushing or shearing during mechanical testing, as well as the movement of material between containers and sieves in the multi-stage washing process. Although the original soil PSD was not obtained exactly, the soil constituents were almost fully recovered and therefore it would be possible for the earthen materials to be used again. Furthermore, the recycled materials remain mostly within the MOPT [28] and AFNOR [31] gradation guidelines for earthen construction materials as seen in Figs. 4a and 4b.

The xanthan gum stabilised samples exhibited a different response to the recycling process in comparison to that of guar gum stabilised samples. Fig. 4c shows the PSD curves from Cycle 1 of xanthan gum recycling washes. After the first stage of the first cycle of recycling, the curve plots below the particle size of the unamended soil mixture (Soil 2-7-1) at 6.3 mm, meaning that there was material with particle sizes of greater than 6.3 mm in the xanthan gum stabilised sample. This indicates that larger and more stable soil agglomerations would be formed due to xanthan gum. As an anionic polysaccharide, xanthan gum would essentially form stronger ionic bonds with the fine fraction of soil mixture, especially with clay particles. These stronger bonds essentially make the soil agglomerations resilient to immediate disintegration against water washing or immersion [23,43]. After the second stage of washing with β -mannanase, the upper portion of the particle size distribution (for coarser soil fractions) is in between the unamended soil mixture and the Stage 1 curve. This indicates that the mannanase solution at the concentration used for recycling has been effective to an extent in recovering soil particles for xanthan gum stabilised soils that could not be recovered through water washing alone. Considering that the soil agglomerations formed by xanthan gum are through a complex

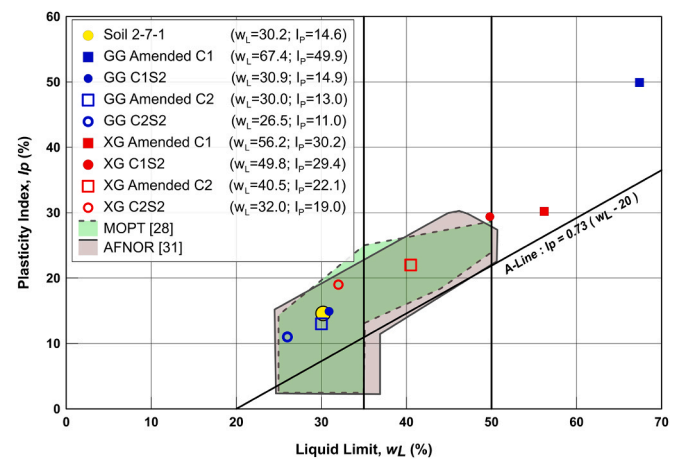


Fig. 5. Plasticity properties of unamended, amended and recycled soil mixtures used in this study.

network of adhesion, hydrogen and ionic bonds, the recovery made by β -mannanase shows a promising potential for recycling.

The particle size distribution for the second cycle of recycling for the xanthan gum-stabilised soil is presented in Fig. 4d. Even in the second cycle, the observed trends of recycling were similar to that of the first cycle of recycling for both stages. In the second cycle of recycling, β -mannanase washing recovered coarser fractions of the soil mixture indicating its effectiveness in recovering coarser sand and gravel component of the soil mixture. However, there is still less material recovery for finer fractions of the soil mixture (i.e. silt and fine sand particles) due the stronger chemical bonds established by xanthan gum. The material falls almost completely below the limits of the MOPT [28] and AFNOR [31] guidelines which would mean additional finer fractions would need to be added for it to be used again for manufacturing earthen construction materials.

3.2. Plasticity properties

The plasticity properties of biopolymer-stabilised soil samples before and after the second stage of recycling for both cycles are plotted on a plasticity chart in Fig. 5. It can be observed from Fig. 5, that the addition of guar gum significantly influences the plasticity properties of the soil mixture. The hydrogels formed by guar gum within the soil exhibit greater water absorption due to the high hydrophilic propensity of the numerous hydroxyl groups present in guar gum [40,44]. The influence of the high hydrophilic propensity of guar gum is more prominent at the liquid limit of the soil as more amount water would be needed to neutralise hydrogen bonds and achieve the liquid limit state [44]. The increased liquid limit value pushes the plasticity range of guar gum amended soil mixture comparable to that of high-plasticity clays (CH). After the completion of the first cycle of recycling (GG C1S2), the plasticity values of the recycled soil mixture fall back to values similar to those of the unamended soil mixture. This fallback of the plasticity values to the original values highlights the effectiveness of β -mannanase in neutralising and/or removing guar gum strains formed within the soil and aiding the recycling process. It is very interesting to note that before the second cycle of recycling, the liquid limit value of the guar gum amended soil was very similar to that of the unamended soil mix. Even after adding guar gum, the amended soil does not exhibit higher plasticity values as observed before the first cycle of recycling. This inability to achieve higher plasticity values for the guar gum stabilised sample may be attributed to two reasons. Firstly, β -mannanase would have essentially neutralised the hydrogels formed around clay particles, rather than completely removing them, causing hindrance in interaction between the added guar gum and the clay particles within in the recycled soil mixture. Secondly, some remnants of β -mannanase may have

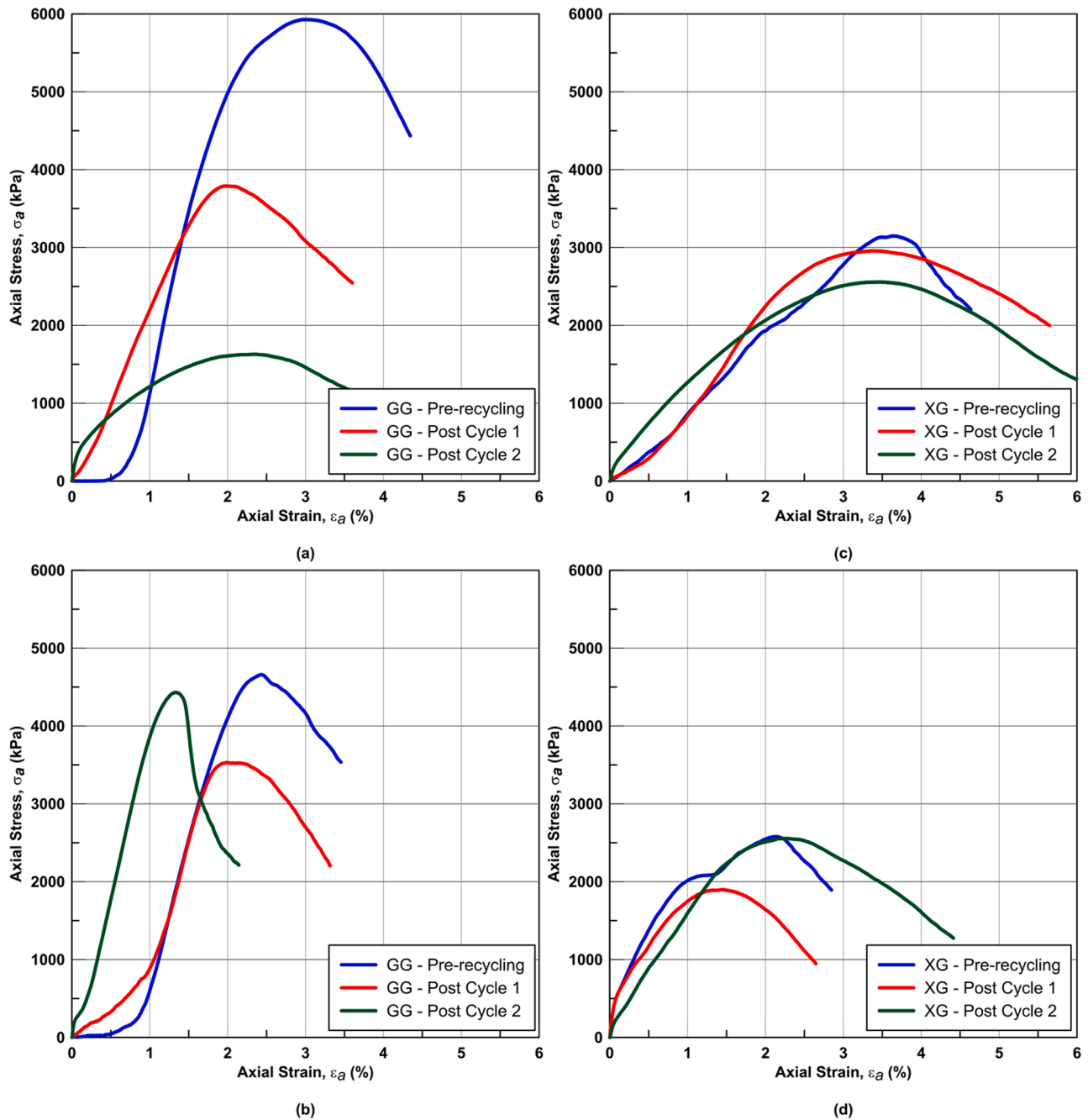


Fig. 6. Stress-strain behaviour in UC tests (for each condition only one test from the three replicates is shown): (a) guar gum, 7 days (b) guar gum, 28 days (c) xanthan gum, 7 days (d) xanthan gum, 28 days.

prevailed from the first cycle of recycling, which would additionally hinder the process of inducing plasticity to the soil. This hypothesis seems to be a reasonable justification, considering the plasticity values fall back even farther after the second cycle of recycling (GG C2S2). Though the plasticity values of the recycled soil mixtures have reduced after two rounds of recycling, they remain well within the recommended values of plasticity as per MOPT [28] and AFNOR [31] indicating their ability to be used again for preparation of earthen construction material for future uses.

In the case of xanthan gum, the addition of the biopolymer increases both the liquid and plastic limits, similar to the behaviour noticed with guar gum. However, the influence of xanthan gum is more pronounced on the plastic limit of the soil than the liquid limit. Xanthan gum has comparatively lesser water absorption capabilities than guar gum due to

its intrinsic chemical characteristics [40]. Further, these characteristics along with the helix structure of the xanthan gum lead to the formation of more stable soil agglomerations, that are bonded with stronger ionic bonds along with hydrogen bonds. The comparatively lesser water absorption capacity and more stable soil agglomerations of the xanthan gum amended soil lead to the observed plasticity values, which after the first cycle of recycling (XG C1S2) seem to have reduced only slightly. This slight reduction in Atterberg limits for recycled xanthan gum samples is primarily due to the removal of hydroxyl groups from xanthan gum hydrogels by β -mannanase. After the first cycle of recycling, the plasticity values fall just outside the recommended plasticity values as per MOPT [28] and AFNOR [31] indicating that the recycled soil mixture is tentatively usable for manufacturing earthen construction materials. Before the second stage of recycling, the plasticity values of

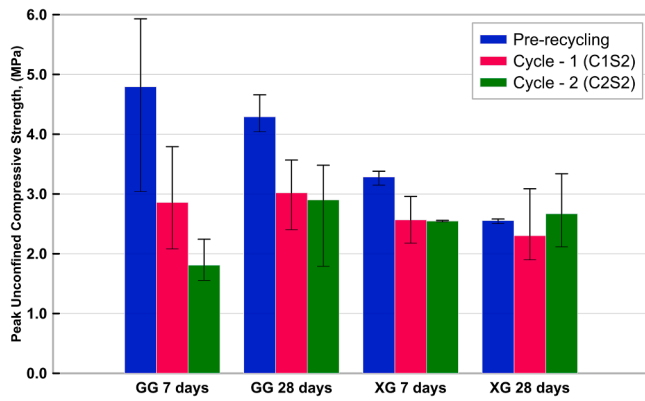


Fig. 7. Plot of the UCS of samples at the end of each recycling cycle for each biopolymer stabilisation, with the two curing conditions of 7 days and 28 days.

the re-amended soil mixture are observed to be reduced. This reduction in the plasticity values is primarily due to the formation of more stable soil agglomerations, meaning less water would be needed to achieve both liquid and plastic limit states. On recycling with β -mannanase (XG C2S2), the large number of weaker hydrogen bonds formed by hydroxyl groups are easily removed and the recycled soil mixture with a greater

number of stable soil agglomerations needs comparatively less water to achieve the observed plasticity behaviour. It is interesting to note that, after the second cycle of recycling the plasticity values are well within the recommended value highlighting their ability to be used again.

3.3. Mechanical strength results

The biopolymer-stabilised earthen construction material was tested for its compressive and tensile strength properties before recycling and after each complete cycle of recycling. Figs. 6 and 7 present the stress-strain behaviour under compression and peak UC compression respectively for both guar gum and xanthan gum after each cycle of recycling for the two different curing periods. Fig. 8 presents typical failure patterns of UC compression samples observed before and after each cycle of recycling for both guar and xanthan gum stabilised earthen construction materials. Figs. 9 and 10 present peak tensile strengths and typical failure patterns for tensile specimens for both guar gum and xanthan gum after each cycle of recycling.

The guar gum samples cured for 7 and 28 days have high initial peak stresses and strength due to the strong hydrogel bonds formed between the biopolymer and the soil particles. However, after each cycle of recycling there is a noticeable decrease in the strength. This reduction in strength for guar gum samples after recycling is more pronounced during the 7-day curing period than the 28-day. As seen previously,

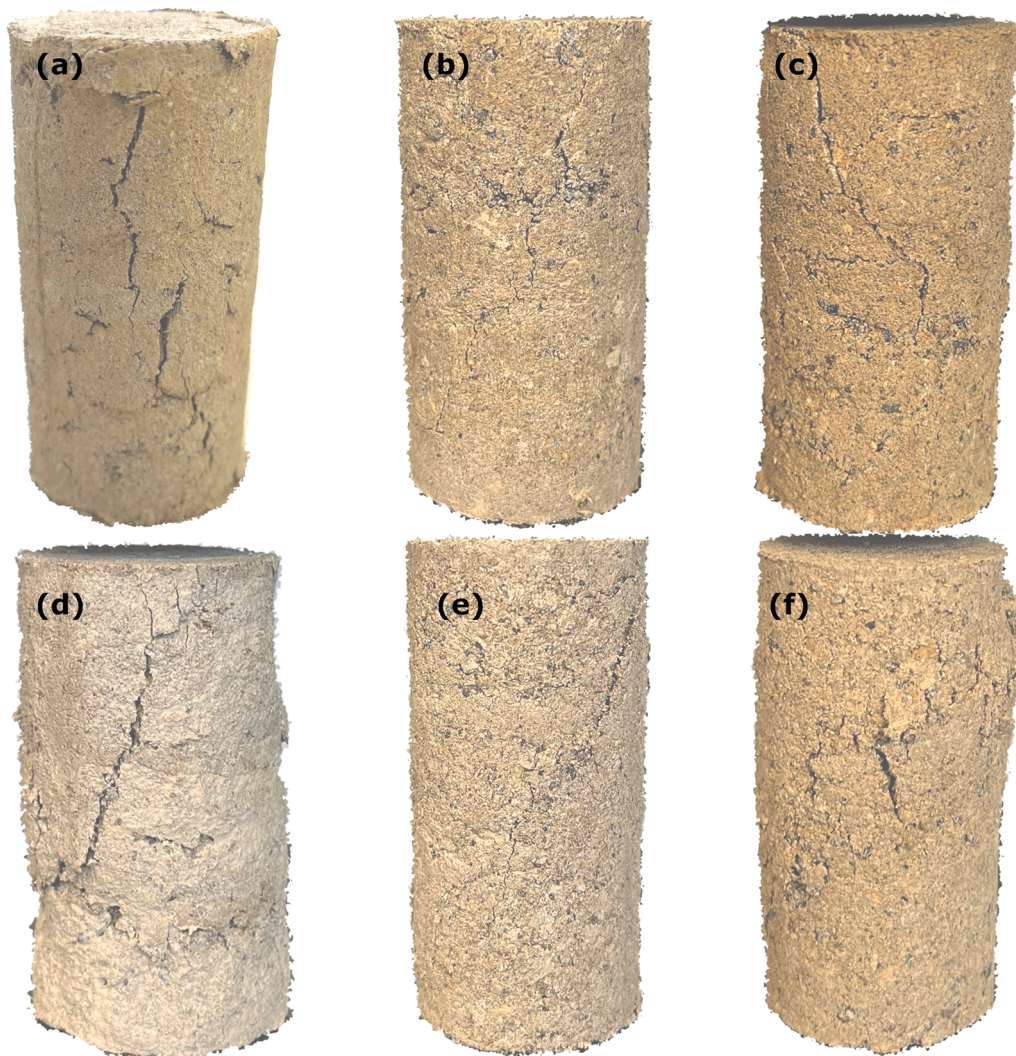


Fig. 8. Typical UC failure patterns observed for all samples: guar gum samples (a) pre-recycling, (b) after Cycle 1 (C1S1), (c) after Cycle 2 (C2S2), for xanthan gum samples (d) pre-recycling, (e) after Cycle 1 (C1S1), and (f) after Cycle 2 (C2S2).

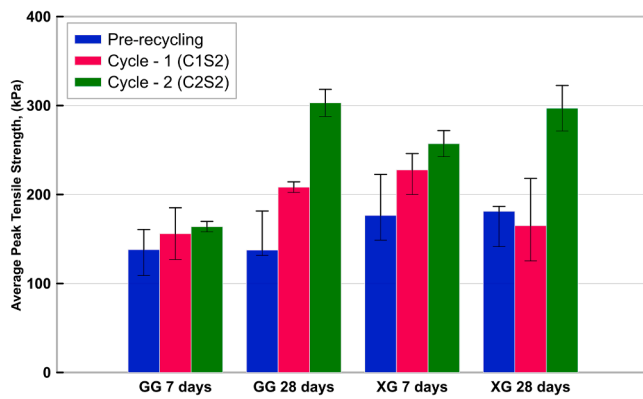


Fig. 9. Plot of the peak tensile strength at the end of each recycling cycle for each biopolymer stabilisation, with the two curing conditions of 7 days and 28 days.

recycling guar gum samples with β -mannanase has been efficient in terms of reviving the original particle size distribution, however, the neutralised guar gum strains and residual β -mannanase after each cycle of recycling hinder the biopolymer hydrogel-clay interactions resulting in reduced plasticity. The neutralised guar gum strains and/or residual β -mannanase may set on the surfaces of the soil agglomerations formed during the initial stabilisation process and may act as a barrier to the freshly added guar gum. Further, these residual layers from each cycle of recycling and the newly formed hydrogels might essentially lead to smoother particle-to-particle interaction thus reducing particle friction, which is a key component of compressive strength in the soil. At the early stages of curing (7 days), the hydrogels formed by guar gum are essentially in a thick gelatinous state which eventually dry out to a thinner crystalline state after 28 days [45]. These morphological changes of the biopolymer hydrogel have a significant influence on the mechanical behaviour of the guar gum amended soil [18] leading from semi-ductile to semi-brittle failure between 7-day and 28-day curing period (Figs. 6a and 6b). The reduced peak stresses with each cycle of recycling are primarily due to the reduced particle friction which does not contribute to the overall strength, and thus resulting in the reduction of the peak compressive strength for guar gum samples (Fig. 7). Interestingly, the tensile strength of guar gum samples tends to increase with each cycle of recycling (Fig. 9). With lesser hydrogel-to-soil interactions

after each cycle of recycling, the hydrogels formed within the recycled samples tend to consolidate and wrap around soil agglomerations, and tend to achieve higher tensile stress under the applied load.

Unlike guar gum samples, xanthan gum samples exhibited a different mechanical behaviour revealing a more ductile behaviour after each cycle of recycling at both curing periods (Figs. 6c & 6d). Further, the peak unconfined compressive strengths observed were in the proximity of ~ 2500 kPa. As discussed previously, the intrinsic chemical characteristics of xanthan gum being an anionic polysaccharide have a different primary mechanism in controlling the strength. Xanthan gum would essentially form more stable soil agglomerations which are fairly resilient against disintegration on washing with β -mannanase. Only weaker soil agglomerations formed by hydroxyl groups of the biopolymer would be separated on being washed with β -mannanase. On the re-addition of the xanthan gum before the second cycle of recycling, the hydrogels would form around the previously formed soil agglomerations but effectively have much less interactions with clay particles than in the initial soil mix. However, unlike guar gum, hydrogels formed by xanthan gum after each cycle of recycling seem to coherently bond with soil agglomerations and any residual xanthan gum strains highlighting the regenerative capabilities of xanthan gum [26]. Concerning the tensile strength, with each cycle of recycling, the tensile strength of the xanthan gum samples has increased (Fig. 9). Like guar gum, the increase in tensile strength after each cycle of recycling is attributed to how hydrogels tend to consolidate and wrap around soil agglomerations. The regenerative capabilities of xanthan gum should be observed in conjunction with both compressive and tensile strengths achieved after each cycle of recycling. The consistent compressive strength and increasing tensile strength suggest the resilience of soil agglomerations formed against washing [26]. However, the use of β -mannanase would essentially eliminate excess hydrophilic hydroxyl groups from xanthan gum samples, enabling the recycled soil mixture to be used again for manufacturing earthen construction material.

4. Conclusions

The findings from this novel study highlight the regenerative soil stabilising capabilities of guar and xanthan gum, the effectiveness of using β -mannanase to recycle biopolymer stabilised earthen construction materials, and the capability of using the recycled soil mix to manufacture earthen construction material again. As a recycling agent,

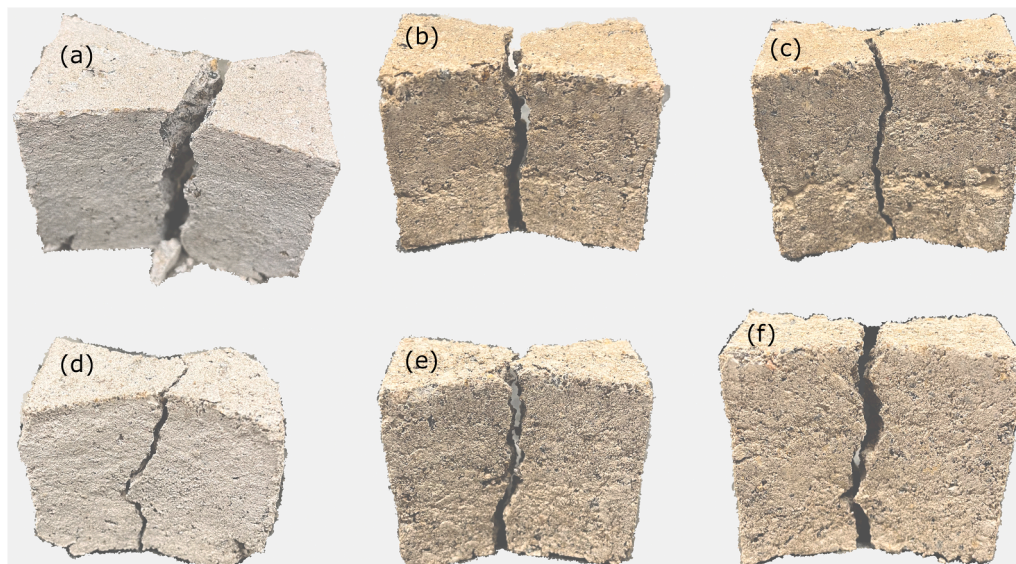


Fig. 10. Typical tensile failure patterns observed for all samples: guar gum samples, (a) pre-recycling, (b) after cycle 1 (C1S1), (c) after cycle 2 (C2S2), for xanthan gum samples (d) pre-recycling, (e) after cycle 1 (C1S1), and (f) after cycle 2 (C2S2).

β -mannanase showed its effectiveness in thoroughly removing guar gum strains from stabilised earthen material and thus retrieving most of the original soil mixture in terms of its particle size distribution and soil plasticity. Repeated washing cycles with β -mannanase appear to neutralise the remaining guar gum residues, resulting in reduced compressive strengths. However, tensile strengths remain unaffected, indicating the limited regenerative capabilities of guar gum. In the case of xanthan gum, the intrinsic chemical properties of the biopolymer allowed the soil mixture to form more stable agglomerations that resisted recycling. However, β -mannanase still managed to break down weaker soil agglomerations, enabling the recovery of smaller soil fractions. Xanthan gum's regenerative capabilities allowed it to effectively re-establish bonds with the newly added biopolymer, even after recycling within the soil mixture, leading to improved mechanical performance of the material. The findings from this study opens two exciting research avenues, first, to explore bio-additives like β -mannanase for recycling biopolymer-stabilised earthen materials and second, to investigate the regenerative capabilities of guar and xanthan gums specifically in the context of soil stabilisation. Recycling of biopolymer-stabilised earthen construction materials would enable the completion of the life cycle of the material, a crucial step for classifying earthen construction materials as sustainable, while the regenerative capabilities of biopolymers would enable multiple uses of the recycled earthen construction material.

CRediT authorship contribution statement

Pravara Muguda: Writing – review & editing, Validation, Supervision, Methodology, Conceptualization. **Mia Wyndham:** Writing – original draft, Methodology, Investigation.

Declaration of Competing 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.

Data availability

Data will be made available on request.

References

- [1] H. Schroeder, Sustainable Building with Earth, Springer International Publishing, Cham, 2016, <https://doi.org/10.1007/978-3-319-19491-2-7>.
- [2] D. Gallipoli, A.W. Bruno, C. Perlot, N. Salmon, Raw Earth Construction: Is There a Role for Unsaturated Soil Mechanics, Taylor & Francis Group, London, UK, 2014.
- [3] J.-C. Morel, R. Charef, E. Hamard, A. Fabbri, C. Beckett, Q.-B. Bui, Earth as construction material in the circular economy context: practitioner perspectives on barriers to overcome, Philos. Trans. R. Soc. B: Biol. Sci. 376 (2021) 20200182, <https://doi.org/10.1098/rstb.2020.0182>.
- [4] V. Maniatidis, P. Walker, A Review of Rammed Earth Construction, Bath, United Kingdom, 2003.
- [5] C. Lax, Life Cycle Assessment of Rammed Earth, University of Bath, 2010.
- [6] D. Maskell, A. Heath, P. Walker, Comparing the environmental impact of stabilisers for unfired earth construction, Key Eng. Mater. 600 (2014) 132–143, <https://doi.org/10.2966/scrp>.
- [7] R. Geyer, B. Kuczenski, T. Zink, A. Henderson, Common misconceptions about recycling, J. Ind. Ecol. 20 (2016) 1010–1017.
- [8] V. Cristini, C. Mileto, F.V. López-Manzanares, J.R.R. Checa, Recycling of bricks in rammed earth walls. Vernacular Heritage & Earthen Architecture: Contributions for Sustainable Development, Taylor & Francis Group, London, 2014, pp. 383–388.
- [9] A. Arrigoni, R. Pelosato, G. Dotelli, C.T.S. Beckett, D. Ciancio, Weathering's beneficial effect on waste-stabilised rammed earth: a chemical and microstructural investigation, Constr. Build. Mater. 140 (2017) 157–166, <https://doi.org/10.1016/j.conbuildmat.2017.02.009>.
- [10] E. Pelicaen, R. Novais Passarelli, E. Knapen, Reclaiming earth blocks using various techniques, IOP Conf. Ser. Earth Environ. Sci. 1363 (2024) 012100, <https://doi.org/10.1088/1755-1315/1363/1/012100>.
- [11] J. Plank, Applications of biopolymers and other biotechnological products in building materials, Appl. Microbiol. Biotechnol. 66 (2004) 1–9, <https://doi.org/10.1007/s00253-004-1714-3>.
- [12] D. Mudgil, S. Barak, B.S. Khatkar, Guar gum: processing, properties and food applications – a review, J. Food Sci. Technol. 51 (2014) 409–418, <https://doi.org/10.1007/s13197-011-0522-x>.
- [13] N. Thombare, U. Jha, S. Mishra, M.Z. Siddiqui, Guar gum as a promising starting material for diverse applications: a review, Int. J. Biol. Macromol. 88 (2016) 361–372, <https://doi.org/10.1016/j.ijbiomac.2016.04.001>.
- [14] A. Becker, F. Katzen, A. Pühler, L. Ielpi, Xanthan gum biosynthesis and application: a biochemical/genetic perspective, Appl. Microbiol. Biotechnol. 50 (1998) 145–152, <https://doi.org/10.1007/s002530051269>.
- [15] J. Krishna Leela, G. Sharma, Studies on xanthan production from Xanthomonas campestris, Bioprocess Eng. 23 (2000) 687–689, <https://doi.org/10.1007/s004499900054>.
- [16] D. Saha, S. Bhattacharya, Hydrocolloids as thickening and gelling agents in food: a critical review, J. Food Sci. Technol. 47 (2010) 587–597.
- [17] I. Chang, J. Im, G.C. Cho, Introduction of microbial biopolymers in soil treatment for future environmentally-friendly and sustainable geotechnical engineering, Sustainability 8 (2016), <https://doi.org/10.3390/su8030251>.
- [18] S. Muguda, S.J. Booth, P.N. Hughes, C.E. Augarde, C. Perlot, A.W. Bruno, D. Gallipoli, Mechanical properties of biopolymer-stabilised soil-based construction materials, Géotech. Lett. 7 (2017) 309–314, <https://doi.org/10.1680/jgele.17.00081>.
- [19] I. Chang, A.K. Prasadhi, J. Im, H.D. Shin, G.C. Cho, Soil treatment using microbial biopolymers for anti-desertification purposes, Geoderma 253–254 (2015) 39–47, <https://doi.org/10.1016/j.geoderma.2015.04.006>.
- [20] A. Soldo, M. Miletić, M.L. Auad, Biopolymers as a sustainable solution for the enhancement of soil mechanical properties, Sci. Rep. 10 (2020) 1–13, <https://doi.org/10.1038/s41598-019-57135-x>.
- [21] H. Sulaiman, M.R. Taha, N. Abd Rahman, A. Mohd Taib, Performance of soil stabilized with biopolymer materials – xanthan gum and guar gum, Phys. Chem. Earth Parts A/B/C 128 (2022) 103276, <https://doi.org/10.1016/j.pce.2022.103276>.
- [22] M.U. Qureshi, I. Chang, K. Al-Sadarani, Strength and durability characteristics of biopolymer-treated desert sand, Geomech. Eng. 12 (2017) 785–801, <https://doi.org/10.12989/gae.2017.12.5.785>.
- [23] S. Muguda, G. Lucas, P.N. Hughes, C.E. Augarde, C. Perlot, A.W. Bruno, D. Gallipoli, Durability and hygroscopic behaviour of biopolymer stabilised earthen construction materials, Constr. Build. Mater. 259 (2020) 119725, <https://doi.org/10.1016/j.conbuildmat.2020.119725>.
- [24] S. Muguda, P.N. Hughes, C.E. Augarde, C. Perlot, A.W. Bruno, D. Gallipoli, Geotechnical characterisation of recycled biopolymer-stabilised earthen materials, in: Proceedings of the XVII ECSMGE-2019, Reykjavik, Iceland, 2019.
- [25] A.W. Bruno, B. Scott, Y. D'Offay-Mancienne, C. Perlot, Recyclability, durability and water vapour adsorption of unstabilised and stabilised compressed earth bricks, Mater. Struct. 53 (2020) 149, <https://doi.org/10.1617/s11527-020-01585-7>.
- [26] A. Soldo, M. Miletić, Durability against Wetting-drying cycles of sustainable biopolymer-treated soil, Polymers 14 (2022) 4247, <https://doi.org/10.3390/polym14194247>.
- [27] M. Olivier, A. Mesbah, Influence of different parametres on the resistance of earth, used as a building material, Mud Archit. (1987).
- [28] MOPT, Bases Para el Diseño y Construcción con Tapial, Centro de Publicaciones, Secretaría General Técnica, Ministerio de Obras Públicas y Transportes, Madrid, Spain, 1992.
- [29] H. Houben, H. Guillaud, Earth Construction. A Comprehensive Guide, Distributeur Craterre-Eag, 1994.
- [30] H. Guillaud, T. Joffroy, P. Odul, CRATERRE-EAG, Compressed Earth Blocks–Manual of Design and Construction: Volume II, 1995.
- [31] AFNOR, Compressed earth blocks for walls and partitions: definitions – Specifications – Test methods – Delivery acceptance conditions, 2001.
- [32] BS 1377-2, Methods of Test for Soils for Civil Engineering Purposes – Part 2: Classification Tests, BSI, London, 1990.
- [33] BS 1377-4, Methods of Test for Soils for Civil Engineering Purposes – Part 4: Compaction Tests, BSI, London, 1990.
- [34] S. Muguda, P.N. Hughes, C.E. Augarde, C. Perlot, A.W. Bruno, D. Gallipoli, S. Muguda, P.N. Hughes, C.E. Augarde, Cross-linking of biopolymers for stabilizing earthen construction materials, Build. Res. Inf. (2021) 1–13, <https://doi.org/10.1080/09613218.2021.2001304>.
- [35] P.S. Chauhan, N. Puri, P. Sharma, N. Gupta, Mannanases: microbial sources, production, properties and potential biotechnological applications, Appl. Microbiol. Biotechnol. 93 (2012) 1817–1830.
- [36] A. Dawood, K. Ma, Applications of microbial β -mannanases, Front. Bioeng. Biotechnol. 8 (2020) 598630.
- [37] B.R. Mohapatra, Characterization of β -mannanase extracted from a novel Streptomyces species Alg-S25 immobilized on chitosan nanoparticles, Biotechnol. Biotechnol. Equip. 35 (2021) 150–161.
- [38] P.K. Srivastava, M. Kapoor, Recombinant GH-26 endo-mannanase from Bacillus sp. CFR1601: biochemical characterization and application in preparation of partially hydrolysed guar gum, LWT-Food Sci. Technol. 64 (2015) 809–816.
- [39] R.A. Stirling, P. Hughes, C.T. Davie, S. Glendinning, Tensile behaviour of unsaturated compacted clay soils – a direct assessment method, Appl. Clay Sci. 112–113 (2015) 123–133, <https://doi.org/10.1016/j.clay.2015.04.011>.
- [40] M.D. Torres, R. Moreira, F. Chenlo, M.J. Vázquez, Water adsorption isotherms of carboxymethyl cellulose, guar, locust bean, tragacanth and xanthan gums, Carbohydr. Polym. 89 (2012) 592–598.
- [41] BS 1377-7, Methods of Test for Soils for Civil Engineering Purposes – Part 7: Shear Strength Tests (Total Stress), BSI, London, 1990.

- [42] Enzyme Supplies Limited, Mannanase 50K Data Sheet, Oxford, UK, 2023. (<https://enzymesupplies.com/all-products/food-beverage/general-foods/mannanase-50k/>).
- [43] I. Chang, J. Im, A.K. Prasadhi, G.C. Cho, Effects of Xanthan gum biopolymer on soil strengthening, *Constr. Build. Mater.* 74 (2015) 65–72, <https://doi.org/10.1016/j.conbuildmat.2014.10.026>.
- [44] R.A. Nugent, G. Zhang, R.P. Gambrell, Effect of exopolymers on the liquid limit of clays and its engineering implications, *Transp. Res. Rec.: J. Transp. Res. Board* 2101 (2009) 34–43, <https://doi.org/10.3141/2101-05>.
- [45] R. Chen, *Bio Stabilisation for Geopolymer Enhancement and Mine tailings Dust Control*, The University of Arizona, 2014. (<http://hdl.handle.net/10150/319999>).