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Moisture buffer, fire resistance and insulation potential of novel bio-clay plaster

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Abstract

The novel bio-clay plaster was formulated by adding hemp powder to clay plaster to improve the performance of buildings by moisture buffering the indoor climate, providing a fire insulation layer to encapsulate inflammable bio-based substrates and enhancing air tightness within the system by closing joints. The results showed the plaster formulation influences drying shrinkage, thermal conductivity, density and the moisture buffer value of the plaster. The thermal conductivity of the novel bio-clay plaster decreased with a decreasing proportion of Earthen Clay. The proportion of hemp powder had little effect on the thermal conductivity of the plaster, whereas the amount of hemp powder had a slightly effect on the moisture buffering property. The novel bio-clay plaster exhibited improved fire resistance. The detailed hygrothermal characterization using the measurement of moisture buffer value and thermal conductivity analysis indicated that the novel bio-clay plaster showed a good moisture buffering capability, even for small thicknesses, and low thermal conductivity, with the potential to develop industrially viable products.

Keywords: Bio-clay plaster, microstructure, moisture buffer value, thermal conductivity, fire resistance

Introduction

The use of bio-based insulation materials in the construction sector experienced an impressive expansion during the last decades with a growing awareness of the need to reduce the environmental impact of the construction sector [1-3]. Bio-based materials possess thermal performances similar or close to traditional insulation materials (mineral and synthetic), which are already commercialized [4]. There is a growing interest in the utilization of natural bio-aggregates as organic fillers and for the reinforcement of

lightweight bio-composites in sustainable construction [5-7]. The bio-composite of hemp concrete, which is composed of hemp shiv, as the plant aggregate, mixed with lime as binder and water, has been extensively studied. Because hemp shiv has low density, low thermal conductivity, high hygric properties and low processing costs [8-11]. The hemp concrete has a low thermal conductivity between 0.08 and 0.16 W/(m.K) depending on density, mix ratio and humidity [4, 12]. The hygric property of hemp concrete have been reported to be over $2 g/(m^2 \cdot RH)$, which represents excellent moisture buffering using the NORDTEST protocol [13-15]. Hemp concrete has the ability to regulate humidity inside buildings by absorbing and releasing water vapour molecules due to its low density and complex microstructure which improves the thermal comfort of buildings. Moujalled et al experimental and numerical evaluation of the hygrothermal performance of a hemp lime concrete building in a long-term case study. The results showed the hemp lime concrete has an excellent moisture buffer performance [16]. The large surface area of plastered walls and their exposure to the internal climate offers significant potential to passively assist in the regulation of the internal environment for improved occupant wellbeing. There has been growing interest in the use of exposed lime hemp plaster surfaces for the passive regulation of indoor temperature and humidity levels. A number of studies have demonstrated that the lime hemp plaster can enhance moisture buffering potential compared to many other conventional materials [17, 18]. In addition, the presence of clay can improve the mechanical and fire retardancy properties of the composite material. McGregor et al. reported that unfired clay masonry has a much higher moisture buffering value, which can be used to regulate indoor humidity [19]. However, clay is not good thermal insulator. Several studies have added hemp shiv to clay composites to reduce the thermal conductivity [20, 21]. The results of the experiments showed that hemp clay composites had a similar thermal conductivity to hemp concrete with the potential to use clay as an alternative to lime in order to reduce the environmental impact. Studies showed that clay plaster has significant potential to control indoor air quality due to its direct exposure to the indoor environment and large surface area [22, 23]. Natural clay plasters are breathable, non-toxic, release no volatile organic compounds (VOCs) into the atmosphere and are 100% biodegradable. The incorporation of bio-aggregates within clay plasters has potential to improve indoor environment quality through passive humidity buffering. In this study, novel bio-clay plasters containing hemp powder were studied in combination with different binders and mineral aggregates. The hygric and thermal properties of novel bio-clay plasters were measured. The fire resistance and hygric properties were tested. These novel bio-clay plasters are constituents of the ISOBIO panel system (ISOBIO European project) for internal wall construction. They can provide a fire and liquid water protective layer to the insulation system allowing high water vapour permeation and improved indoor comfort by moisture buffering. The industrial production of novel bio-clay plasters will be a step forward in introducing bio-based solutions into construction markets.

Materials and Experiment

Materials

It is well known that the physical properties of materials are influenced by manufacturing process including curing conditions, age of concrete, kind and content of binder. The standard for industrial produced clay-based plaster in Europe is the German standard DIN 18947 [24]. The standard is valid for all clay plaster with thicknesses greater than 3 mm. Such clay plaster can be used as under coat or upper coat on different substrates. The standard defines the constituents of clay plaster including allowed components and excluded components [25]. The components selected for this research, permitted by DIN 18947, were natural earthen clay (Claytec Company, Germany) and hemp powder (Cavac Company, France). The hemp plant was sourced and harvested in North West France. The hemp powder was produced by mechanical de-fibring, chopping, grading and de-dusting by the CAVAC cooperative (France). Its chemical and multi-physical properties are given in [11]. Other aggregate was sand (Claytec Company, Germany) and mineral binder was pumice (Claytec Company, Germany).

Mix proportion and composite production

The compositions were made by the mechanical mixing of wetted mineral materials and added hemp powder on an existing production line by Claytec (Germany). Nine different under coat and upper coat plasters were prepared according to DIN 18947. Three experimental mixtures (Clays 3, 5 and 6) were finally selected and tested, based on priorities including heat transfer and workability. The composition of the Bio-clay plaster is presented in Table 1. For reasons of comparison, a mineral addition commonly used to improve the fire resistance of mixes was added to experimental mixture Clay5. The "Thermosilit" fire resistant addition is an extruded volcanic silicate produced in Austria and is mainly used in lime plaster formulations. No additional water and no additional energy for drying are necessary for the production of the novel mixtures. The basic mineral ingredients clay, sand and pumice were mixed in a natural damped state as shown in Figure 1. Hemp powder was added to the mixing machine separately. For Clay3, Clay5 and Clay6, 10-15 volume% (i.e. 33, 35 and 37w% respectively) of mixing water was required for plaster application One batch, batch1, is produced to ensure equivalent rheology (table 1) and the second, batch2, is produced using the same water content.

The clay specimens were prepared in 140x160 mm (batch1) and 150×150 mm (batch2) moulds with a thickness of 13-70 mm. The cast clay panel was covered and left to set for 5 days in a conditioning room. Then the specimen was removed from the mould and left to cure for 28 days in the conditioning room (at 23 $^{\circ}$ C and 50 $^{\circ}$ RH for drying). Measurements of the thickness of specimens were reported to the nearest 0.1 mm. The mean density of the specimens is given in Table 2 for each batch.

Table 1: Bio-clay plaster composition

Туре	Binder	Bio-aggregates	Mineral	Additives
			aggregates	

Clay3	Earthen Clay 57w%	Hemp powder 4w%	Pumice 0-1: 28w% Sand 0-2: 11w%	
Clay5	Earthen Clay 6ow%	Hemp powder 2.5w%	Pumice 0-1: 25w% Sand 0-2: 10w%	Thermosilit 2.5w%
Clay6	Earthen Clay 50w%	Hemp powder 2w%	Pumice 0-1: 20w%	
			Pumice 0-3 : 20w%	
			Sand o-2: 8w%	



Clay5 Figure 1 Pictures of each mixed clay plasters

Table 2: Mean density (kg/m^3) of specimens.

Type Batch1		Batch2
Clay3	1399.86±6.01	1503.38±7.40
Clay5	1280.56 ± 8.74	1377.56 ± 3.76
Clay6	1287.98±14.79	1303.10 ± 4.48

Characterisation:

The microstructure of the bio-clay composite was characterised using a Scanning Electron Microscope (SEM) (JEOL SEM-6480LV, Tokyo, Japan). All images were taken at an accelerating voltage of 10 kV. The sample surfaces were coated with a thin layer of gold using an HHV500 sputter coater (Crawley, UK) to provide electrical conductivity, sending electrons to earth [26].

Thermal properties

The thermal conductivity was measured using an ISOMET 2114 electronic thermal properties analyzer (based on IEC EN 61010 standard) with a surface probe ranging from 0.04 to 1.0 W/(m.K). This measurement was based on analysis of the temperature response of the composites to heat flow impulses. Heat flow was excited by electrical heating of the resistor heater inserted into the probe, which was in direct heat contact with the tested

specimen. The specimens were pre-conditioned at 23 °C with a relative humidity of 50%. The measurements were made under ambient conditions in triplicate on 16 mm thickness specimens from batch2.

Moisture buffering value measurement

The hygric behaviour of the clay plaster was studied by measuring the moisture buffer value of the materials according to the NORDTEST method [27]. A set up was designed where specimens of each material were introduced in a climatic chamber. For batch1, three specimens were produced for each clay plaster and each thickness : 13, 40 and 70 mm. For batch2, three specimens are produced with a thickness of 16 mm. The required thickness recommended by Claytec for novel plasters is about 15 mm. The higher thicknesses allowed the study of the effect of thickness on performances.

The specimens were sealed on five out of six sides using aluminium film as shown in Figure 2 to ensure vapour exchange only occur through a single face of the specimen. The specimens (beforehand dried for batch1) were pre-conditioned at 23 °C at a relative humidity of 50%. The specimens were exposed to a daily cyclic variation of ambient relative humidity (8 hours at 75%RH and 16 hours at 33 %RH) in a climate chamber (Vötsch VC4060). The moisture buffer value was then calculated from moisture uptake and release with the formula:

 $MBV = \Delta m / (A (RH_{high} - RH_{low}))$ (1)

where MBV is the moisture buffer value (g/(m^2 . %RH)), Δm is the moisture uptake/release during the period (g) measured with a balance, A is the open surface area (m^2), RH_{high}/low is the high/low relative humidity level (%). For each material, the MBV was the average value of the tested specimens. Temperature and relative humidity were measured continuously with sensor SHT75 and with the sensor in the climatic chamber; the air velocity in the surroundings of the specimens ranged from 0.1 to 0.4 m/s for horizontal velocity and was lower than 0.15 m/s for vertical velocity. For some of the tests, a computer was connected to the balance and automatically recorded the weight of specimens.



Figure 2 Photo images of specimens for moisture buffer testing

The specific validated standard for clay plaster in Europe is DIN18947 [24]. According to DIN 18947, the highest water vapour adsorption class is III under the following test procedure. Water vapour sorption of clay plaster has to be determined with clay plaster specimens of at least 15 mm thickness. The test specimen is sealed (e.g. steel moulds) at five edges, to ensure that sorption can only get through one area. This testing area has a size of 1.000 cm². To reach the sufficient thickness of 15 mm thin layer clay plasters are applied on undercoat plaster of compatible material (according to producer advice) up to 15mm thickness for a valid testing specimen. After hardening of the surface of a clay plaster specimen, according to product specification, the air moisture sorption has to be determined. The specimen of the same size is conditioned in a climate chamber at 23 (± 2)°C temperature and 5% (± 5%) relative air humidity until a constant mass is reached. Constant mass is the results of two subsequent weightings at an interval of 24 hours that do not vary by more than 0.2 %. The humidity should be increased at constant air temperature up to (80 ± 5) % relative humidity. The increase in mass of the specimen after 0.5 h, 1 h, 3 h, 6 h and 12 h has to be determined. The measurements were performed with a balance having an accuracy of ≤ 0.01 g. The measured values were extrapolated over an area of one square meter. The classification was based on classes for Water Vapour Sorption (WVS) described in Table 3.

	Water Vapour Adsorption of Clay Plasters						
CLASSES		after					
	0,5 h	0,5h 1h 3h 6h 12h					
		g/m²					
WS I	≥ 3,5	≥ 7,0	≥ 13,5	≥ 20,0	≥ 35,0		
WS II	≥ 5,0	≥ 10,0	≥ 20,0	≥ 30,0	≥ 50,0		

Table 3 WVS-Classification DIN 18947

WS III	≥ 6,5	≥ 13,0	≥ 26,5	≥ 40,0	≥ 65,0
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Fire resistance

The fire behaviour of novel bio-clay plaster as a protective layer for bio-based substrates was studied according to the revision of the EN1995-1-2:2022 standard [28]. The fire resistance time t_{ins} of the bio-based elements depends on the sum of protection times (tprot,i) of each element which can be calculated by:

$$t_{ins} = \sum_{i=1}^{n-1} t_{prot,i} + t_{ins,n}$$
(2)

Where t_{ins} is the temperature increase time on the unexposed side of the wall, $t_{ins,n}$ plus the sum of the fall-off time of each layer $t_{prot,i}$. In the case of timber constructions with clay claddings, the delay in starting carbonization due to protection t_{ch} is calculated as follows:

$$t_{ch} = t_{prot, clay} = h_p - 7$$
 (3)

where h_p is the thickness of clay plaster in mm and results in t_{ch} minutes. The tests were tested on ISOBIO hemp panels (50 mm thickness, 200 kg/m³ made of hemp shiv glued with a bio-binder) with 10 mm novel clay plaster as protection layer compared to unprotected ISOBIO hemp panels.

The "Cross Section Method" was employed to calculate the charring depth d_{char} of timber after ignition until a critical mass loss occurs. Figure 3b shows the charring depth of timber clad with clay plaster and clay board (total thickness 40 mm) after fire test (CONE calorimeter). The unprotected charring rate β_0 of timber and other wooden building material is known and given. The protection layers delay the starting time (t_{ch}) of charring β_0 (green line in Figure 3a) and reduce the unprotected charring rate by a factor of k₂ (yellow line in Figure 3a). After fall-off or failure of the protection layer (t_f) the charring rate increases again but at a different rate. The protection layers can shift the charring curve down giving an extended period of resistance.



Figure 3 Charring depth of timber using Cross-Section Method (a) and image of timber cladded with clay plaster after fire test (b)

Drying shrinkage

In order to fully validating the final formulation of clay plaster to be used for the ISOBIO system, a set of tests were performed to evaluate the behaviour of the different formulations during the drying phase. Test specimens of batch1 were produced with 3 different thicknesses: 13, 40 and 70 mm.

Results and discussion

Morphology characterisation

Figure 4 shows the microstructure of Clay3 specimens. It is clear that sand and pumice particles were well mixed in the clay matrix and the clay binder was well adhered to the mineral aggregates (Figure 4a). Figure 4b clearly shows that the cellular hemp powder was well mixed with sand particles and the clay plaster well adhered to hemp powder. The pores in the hemp powder were not filled by clay plaster. The supporting information compares the microstructure of the three clay plaster compositions at increasing magnification from x50 to x2000. Results showed all specimens contained hemp powder, which was not always apparent in cross-sections.





(b)

Figure 4 Microstructure SEM images of Clay3 specimens (a) sand and pumice particles in a clay matrix and (b) cellular hemp powder in a clay matrix

Thermal properties

The measurement of thermal conductivity, thermal diffusivity, volume heat capacity at a mean temperature of 25° C are shown in Table 4 and Figure 5. It is evident that Clay3 specimens had the highest thermal conductivity of about 0.64 W/(m.K). Clay6 specimens had the lowest thermal conductivity of around 0.52 W/(m.K). This is due to the Clay6 specimens having the lowest density at 1306 kg/m³, which relates to the lower proportion of Earthen Clay binder (50%), while Clay3 and Clay5 have 57% and 60% of the Earthen Clay binder respectively. Clay3 and Clay5 had a similar volumetric heat capacity about 1.45×10⁶ J/m³.K., while Clay6 specimens had a volumetric heat capacity of 1.38 ×10⁶ J/m³.K. Thermal conductivity data for clay in the literature shows that it increases with an increase in density and moisture content, ranging from 0.8 to 1.0 W/(m.K) [29]. The results showed that the thermal conductivity of bio-clay plaster was lower than the thermal conductivity of bio-clay plaster did not decrease with an increase of the ratio of hemp powder, but remained proportionate to density, with the higher density specimens having higher thermal conductivity.

	Aver λ (W/(m.K))	St. Dev. (%)	cp (10 ⁶ J/m ³ .K)	St. Dev. (%)	Tmean (°C)	ΔТ (К)
Clay3-1	0.6509	0.092	1.4389	0.077	25.492	9.8313

Table 4 Thermal properties of Clay specimens measured by ISOMET

Clay3-2	0.645	0.122	1.4392	0.028	25.314	9.8436
Clay3-3	0.6372	0.09	1.4774	0.028	25.756	9.8362
Clay5-1	0.5839	0.214	1.4432	0.051	25.81	9.8393
Clay5-2	0.5975	0.147	1.5224	0.05	25.314	9.8112
Clay5-3	0.5918	0.292	1.4667	0.041	25.471	9.8401
Clay6-1	0.5265	0.138	1.3911	0.042	25.446	9.812
Clay6-2	0.5289	0.143	1.368	0.052	25.875	9.8464
Clay6-3	0.5174	0.137	1.4029	0.105	25.872	9.8568



Figure 5 Thermal conductivity of clay specimens as a function of density

Pumice reduced the density of novel mixture in combination with hemp powder below the level of standard undercoat plaster. Typical undercoat clay plasters have a density class of 1.8 and 1.9 or according to DIN 18947 equivalent densities ranging between 1.800 kg/m³ and 2.000 kg/m³. The bio-clay plaster was in the range between 1.400 kg/m³ and 1.600 kg/m³.

Thick layer clay plaster as Clay3, 5 and 6 display a thermal inertia behaviour. Figure 6 compares the volumetric heat capacity of different solid building materials. For example, the mixture Clay3 can be ranked at the same level as lime and sandstone. Derived from product data beton (concrete) and lime/cement mortar showed a 10-20% higher heat capacity compared to Clay3.



Figure 6 volumetric heat capacity of different solid building materials

MBV Measurements

Figure 7 shows the measured weight change per square meter response of one of the specimens (Clay3 - batch2) when it was subjected to cycles that varied the ambient humidity between 33% and 75% relative humidity for 16 and 8 hours respectively. The choice of stable cycles and the moisture uptake were marked in Figure 7.

All the specimens showed good moisture buffer values between 1.2 and 1.6 $g/(m^2.%RH)$ based on the NORDTEST project classification, shown in Figure 8. The results showed that the moisture buffer value was not a function of the bulk density of the materials. It depends on the moisture transfer and moisture storage capacity of the materials, which is highly linked to the porosity and chemical composition, pore size distribution and the pore structure (30, 31).

MBV values of Clay6 are lower than those of Clay3 and Clay5 plasters for both batches. It appears to be attributed to the lowest percentage of hemp powder and highest pumice content in Clay6. The Clay3 specimen showed the higher ratio of hemp powder, which

confers extra porosity and pore surface area. For all the tested thicknesses (Figure 9), the best MBV value is obtained with Clay5. The specimen thickness clearly more influences the evolution of MBV than density. According to Nordtest protocol, all tested plasters are classified as good hygric regulators (MBV ranging between 1 and 2 g/(m².%RH)). Figure 9 underlines that the MBV increases with thickness due to moisture penetration depth. However, this effect is not so much and it is thus not necessary to increase the thickness in order to increase MBV (from 10 to 70 mm, the MBV increases by 15% max). Few millimetres of plaster are sufficient to bring the main part of the performances. The moisture buffer value of the bio-clay plaster was higher than the classic building materials listed in the NORDTEST project. For example, the moisture buffer value of gypsum board is between 0.6 and 0.8 g/(m².%RH).



Figure 7 Determination of MVB from dynamic measurements. (Clay3 – batch2)



Figure 8 Moisture buffer value of clay specimens as a function of density



Figure 9 Moisture buffer value of clay specimens versus specimen thickness – batch1

WVS-Classification

The dotted lines in Figure 10 characterize the classification of moisture sorption properties with the lowest class as WS I and the highest class as WS III. The blue coloured curves

describes the performance of Clay3 plaster, the red curve presents the performance of Clay6 plaster, the green curve near WS III line represents Clay5 plaster (lowest curve). The time intervals and the conditioning of the specimens differ between the DIN and Nordtest standards. Despite differences in methods between DIN and Nordtest, the water vapour sorption performance value can be deduced from the Nordtest test and would be 50 g/m² after 8 h (Figure 7). This result gives good reasons to expect a WS III classification according to DIN 18947.



Figure 10 Water vapour adsorption of clay plaster according to DIN 18947

Fire resistance

The component additive method was used to test fire resistance conferred by clay plaster to the Cavac hemp panel according to mass loss, which indicated improved performance. Figure 11 shows that the mass loss of CAVAC hemp panels protected by a 10 mm plaster (grey, orange and blue curves), was significantly lower than unprotected hemp composites which showed a mass loss of > 70%. This can be seen as a tendency, which is relevant not only for direct application of clay plaster on rigid hemp insulation panels, e.g interior insulation but also for the ISOBIO system, where 15 mm clay plaster is applied. In addition, the Clay3 and Clay6 plaster layer showed a similar fire protection behaviour compared to Clay5 plaster layer, which had a 2.5% mineral fire retardant in the mixture.



Figure 11 Mass loss of Cavac hemp composites under fire test without and with 10 mm clay plasters 3, 5 and 6

Drying shrinkage

Figure 12 shows that some cracks appeared at the surface of the thinnest specimens (13 mm). A large detachment can be observed around the specimen for the other thicknesses. After weight stabilization in a conditionning chamber (23°C and 50% of humidity), specimen density is measured. As a shrinkage appears, all specimens are dimensioned to estimate accurately the apparent density and axial and volumetric shrinkages as shown in Table 5.



Figure 12 Images of bio-clay plaster specimens in the mould after drying test

	ΔL/L mm/m	ΔW/W mm/m	ΔT/T mm/m	ΔV/V dm³/m³	Density (kg/m³)
	L=Length	W=Width	T=Thickness	V=Volume	
Clay3 13 mm	8.6	0.4	202.6	211.7.0	1400.9 ± 0.9
Clay3 - 40 mm	16.4	18.7	45.2	80.2	
Clay3 - 70 mm	3.36	21.5	43.9	68.8	
Clay5 - 13 mm	8.9	3.6	189.6	202.1	1281.3 ± 0.5
Clay5- 40 mm	23.6	19.6	50.4	93.6	
Clay5 - 70 mm	3.0	17.6	47.5	68.1	
Clay6 - 13 mm	6.9	0.8	123.8	131.5	1289.1 ± 0.8
Clay6 - 40 mm	20.9	13.6	39.4	73.8	
Clay6 - 70 mm	-1.6	9.9	25.5	33.8	

Table 5 Drying shrinkage measurements of bio-clay plaster (mean of the three specimens)

Axial shrinkage is major for the thickness for all formulations. The high reduction was nearly 2 mm for the 13 mm thickness specimens. Volumetric drying shrinkage is presented in Figure 13. For the lower initial thickness (13 mm), the shrinkage is the same for the three types of clay formulations. The volumetric variation due to drying cannot be ignored (13 to 21 %) for all the formulations. The shrinkage decreases with the increase of the thickness of the specimen; but while Clay3 and Clay5 present a similar evolution, it is more marked for Clay6 formulation. This appears to be associated with the lower percentage of hemp powder and higher pumice content.



Figure 13 Evolution of the relative volume vs initial thickness for the three bio-clay plasters

Conclusions

In this work, the novel use of hemp powder as a component in clay plaster was studied on their specific mixture in combination with different binders and mineral aggregates. The bio-clay plaster has a great potential for a lightweight solution compared to the ready-touse coatings currently used for the protection of the thermal insulating composite system. It provides a fire and insulation protective layer to the insulation system while maintaining high water vapour permeation and improved indoor comfort through the provision of thermal inertia and moisture buffering. The thermal conductivity of bio-clay plaster was lower than the thermal conductivity of normal clay due to the presence of hemp powder. This presence reduced also the final density. The moisture buffer value of the bio-clay plaster was higher than classic building materials. This hygric properties increase with thickness and a thickness of 15 mm appears as a good compromise. The proportion of hemp powder had little impact on the thermal conductivity of the plaster, but a limited impact on the moisture buffering characteristics. Despite differences in methods between DIN and Nordtest, the water vapour sorption performance according to the Nordtest showed a good agreement with the measurement results according to DIN 18947. The fire test showed that the bio-clay plaster offered good fire protection to the ISOBIO panel. Shrinkage tests showed that the shrinkage decreases with the increase of the thickness of the specimen. The industrial production of novel clay-based plaster will be a step forward to market penetration in construction markets with developed and tested bio-based solutions.

Conflict of interest

The authors declare that they have no conflict of interest.

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