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What Makes Cow-Dung Stabilised Earthen Block Water-Resistant?

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Abstract. The water-resistance of cow-dung has made it a widely used stabiliser in traditional earthen structures in several Asian and African countries. Multiple studies have shown an improvement in water-resistance with the addition of cow-dung, but none provides insight into this behaviour. The present study investigates the water-resistance behaviour of cow-dung stabilised earthen blocks through an extensive experimental programme to identify and characterise the components of cowdung responsible for its water-resistance. Fresh cow-dung was collected and separated into fibres (>63 µm), medium-sized microbial aggregates (1-63 µm) and small-sized microbial aggregates (0.5-7 µm). Each component was mixed with soil and samples were prepared at different water contents (optimum water content corresponding to the highest dry density and water content higher than optimum) and compacted with 2.5 MPa force to prepare compressed blocks. The water-resistance of these blocks was evaluated through the immersion and modified drip/rain test. It was found that the small-sized microbial aggregates are almost entirely responsible for water-resistance behaviour of cow-dung stabilised earthen blocks. Small-sized microbial aggregates were further characterised by gas chromatography, mercury intrusion porosimetry, N₂- BET surface area, zeta potential measurement and electron microscopy. The results indicate that the small-sized microbial aggregates are composed of clay-sized negatively charged particles that are rich in fatty acids. The hydrophobicity of these particles is hypothesised to be responsible for water-resistance behaviour. These insights are further used to produce stabilised blocks that performed at least 30 times better than the unstabilised blocks in both water-resistance tests. The study concludes with practical recommendations for the use of wet cow-dung over dry cow-dung and a reduction of fibre content to increase the water-resistance of earthen blocks.

1 Introduction

The availability and water-resistant properties of cow-dung have made it a widely used plastering material and stabiliser in traditional earthen structures. Cow-dung stabilised earthen structures are widely spread across Asian and African countries such as India, Burkina Faso, Swaziland and Botswana (Agarwal, 1981; Kulshreshtha et al., 2020; Millogo et al., 2016; Ngowi, 1997; Vilane, 2010). Multiple studies have shown the positive impact of cow-dung on water-resistance performance (Lekshmi et al., 2020; Millogo et al., 2016; Ngowi, 1997; Vilane, 2010), but only one study proposes a mechanism to support these observations. Millogo et al. (2016) proposes that the reaction between cow-dung (amine compounds) and soil (quartz) under an alkaline condition results in formation of an insoluble compound that glues the soil aggregate together and improves the strength and water resistance. However, the compound was undetected in the testing and the alkaline condition was reported to result from the fermentation of cow-dung (measured pH of 12), which is significantly higher than the pH range of 6.5-9 reported in the literature (Huang et al., 2017; Rao et al., 2017;

Whalen et al., 2000). Therefore, there is a need for a comprehensive study to provide insight on water-resistance of cow-dung stabilised earthen materials.

Cow-dung is known to be composed of plant fibres, microorganisms, amine compounds, potassium, fragments of intestinal tissues and traces of sulphur, calcium, iron, magnesium, and manganese (Garg and Mudgal, 2007; Gupta et al., 2016; Millogo et al., 2016). Although water resistance was anecdotally attributed to fibres of cow-dung in a field survey (Kulshreshtha et al., 2020), their role in improving the water resistance is unclear (Laborel-Préneron et al., 2016). Therefore, an investigation into the role of various components of cow-dung, especially microbial biomass, may provide insights for water-resistance and could lead to the identification of compounds responsible for water-resistance of cow-dung stabilised earthen blocks.

The present study investigates the water-resistance behaviour of cow-dung stabilised earthen blocks through an extensive experimental programme to identify and characterise the components of cow dung responsible for its water-resistance. The obtained insights were then used to layout practical recommendations and guidelines that would be valuable for practitioners building earthen structures stabilised with cow-dung.

2 Materials and Methods

2.1 Soil

The soil used in the investigation was supplied by Oskam V/F (Netherlands). This soil is used in the commercial production of compressed stabilised earthen blocks (CSEB) in the Netherlands. A thorough characterisation of the soil was carried out and the results are summarised in Tab. 1.

Properties	Value	Method	Properties	Value	Method
Grain size distribution			Atterberg limits		
Clay (<0.002 mm) [%]	14.8	Hydrometer	Liquid Limit [%]	28.8	Falling cone
Silt (0.002-0.074 mm) [%]	16.5	Wet sieving	Plastic Limit [%]	15.2	Thread
Sand (0.075-4.74 mm) [%]	68.5	Wet sieving	Plasticity Index [LL-PL]	13.6	
Fine gravel (4.75-6.74 mm)	0.2	Wet sieving	Unified soil classification	CL	
[%]			system		
Cation exchange capacity		Co (III)-	Natural water content [wt	3.5	Oven drying at
(meq/100 g)		hexamine	%]		105°C
Bulk soil	9.6		Compaction		Standard
			characteristics		proctor
Clay fractions ($<0.002 \text{ mm}$)	78.7		Maximum dry density	1980	
			$[kg/m^3]$		
Pre-dominant clay minerals		XRD	Optimum Moisture content	11.1	
[wt %]		(<0.002mm)	[%]		
Smectite	37.2		Specific Gravity	2.6887	Ultrapycnometer
рН	7.36	pH meter	Loss on ignition [%]	1.15	Heating at
					550°C

Tab	1:	Summary	of soil	properties
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2.2 Collection of fresh cow-dung and separation of components

Fresh cow-dung was collected from a biological farm 'Hoeve Biesland' located in Delfgauw, Netherlands. It was collected in plastic buckets (with airtight lid) and stored at room temperature until its usage. The solid content in the fresh cow-dung was measured to be 10.8%, and its pH was measured to be 7.55.

Various components of fresh cow-dung were extracted through sieving, followed by centrifugation (5000 rpm for 10min) as shown in Fig. 1. The components were separated into Fibres, Medium-Sized Microbial Aggregates (MSMA) and Small-Sized Microbial Aggregates (SSMA). The particle size (mean and range) for microbial biomass was measured in the particle size analyser (Eyetech, Ankersmid), and is included in the figure. The investigated solid cow-dung is composed of 42% fibres (by weight) and 58% (by weight) microbial aggregates.



Fig. 1: Extraction of various components from fresh cow-dung. The SSMA and MSMA stands for Small-sized microbial aggregates and Medium-sized microbial aggregates respectively

2.3 Preparation of stabilised earthen blocks

Stabilised mixtures were prepared by adding calculated amounts of stabilisers to the soil and mixing it thoroughly by hand for 5-10 minutes. In the soil and cow-dung mixture, the amount of fresh cowdung added to the soil was equivalent to 2% dry cow-dung (by weight). This amount was chosen based on the desired consistency for manufacturing compacted blocks and proven effectiveness shown in existing literature on cow-dung stabilised earthen material (Millogo et al., 2016). The amount of all three extracted components was determined by their respective proportion in cow-dung. For example, SSMA was seen to be 34% of the solid mass of cow-dung. Therefore, the corresponding amount of SSMA added in soil was equivalent to 0.68% (34% of 2) dry mass of SSMA. All the components, except fibres, were extracted from the fresh cow dung and used within 24h. All mixtures were prepared at an optimum moisture content (OMC) corresponding to the maximum dry density (appox. 11% for all mixtures; soil+ cow-dung: 13%), and a water content higher than optimum (16-17%) based on values obtained through the Harvard miniature compaction test (Humboldt, USA). The Harvard miniature compaction apparatus was preferred over the commonly used standard Proctor apparatus due to limited available material (~130-160g) and effort required in compaction. If required, water was added to or evaporated from the stabiliser before mixing it with the soil. The prepared mixtures were kept in metal containers sealed with plastic sheet and stored for 5 days at room temperature. 5 days storage was adopted because ageing of soil-stabiliser mix reduced the smell of cow-dung considerably.

After 5 days, the soil-stabiliser mixture was used to prepare compressed stabilised earthen blocks (CSEB). The preparation process for the production of the earthen blocks can be viewed in this video: https://youtu.be/yc37SiTtrFM. The mixture was filled in a self-designed assembly capable of producing 9 blocks of $40 \times 40 \times 40$ (±1) mm³ simultaneously. The amount of material added to the assembly was calculated based on the desired dry density of the block. Each block was compacted with 2.5 MPa force. No releasing agent (e.g. oil) was used in the mould, and it was dismantled immediately after compaction. These blocks were cured for 14 days in a temperature (20°C) and humidity (55%) controlled room.

2.4 Durability tests

Out of the nine blocks prepared for each type of mix, 3 were used for an immersion test, 3 for an accelerated erosion test and 3 for compression strength testing. The immersion test and accelerated erosion test are appropriate to assess the efficacy of stabiliser (Beckett et al., 2020) and therefore, were chosen for the experiment. The immersion test was carried out by placing the soil blocks in a glass jar filled with water and images were captured with a digital camera (Canon 70D) at different time intervals spread over 24 hours (1-5 min (video),10min, 15 min, 20 min, 30 min, 1h, 2h, 4h, 6h, 9h, 12h and 24 hours). The accelerated erosion test assembly consists of water dripping from a

showerhead (placed at a height of 30cm) above the block, which is oriented at 27° to horizontal. The rate of flow of water was adjusted to be 50 ml/min based on a study by (Nakamatsu et al., 2017) and the mass loss (in %) was calculated at 2, 10 or 60 mins based on the resistance of the individual blocks.

In this article, only the results from the immersion test are presented and discussed. The results from accelerated erosion test are occasionally used in this article to quantify the effects of stabilisers. It should be noted that the trends observed in both tests were comparable. Results of the strength tests are excluded from this study. The compressive strength of all the blocks was measured between 2.2 and 3.8 MPa.

2.5 Characterisation tests

Multiple physical and chemical characterisation tests were performed on microbial aggregates (specifically the ones responsible for water-resistance) and compacted blocks to gain insight into the behaviour of the material, with respect to water-ingress and resistance. Zeta potential measurement was conducted using a zeta potential device (Zetasizer Nano ZS) to find the net charge on aggregates and the influence of pH (at solid concentration 0.073%). Environmental scanning electron microscope - (ESEM) was used along with X-ray energy dispersive system (EDS) for element chemical analysis. Images were taken at various magnifications (125x-20000x) to understand the microstructure of the samples. Surface area was measured through N₂- BET (Micromeritics Gemini VII). Mercury intrusion porosimetry (MIP) was conducted using Autopore IV equipment (Micromeritics) to understand the role of pore-structure on water-ingress. Sample preparation and analysis was carried out based on the method used by Bruno (2016). A sessile drop test was also performed on microbial aggregates to study the surface energy (hydrophilic and hydrophobic behaviour) of powder. Gas chromatography-mass spectrometry (GC-MS) was conducted to identify compounds in the microbial aggregates responsible for water-resistance behaviour.

3 Results and Discussion

3.1 Component responsible for water-resistance of cow-dung

The water-resistance of earthen blocks stabilised with the components extracted from cow-dung is visualised in Fig. 2 (using the immersion test). The addition of cow-dung improved the water-resistance of the earthen block significantly. While an unstabilised block completely disintegrated within 15 minutes of immersion, a cow-dung stabilised earthen block survived for over 12 hours (first visible cracks appearing at 2 hours). It can be clearly observed that the small-sized microbial aggregates (SSMA) are almost entirely responsible for the water-resistance behaviour. In accelerated erosion test, the mass loss in both cow-dung stabilised and SSMA stabilised blocks was less than 0.5% after 1 hour, whereas the mass loss in unstabilised blocks was about 5% after 2 minutes. Therefore, the addition of fresh cow-dung and SSMA results in more than 30 times improvement in water-resistance based on the accelerated erosion test results.



Immersion duration

Fig. 2: Immersion test of stabilised earthen block. The relative percentage of stabiliser added to blocks is also indicated. These blocks were prepared at a water content of 16-17%, higher than the optimum water content. The role of SSMA in providing water-resistance to cow-dung can be clearly observed

3.2 Properties of small-sized microbial aggregates (SSMA)

Small-sized microbial aggregates (SSMA) that constitute approximately one-third of the mass of cow-dung are responsible for water-resistance behaviour of cow-dung. The results from the characterisation study are summarised in Fig. 3. Zeta potential measurement shows that SSMAs are negatively charged aggregates. A decrease in pH results in increase in coagulation, which may also facilitate interaction with aggregates present in the soil. These microbial aggregates have a small surface area $(2m^2/g)$, indicating that they have a non-layered structure and have negligible cation exchange capacity. In contrast, clay particles used in the study have 52 m²/g surface area. ESEM-EDS shows that SSMA contains minerals such as phosphorus, magnesium, sulphur and calcium. These minerals were also observed in previous studies related to cow-dung (Garg and Mudgal, 2007; Millogo et al., 2016). Rod-shaped bacteria were also observed, similar to a study by Rao et al. (2020). The results from gas chromatography indicates that SSMAs are rich in volatile short, long and very long-chained fatty acids. Short-chain fatty acids in a cow's gut are produced through the digestion of dietary fibres by the gut bacteria (Brody, 1999). One of the dominant fatty acids, octadecanoic acid, has been used with silica nanoparticle to prepare water-resistant super hydrophobic coatings (Heale et al., 2018). The hydrophobicity is also observed in SSMA powder and it indicates that the waterresistance behaviour of cow-dung is possibly governed by the hydrophobic properties of SSMA. As observed in Fig. 3, the water droplets have the tendency to slide down the SSMA. After multiple trials, a stable droplet could be achieved which had a contact angle of about 120°. The droplet did not percolate into the material even after an hour. Therefore, the SSMA powder also acts as a barrier to the water ingress. The MIP results shows that the SSMA stabilised earthen block has a higher porosity and larger pore size as compared to unstabilised block. Therefore, pore-filling, as observed in most biological stabilisers, may not be responsible for improved water-resistance. This further reinforces the role of hydrophobic properties of SSMA in resisting water-ingress.



Fig. 3: Summary of characterisation test results on small-sized microbial aggregates (SSMA). The tests have been conducted on fresh SSMA, dried SSMA or powdered SSMA

3.3 Recommendation for practical application: Remove fibres from cow-dung before use

The methodology used to extract the different components in lab, especially centrifugation, cannot be reproduced in the field using local resources. Therefore, tests were also conducted with a combined mixture of small and medium-sized microbial aggregates left after extraction of fibres from cowdung. This cow-dung stabilised sample without fibres was compacted at OMC, making it compatible with compressed block-making machines used in actual practice (use of CSEB machines for compacting wet soil may cause damage to the machine). Stabilisation using cow-dung without fibres results in a significant improvement over cow-dung stabilisation, with the first crack appearing after 6 hours (Fig. 4). Therefore, reduction of fibres from cow-dung before use can improve waterresistance.



Immersion duration

Fig. 4: Immersion test of blocks stabilised with cow- dung, SSMA and cow-dung without fibres. Please note that blocks stabilised without fibres were compacted at OMC, and due to limitation of compaction assembly, the bottom edges were not thoroughly compacted, leading to its disintegration immediately

For field application, it is possible to extract the fibres from the cow-dung using sieves. However, extraction of fibres through sieves can be time-intensive and increase the overall cost of construction. Therefore, a careful assessment should be carried out on the pro and cons of fibre extraction.

3.4 Recommendation for practical application: Use fresh cow-dung

Most of the previous studies on cow-dung utilised dry cow-dung and a drastic improvement of waterresistance was not reported. In order to understand the difference between fresh and dried cow-dung, dry cow-dung stabilised blocks were prepared and the water-resistance was tested. Fig. 5 gives clear visual evidence that dry cow-dung is not as effective a stabiliser as fresh cow-dung. Stabilised blocks made with dried cow-dung did not survive 1h of immersion. In the accelerated water erosion test, the mass loss of dry cow-dung stabilised block after 10 minutes was 1%, whereas the mass loss for fresh cow-dung stabilised was less than 0.5% even after 60 minutes.

The reason behind the significant difference in properties of dry and wet cow-dung is proposed based on the difference in mean sizes of fresh small-sized microbial aggregates (particles in suspension) and dried SSMA powder (produced by grinding dried SSMA through ball milling). When the fresh SSMA were dried and ground, the mean diameter was roughly 10 times more than the fresh SSMA. Therefore, it can be hypothesised that when cow-dung dries, the SSMA (and MSMA) adhere to each other, thus reducing the effective surface area of aggregates significantly. The reduced surface area and increased size of microbial aggregates could narrow its spatial distribution in the block and reduce its interaction with other particles. Therefore, it is recommended to use fresh cow-dung.



Fig. 5: Immersion test of fresh and dry cow- dung stabilised, earthen block. The increase in the mean diameter, thus reduction in surface area, of SSMA upon drying and grinding could be responsible for poor performance of dried cow-dung stabilised blocks

4 Conclusion

Cow-dung is one of the most used, yet one of the least understood biological stabilisers in the field of earthen construction. In the present study, a series of experiments were conducted to identify that small-sized microbial aggregates rich in fatty acids are responsible for the water-resistance of cow-dung and cow-dung stabilised earthen blocks. Contrary to the anecdotal believe in the role of fibres in water resistance, this study provide evidence that reducing fibres in fact can improve the water-resistance of stabilised earthen blocks. It is interesting to note that the pH measurement on fresh and dried cow-dung, and multiple cow-dung-soil mixes were in the range of 6-9. This signifies that an alkaline medium and thereof, formation of insoluble compound, is not a pre-condition for the enhanced water-resistance behaviour of cow-dung stabilised earthen blocks.

With a growing interest in ecological building materials, research and application of earthen materials are expected to grow. In this regard, cow-dung stabilised earthen blocks can offer a significant improvement over unstabilised blocks by using locally available resources. The recommendations proposed in this article can facilitate architects, practitioners, self-builders and natural-building enthusiasts to build earthen houses that are affordable, durable and desirable.

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