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Unfired earth and diagnostics with methylene blue test

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Abstract. Unfired earth is one of the oldest building materials. Nowadays, the unfired earth is getting to forefront of interest for its properties that fit into principles of a sustainable building and improving of microclimate in interiors. Final properties of unfired earth are influenced by composition of earth mixtures and this composition can be very diverse. The essential component of earth mixtures is clay because clay fulfils a function of binder and ensures strength of unfired earth. Methylene blue test is a method that helps determine the type and amount of clay in earth mixtures. Significant part of this paper is focused on description of methylene blue test. Research describes in the paper is focused on methylene blue dye absorption by illite-kaolinite clay and dependence of amount of absorbed dye on percentage amount of clay in earth samples.

Introduction

Unfired earth is one of the oldest building materials. There are a lot of historic and modern buildings from unfired earth, e.g. The Great Wall of China or Berlin's Rammed-Earth Reconciliation chapel. Unfired earth had become minority building material in 2nd half of 20th century and it was replace by modern building material like concrete or steel. Nowadays, the unfired earth is getting to forefront of interest for its properties that fit into principles of a sustainable building and improving of microclimate in interiors. The growing interest is evident from a number of scientific papers or quantity of sales of unfired bricks by HELUZ cihlářský průmysl v.o.s [1–5].

Unfired earth is created from earth mixtures. There are three main components of earth: clay, sand, water. The clay fulfils a function of binder, the sand a function of filling agent and the water serves for activation of bonding properties of the clay and for good earth mixture processing. Final properties of unfired earth are influenced by composition of earth mixtures, especially amount and kind of clay and this composition can be very diverse. This is the main reasons why using of unfired earth in construction is complicated [1, 6–8].

The essential component of earth mixtures is clay because clay fulfils a function of binder, similarly like cement in binder, and ensures strength of unfired earth. Ability to determine amount and type of clay in earth mixtures could help solve this problem. Methylene blue test is a method that helps determine the type and amount of clay in earth mixtures [1, 6, 7, 9–11].

Principle of Methylene Blue Test

Basis of methylene blue test is absorption of methylene blue dye (methylthioninium chloride - $C_{16}H_{18}ClN_3S$) by clay. This experimental method is described in norm ČSN EN 933-9 [9].

Methylene blue solution (concentration 20 g/l) is inserted into earth sample (solution of clay and distilled water) by a burette (Fig. 1). Amount of one dose of methylene blue solution is 2 ml. The added dye solution in earth sample is stirred for 1 minute. After mixing, a drop of clay sample is placed on a filter paper.

This process is repeated and each sample drop with increasing amount of methylene blue solution is marked (Fig. 2). Methylene blue test is finished when a blue ring spreads around a drop sample (mark 5 in Fig. 2) because clay has already absorbed the maximum amount of methylene blue.

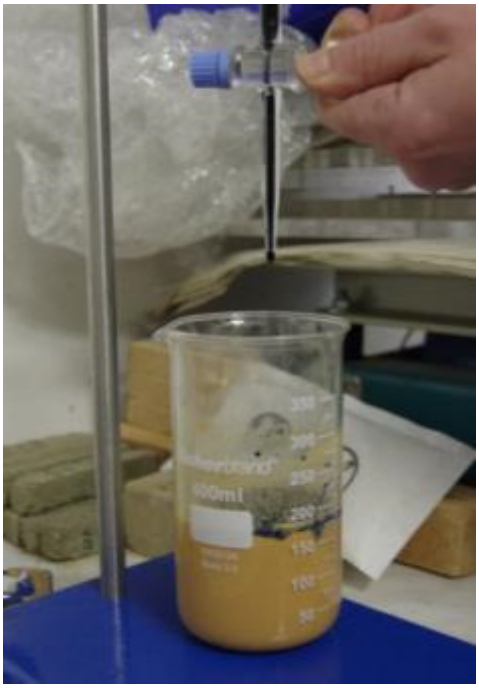

Fig. 1: Dye dosed into the earth sample

Dye solution consumption increases with increasing amount of clay in clay samples. Amount of dye absorbed also depends on a kind of clay. Amount of dye absorbed is determined by the equations 1. Amount of dye absorbed is defined as the amount of weight of dye in gram that is absorbed by 1 kilogram of test material.

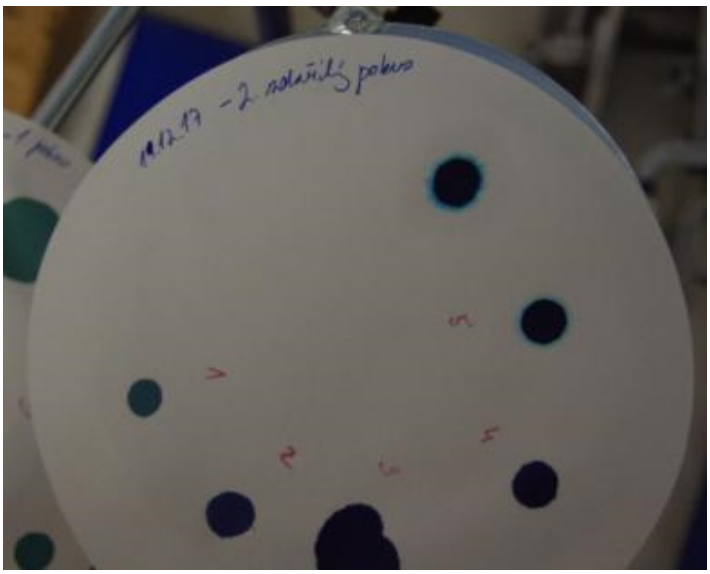

Fig. 2: A result of methylene blue test

$$M_{MB} = \frac{m_s . m_{MB,1ml} . 1000}{M_{ES}} \quad (1)$$

$M_{MB} - \ amount\ of\ dye\ absorbed\ \left[\frac{g}{kg}\right]$
$m_s - \ amount\ of\ methylene\ blue\ solution\ [g]$
$m_{MB,1ml} - \ amount\ of\ methylene\ blue\ in\ 1\ g\ of\ methylene\ blue\ solution\ [g]$
$m_{ES} - \ amounto\ of\ tested\ material\ [g]$

Tested Material and Measured Results

Methylene blue test of illite-kaolinite clay (clay mark KR) is described in this paper. There are 5 types of earth samples. Every type of earth samples includes KR clay, sand and distilled water. Sand and clay ratio is different for these types of earth samples. Methylene blue test is realized 3 times for every type of earth samples.

Table 1: Tested material

Mark of clay	Kind of clay	Clay/sand ratio [-]
KR	Illite-kaolinete	100/0
KR	Illite-kaolinete	75/25
KR	Illite-kaolinete	50/50
KR	Illite-kaolinete	25/75
KR	Illite-kaolinete	0/100

Table 2: Results of experiment

Samples	Number of measure	Amount of dye absorbed [g/kg]	Average amount of dye absorbed [g/kg]	Variance [(g/kg)^2]	Standard deviation [g/kg]
KR 100/0	I. measure	31.68	28.6	5.0	2.2
	II. measure	26.40			
	III. measure	27.72			
KR 75/25	I. measure	25.08	25.52	10.6	3.2
	II. measure	21.78			
	III. measure	29.70			
KR 50/50	I. measure	18.48	18.48	1.2	1.1
	II. measure	17.16			
	III. measure	19.80			
KR 25/75	I. measure	9.24	7.48	1.5	1.2
	II. measure	6.60			
	III. measure	6.60			
KR 0/100	I. measure	0.00	0	0	0
	II. measure	0.00			
	III. measure	0.00			

It was determined that clay-free sand did not absorb any dye. Absorption increases with increasing amount of clay in the sample. Tested earth samples and obtained results are showed in Table 1 and Table 2.

High values of variance, especially for KR 100/0 and KR 75/25 earth samples, is probably because of method of experiment execution.

Methylene blue test was adapted for our experimental measure compared the norm [9]. The main difference is amount of dose dye solution. In the norm is amount of 1 dose 2 ml of dye solution, but we adjusted dosage to reduce experiment time. After added dye solution was earth sample stirred for 1 minute regardless of amount of dye solution added. Example of dye solution dose is showed in Table 3.

We supposed that dependence of amount of absorbed dye on amount of clay would be linear. But, the experimentally obtained data give a nonlinear curve of this dependence (Fig. 3). Reason for obtained these results is probably in method of experiment execution too.

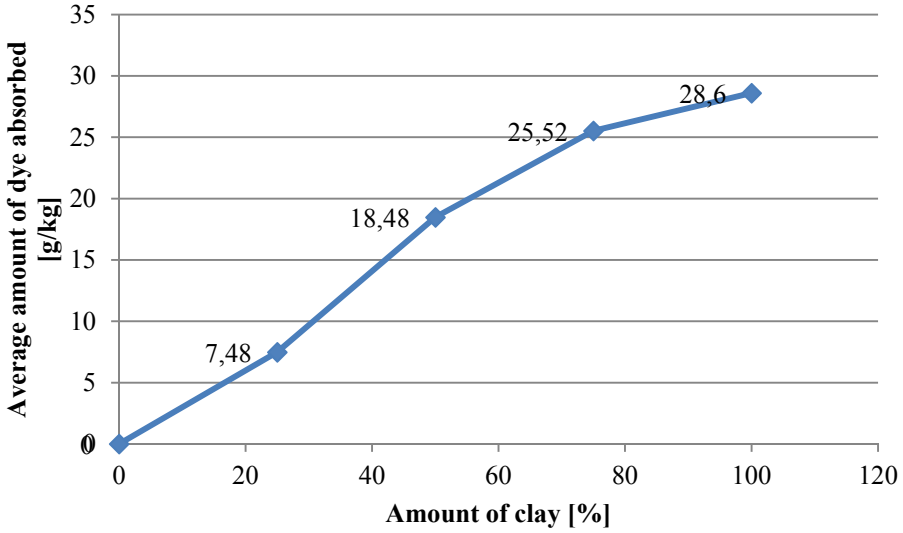

Fig. 3: Dependence of amount of absorbed dye on percentage amount of clay in earth samples

Table 3: Dose of dye solution into earth sample

Earth sample contains clay/sand ratio 50/50		
Number of dose	Amount of dye solution [ml]	Blue ring spreads around
1	20	NO
2	2	NO
3	2	NO
4	2	NO
5	2	NO
6	2	YES

Conclusions

Methylene blue test is good way for facilitating of composition diagnostician of unknown earth. This paper focuses on research of methylene blue dye absorption by illite-kaolinite clay. During this research it was found that the resulting values of absorbed dye are influenced by stirring time of earth sample with dye solution. Method of experiment must be adapted according to these findings for determining more accurate results.

It was determined with certainty that the clay-free material did not absorb methylene blue dye. Furthermore, it was determine that 1 kg illite-kaolinite clay absorbs approximately 29 g methylene blue dye.

Analysis methylene blue dye absorption of other kind of clay is necessary for using of methylene blue test in practical diagnostics of unknown earth.

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