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A relevant characterization of Usumacinta river sediments for a reuse in earthen construction and agriculture

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ABSTRACT

In a context of global climate change and rational use of bio- and natural resources, including Usumacinta River sediments, valorisation of river sediments could become a socio-economic issue in the coming years and contribute to the sustainable development of the region (Project VAL-USES-, 2017).

This paper focuses on a simple and suitable characterisation including rheology, microstructure, mineralogy and geotechnical parameters of sediments for a reuse in earthen construction materials and structures and agriculture.

The implemented methodology is based on usual geotechnical and physicochemical testing plus rheology (viscosity and shear strength) and microstructure (pore size distribution, specific surface area) measurements.

The results confirm the existence of significant correlations between rheology, methylene blue value and geotechnical parameters, pore size distribution and mineralogy. This kind of correlations established allow to reduce the number of tests to be performed and make it possible to propose a more efficient characterisation with simple testing and measurement procedures.

The use of such correlations, may be possible to recommend beneficial uses of sediments in earthen construction, material and structures, and agriculture.

1. Introduction

VAL-USES, 2017 In a context of global climate change and rational use of bio- and natural resources, reusing and valorisation of river sediments could become a socio-economic issue in the coming years and contribute to the sustainable development of the region (**VAL-USES, 2017**).

A survey of the literature on the beneficial use of sediments as earthen constructions reveals that a considerable amount of effort has been devoted to this topic for more than 30 years, mainly in Europe and in the developed countries (**BRGM, 2017; Brils et al., 2014**). Most of these studies, the recent ones in particular, underline the possible reuse of this material for road construction (**Ben Slama et al., 2021; Dubois et al., 2009; Loudini et al., 2020**), brick production (**Gillot et al., 2021; Hussain et al., 2021; Jamshidi-Chenari et al., 2015; Peng et al., 2020; Slimanou et al., 2020; Torres et al., 2009; Yang et al., 2021**), and

component for other building materials (**Beddaa et al., 2020; Safhi et al., 2020; Lim et al., 2020; Yoobanpot et al., 2020**). However, there are various reasons why the development of dredged sediments remains limited. The greatest obstacles to their use come from the demand side, customers being suspicious about the material and fearing that it could be inferior and chemically contaminated (**Cappuyns et al., 2015**). In addition, because dredged sediments are a complex material by nature (often heterogeneous and in constant interaction with the other ecosystem components: water, biota, human), their beneficial use requires an accurate knowledge of their rheology, physicochemical geotechnical, mineralogy and microstructure properties (**BRGM, 2017; Rakshith and Singh, 2017**), which can be called the identity card of the sediment (**Levacher et al., 2006, 2011**). Sediment use in the ceramic industry, for instance, requires a precise understanding of the mineral and elementary components. The physical, chemical and mineralogical properties of sediments are also essential for use in the construction and

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geotechnical domains (Ben Allal et al., 2011; Goure-Doubi et al., 2015; Haurine et al., 2016; Mesrar et al., 2021). Moreover, organic matter, sodicity and salinity risks must be assessed for beneficial use in agriculture (EC, 2012; Martínez-Alvarez et al., 2018; USSLS and United States Salinity Laboratory Staff, 1954). Sediment type, water content and contamination, indeed, are serious obstacles to their technological and potential uses (Chahal et al., 2012; Chahal, 2013). Geological pollution, in particular anthropogenic pollution (Almeida et al., 2001; Buyang et al., 2019; Piazza et al., 2008; Yin et al., 2011) also accounts for their current limited use, particularly in an ever more restrictive regulatory environment. In this context, it is therefore essential to assess

the variety and quantity of pollutants and ensure that they do not exceed the maximum permissible legal thresholds (Journal officiel de la République Française, 2006; Journal officiel de la République Française, 2013; JORF, Journal officiel de la République Française, 2014).

Consequently, prior to any decision on potential valorisation issues, extensive characterisation of the sediments is necessary (Anger et al., 2015). To the authors' knowledge, no characterisation data for the sediments from the Usumacinta River are currently available. The only study available was conducted by Muñoz-Salinas and Castillo (2015) to examine climatic impacts on streamflow and sediment load variability within the Usumacinta River. Other studies involve deep sea sediments

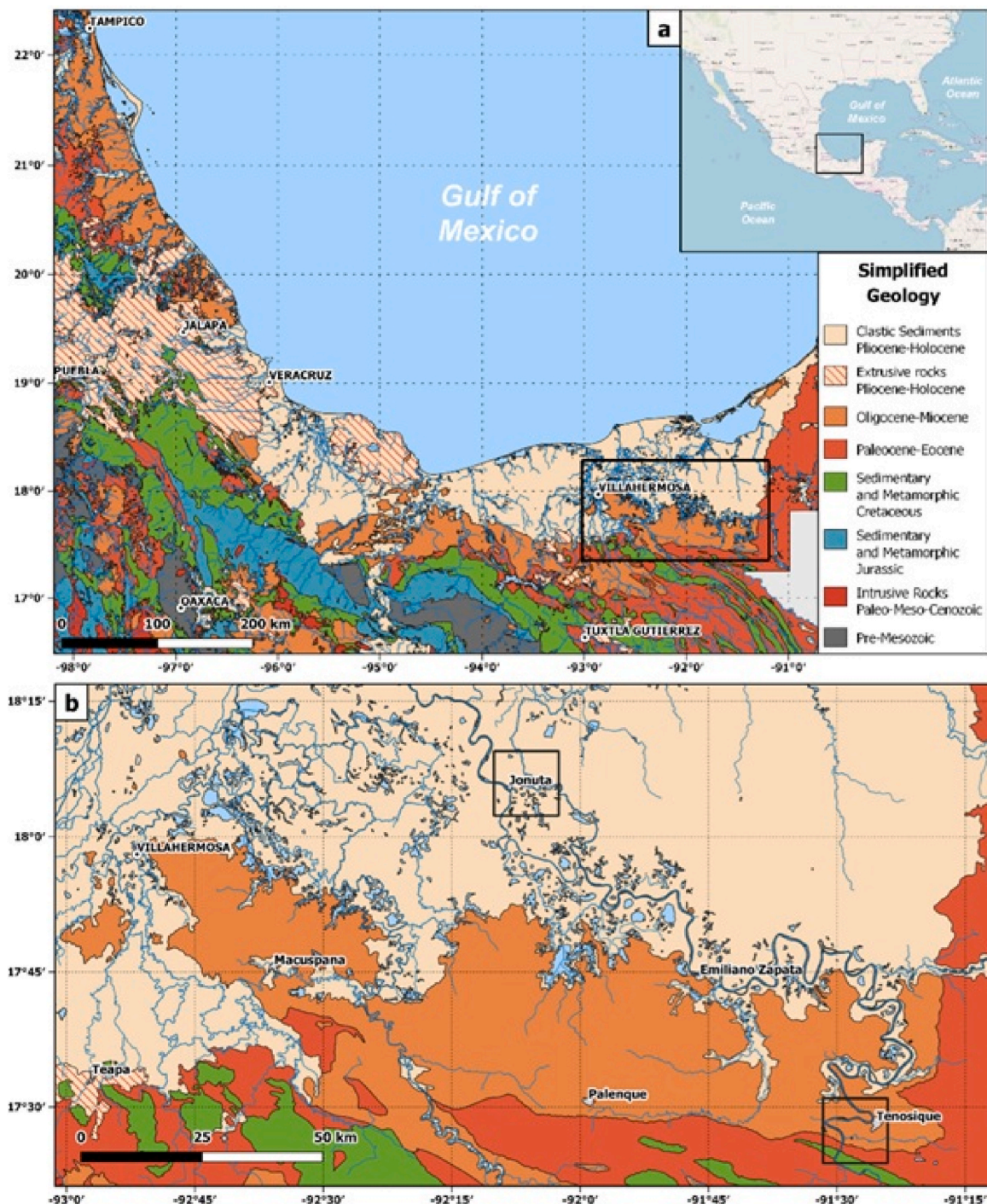


Fig. 1. (a) Geological map of Southern Mexico and (b) Tenosique and Jonuta in the federal state of Tabasco [Colour should be used for Fig. 1].

or sands from the Gulf of Mexico (Armstrong-Altrin et al., 2015a, 2015b; Armstrong-Altrin and Machain-Castillo, 2016; Ramos-Vázquez et al., 2017). They address mineralogy, geochemistry, radiocarbon ages, and distinguish between lithogenic and anthropogenic sources of heavy metals to investigate weathering effects and the origin of the sediments.

The present paper addresses different problems. First, although a few studies mention the Usumacinta River sediments, the sediment characterisation is not extensively documented, particularly to determine appropriate means of valorisation. For this purpose, accurate characterisation of the Usumacinta River sediments is therefore essential. Moreover, the Usumacinta River is a large river flowing through both agricultural and industrial (mainly downstream) areas. The sediment characteristics may then vary widely. The characterisation study carried out here focuses on a particular region along the river, the State of Tabasco. There has never been an engineering investigation conducted on these sediments, which should therefore raise interest within the scientific community. Since this paper also proposes appropriate means of valorising these sediments, the characterisation discussed here is especially comprehensive. Finally, thanks to this thorough characterisation, some correlations between the different properties have been identified and established.

Section 1 of this paper provides a general description of the study. The specific means and methods used to carry out the extensive and comprehensive characterisation of the Usumacinta River sediments are described in Section 2. The geotechnical, physicochemical, rheological, microstructural, and mineralogical properties of the sediments are then presented in Section 3. Finally, the results obtained and the correlations identified between the different properties are discussed in Section 4 and appropriate means of valorisation taking into account the specific properties of the Usumacinta River sediments are proposed. The correlations established aim to promote simple methods and measurement techniques that do not require the use of sophisticated and expensive equipment. The key findings are summarised in the conclusions.

2. Materials and method

2.1. Materials

The sediments have been collected from the Usumacinta River in the State of Tabasco, Mexico (Fig. 1a). The Usumacinta River is a 1000 km long river (620 miles). Its source is in the *Sierra de Los Cuchumatanes* in Guatemala, which means that some anthropogenic activities taking place in Guatemala may affect the hydrodynamics, the geomorphology and the composition of the downstream sediments. The Usumacinta River estuary is located in the State of Tabasco in Mexico. Each year, a large amount of sediment is deposited on the estuary floor (Muñoz-Salinas and Castillo, 2015).

Two sets of samples, collected from two different sites near the cities of Tenosique and Jonuta, are studied (Fig. 1b). The sediments have been collected upstream from the cities to avoid any pollution risk due to the nearby urban activities (organic matter, waste, craft and/or industrial waste). The first set of sediment samples is collected at the “Bocca del Cerro” near Tenosique, which forms a natural delimitation for sampling. Indeed, the presence of cliffs upstream from this point makes sampling technically impossible and limits sedimentary deposition (Fig. 2a). Downstream, closer to the Gulf of Mexico, the second sampling site near Jonuta is selected because the flow velocity of the river begins to slow down, which increases the accumulation of sediments (Fig. 2b). As a result of the different geomorphological features of the river at these two sampling locations, the sediments at each site have distinct characteristics based on their origin.

Samples have been collected at five different locations in Tenosique and four different locations in Jonuta. According to Armstrong-Altrin et al. (2015a), Jonuta’s sediments date back to the Pliocene-Holocene age and Tenosique’s ones to the Oligocene-Miocene age (Fig. 1).

The sediments are collected using portable sampling systems (*cone samplers, shovels and buckets*). They are labelled with the letter T (for Tenosique) or J (for Jonuta) and a number corresponding to the site where they are collected. The 2400 L of saturated samples are then stored in 34 hermetic barrels and shipped to France by ship before being



Fig. 2. Sampling locations: (a) Tenosique, near the Boca Del Cerro bridge; (b) Jonuta, on river banks [Colour should be used for Fig. 2].

distributed to three French laboratories in three different cities.

The tested sediments are considered inert and not hazardous except for Nickel whose highest concentration occurs to be 5 times the S1 threshold level (Table 1).

Nickel has properties of insolubility and stability in aqueous media (Cappuyns and Swennen, 2006). Nevertheless, several possibilities of nickel processing exist in the literature: chemical extraction, bio-leaching, electroreclamation and supercritical fluid extraction (Babel & del Mundo Dacera, 2006). These are cost-effective methods for economy, having an impact on the environment that cannot be neglected. Phytoremediation seems to be an interesting alternative treatment solution (Yan et al., 2020), that meets the targeted socio-economic and environmental contexts, and then Usamacinta River sediments can be beneficial in earthen construction and in agriculture.

2.2. Testing methods

Many tests were carried out on the sediments to determine their rheological, geotechnical, physicochemical, microstructural, and environmental properties, as illustrated in Fig. 3.

2.2.1. Rheological analysis

The rheological analysis consists of measuring the viscosity of the sediment samples as a function of the shear rate. The measurements are carried out using the MCR 52 rheometer developed by the Anton Paar Ltd.

2.2.2. Geotechnical analysis

A geotechnical analysis consisting in the determination of grain size distribution, Atterberg limits, and presence of clay using standardized testing protocols is carried out. Testing methods and standards are summarised in Table 2.

2.2.3. Physicochemical characterisation

Several tests were performed to determine the physicochemical properties including organic matter content, pH values, electrical conductivity, CaCO₃, cation exchange capacity, sodium adsorption ratio and mineralogy of the sediments.

The organic matter content is obtained according to Standard XP P94-047, 1998. This protocol consists of determining the mass loss of a sample, previously dried at 50 °C and sieved at 2 mm, after calcination in an oven at a temperature between 450 °C and 500 °C for 3 h.

Table 1
Heavy metal and pollutant levels in Tenosique and Jonuta sediments.

Heavy metal and pollutant	unit	S1 level	T1	T2	T3	T5	T6	J1	J3	J4	J5
Arsenic (As)	mg/kg D.M.	30	2.73	2.31	1.79	3.02	2.75	2.81	2.71	5.19	2.18
	sample/S1 level	–	0.091	0.077	0.06	0.101	0.092	0.094	0.09	0.173	0.073
Cadmium (Cd)	mg/kg D.M.	2	0.4	0.4	0.4	0.4	0.4	0.44	<0.4	<0.4	<0.4
	sample/S1 level	–	0.2	0.2	0.2	0.2	0.2	0.22	<0.2	<0.2	<0.2
Chromium (Cr)	mg/kg D.M.	150	108	76.1	23	98.6	96.3	103	99.8	131	96.5
	sample/S1 level	–	0.72	0.507	0.153	0.657	0.642	0.687	0.665	0.873	0.643
Copper (Cu)	mg/kg D.M.	100	17.2	10.8	11.4	15.4	14.5	18.3	15.8	20.5	15.8
	sample/S1 level	–	0.172	0.108	0.114	0.154	0.145	0.183	0.158	0.205	0.158
Mercury (Hg)	mg/kg D.M.	1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1
	sample/S1 level	–	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1
Nickel (Ni)	mg/kg D.M.	50	164	107	44.5	147	141	168	157	256	161
	sample/S1 level	–	3.28	2.14	0.89	2.94	2.82	3.36	3.14	5.12	3.22
Lead (Pb)	mg/kg D.M.	100	9.49	6.53	8.18	9.97	7.78	16.5	9.2	11.3	8.68
	sample/S1 level	–	0.095	0.065	0.082	0.1	0.078	0.165	0.092	0.113	0.087
Zinc (Zn)	mg/kg D.M.	300	46.6	36.8	42.7	42.4	40.8	71.4	45.9	40.2	43.8
	sample/S1 level	–	0.16	0.12	0.14	0.14	0.14	0.24	0.15	0.13	0.15
PCB total	mg/kg D.M.	0.68	<0.001	<0.001	<0.001	<0.001	<0.001	<0.001	<0.001	<0.001	<0.001
	sample/S1 level	–	<0.001	<0.001	<0.001	<0.001	<0.001	<0.001	<0.001	<0.001	<0.001
PAH total	mg/kg D.M.	22.8	0.11	0.012	0.2	0.049	0.025	0.071	0.12	0.014	0.031
	sample/S1 level	–	0.005	0.001	0.009	0.002	0.001	0.003	0.005	0.001	0.001

D.M. = dry mass.

S1 level is given by order appended to article R. 214–1 of the French Decree (August 9, 2006).

CaCO₃ is obtained using a calcimeter according to Standard NF P94-048, 1996. The pH and the electrical conductivity values are measured simultaneously according to Standards NF ISO 10390 and NF ISO 11265. The mineralogical analyses are carried out using X-ray diffraction (XRD) on samples, initially sublimated and crushed using a ring grinder. The cation exchange capacity (CEC) is obtained from the results of the Methylene Blue tests (Table 2) or directly measured using the spectrophotometer JENWAY 73 series, according to Standard NF EN ISO 23470 (Table 3). The Sodium Adsorption Ratio (SAR) is calculated from (Na⁺), calcium (Ca²⁺) and magnesium (Mg²⁺) values. The tests conducted to obtain Na, Ca, and Mg concentrations are carried out using Inductive Coupled Plasma/Atomic Emission Spectrometry (ICP/AES) according to Standard NF EN ISO 11885 (Table 4).

2.2.4. Environmental analysis

The environmental analysis focuses on the assessment of the pollution level and the determination of Polycyclic Aromatic Hydrocarbons (PAHs), Polychlorinated Biphenyl (PCBs), heavy metals, volatile compounds, chlorophenols and glyphosates contained in the sediments and of chloride, nitrate, nitrite and sulphate contents in the river water. The sample preparation consists of drying the sediments at 40 °C, sieving the samples using a 2-mm sieve, filtering them through a 0.45-µm screen and, finally, dissolving them in KCl. All the tests conducted are described in Table 4.

2.2.5. Microstructural analysis

The microstructural analysis is conducted to determine the specific surface area (SSA), the N₂ adsorption isotherm and the pore size distribution. The specific surface area (SSA) of the nine samples, corresponding to the nine sampling sites, is obtained using two different methods. First, SSA is directly deduced from the Methylene Blue test results. Then, SSA is determined according to N₂ adsorption. The tests are carried out on self-weight consolidated samples using the TriStar II Plus apparatus. The procedure is based on the Brunauer, Emmett and Teller's method (BET method) (Petersen et al., 1996; Dogan et al., 2007) for the SSA determination and on the Barrett, Joyner and Halenda's method (BJH method) for the determination of N₂ isotherm and pore size distribution. The samples are first frozen and then sublimated for 48 h to preserve the pore structure of the porous media. Residual air trapped within the samples is removed by heating at a temperature of 50 °C prior to nitrogen injection. Nitrogen is injected and the volume of adsorbed gas is measured (Cheng and Heidari, 2018). The adsorption

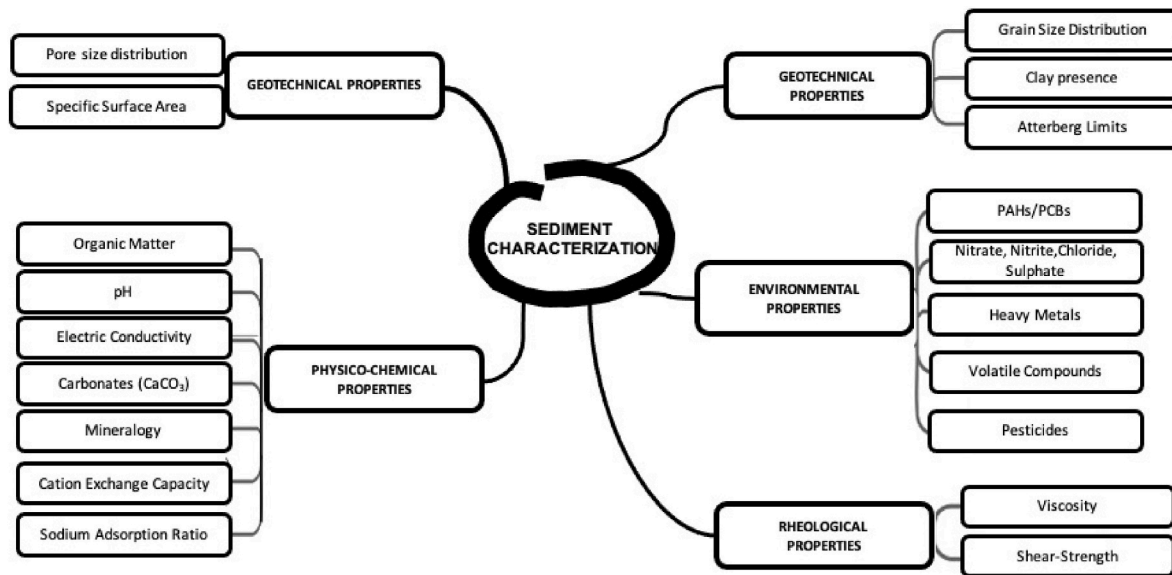


Fig. 3. Usumacinta River sediment characterisation test scheme.

Table 2

Testing methods and standards used to characterize the geotechnical properties of the sediments.

Characteristics	Tests	Standards
Grain size distribution	Laser granulometry	
Atterberg limits (LL, PL)	Fall cone test (LL)* Casagrande test (LL)* Rolled thread method (PL)	NF EN ISO 17892-12, 2018 NF EN ISO 17892-12, 2018 NF EN ISO 17892-12, 2018
Clay presence	Methylene Blue Test	NF P94-048, 1996

Note: LL: Liquidity Limit; PL: Plasticity Limit; * Liquidity limit (LL) is measured using two different methods (fall cone test and Casagrande test). The final value of the liquidity limits was the mean value of the results of the two methods.

Table 3

Testing methods and standards used to characterize the geotechnical properties of the sediments.

Characteristics	Tests	Standards
Organic matter	Calcination	XP P94-047, 1998
CaCO ₃	Calciometer	NF P94-048, 1996
pH and EC	multiparameter probe	NF ISO 10390 and NF ISO 11265
Mineralogical analyses	X-Ray Diffraction (XRD)	
CEC	Spectrometry	NF EN ISO 23470

isotherm is then obtained. Since the nitrogen temperature and pressure are known, the specific surface area is determined by a formula relating the volume of the adsorbed gas to the gas pressure.

3. Results

The results present the rheological, geotechnical, physicochemical, microstructural, and environmental properties of the Usumacinta River sediments obtained using the above-described methods.

3.1. Rheological properties

Fig. 4 displays the viscosity vs. shear rate curves computed from the results of the sediments tested at the liquidity limit. Regarding the Tenosique samples, two groups of sediments behave differently: (T1, T2) sediments have a higher viscosity than (T3, T5). The viscosity of the

Table 4

Testing methods and standards used to characterize the geotechnical properties of the sediments.

Pollutant examined	Tests	Standards
PAHs/PCBs	Gas chromatography/Mass spectrometry Liquid chromatography/Mass spectrometry	XP X33-012, 2000
Heavy metals	Inductively Coupled Plasma/Atomic Emission Spectrometry (ICP/AES)	NF EN ISO 11885
Volatile Organic compounds	Static Headspace Gas Chromatography/Mass spectrometry (SH - GC/MS)	NF EN ISO 22155
Chlorophenols	Gas chromatography/Mass spectrometry	XP X33-012, 2000 ^a
Glyphosates	Liquid chromatography/Mass spectrometry	XP X33-012, 2000
Nitrates, nitrites, chloride, sulphates in water	Spectrophotometry	NF ISO 15923-1

^a Note: The tests have been performed according to Standard XP X33-012, 2000 in 2019 before it was cancelled in 2020.

Jonuta sediments, on the other hand, is similar for the four samples and slightly lower than the viscosity observed for the Tenosique sediments. The viscosity of sediments can be correlated to the value of MBV; the lower viscosity is attributed to higher clay content (Table 5).

3.2. Geotechnical properties

Table 6 summarizes the particle size distribution of all the samples illustrated in Fig. 5. The samples are mainly composed of silty sediments with a silt fraction ranging from 41% to 85%. The percentage of fine sand varies widely between 3.4% and 54.2%. Sediments T3 and J4 which have the lowest sand fraction also have the highest clay content. In general, the clay fraction is low (<15%) compared with silt and sand ones (except Samples T3 and J4).

Critical water contents, i.e., the Atterberg limits, and presence of clay are summarised in Table 7. Based on the grain size, Methylene Blue Values (MBV) and Plasticity Index (PI) values obtained, the sediments are classified according to the French GTR guidelines (NF, 1992). T1, T2, T3 and T6 are classified as A1 (low plasticity silt, loess, alluvial silt, marginally polluted fine sand, low plasticity) while T5, J1, J3, J4 and J5

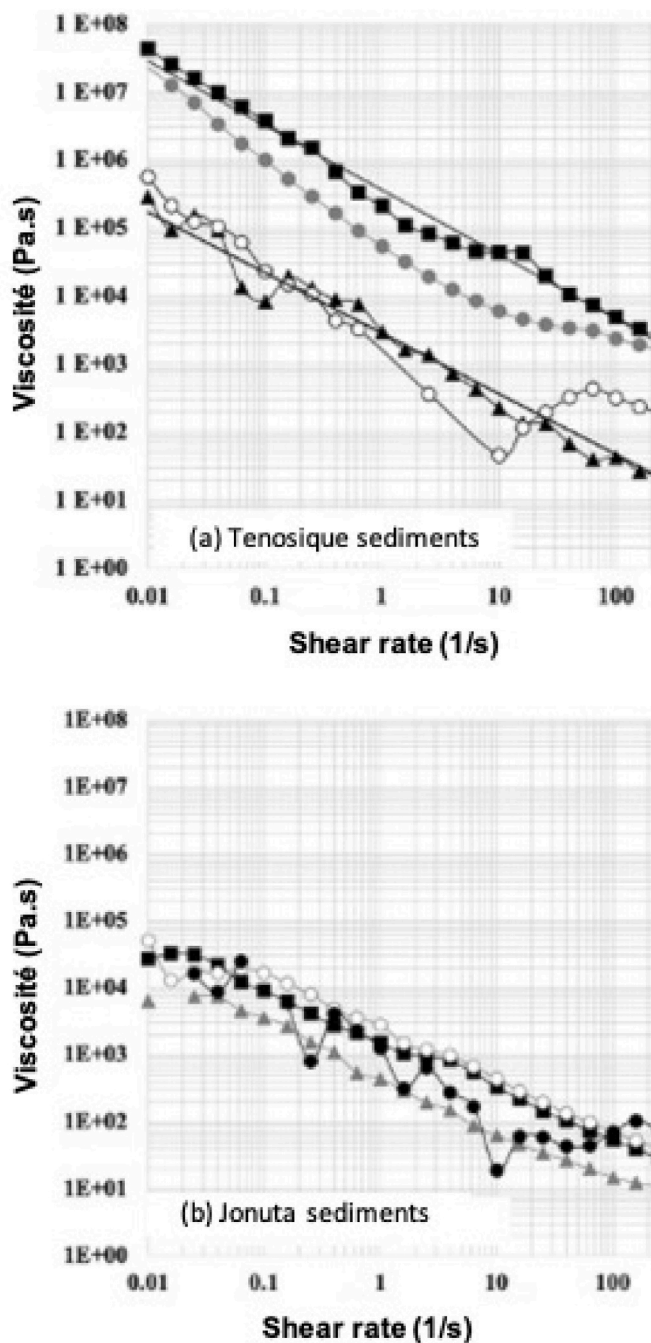


Fig. 4. Rheological behaviour of: (a) Tenosique and (b) Jonuta sediments.

Table 5

Viscosity from low to high values for T samples (Tenosique) and J samples (Jonuta) versus MBV values.

Viscosity order of sediments	T1	T2	T3	T5	J3	J1	J5	J4
MBV	2.3	1.8	3.1	5.7	3.4	4.6	4.2	7.7

are classified as A2 (fine clayey sands, silts, clays and low-plasticity marls).

3.3. Physicochemical properties

Organic matter, pH, electro-conductivity and CaCO₃ levels are

Table 6

Grain size distribution of Usumacinta River sediments.

	Clay (<2.5 μm) %	Silt (2.5–25 μm) %	Sand (>25 μm) %
T1	5.80	48.10	46.10
T2	3.67	50.93	45.40
T3	11.80	84.80	3.40
T5	7.56	63.44	29.00
T6	4.72	41.08	54.20
J1	6.23	51.67	42.10
J3	6.17	47.13	46.70
J4	13.10	79.90	7.00
J5	6.39	55.81	37.80

presented in Table 8. Although the organic matter content of the sediments does not exceed 6%, it is, however, higher than the limit of 3%–4%, beyond which material properties can be modified (Bennert et al., 2000). All the sediment samples have similar pH level and are slightly alkaline. Even if the electro-conductivity values appear scattered, the sediments have a low saline risk (USSLS and United States Salinity Laboratory Staff, 1954). Only Sample J1 presents a medium saline risk. The CaCO₃ level varies considerably from 0 to 9.4%.

3.4. Mineralogical properties

The mineral composition is summarised in Table 9. Sample T3 is not included in this table because the X-ray diffraction results obtained for this sample exhibit a halo, which is a characteristic feature of an amorphous phase and accounts for the absence of minerals. In the remaining eight samples, carbonate and silicate group minerals are identified. Clays, micas, feldspars, cristobalite and quartz form the silicate group. The clay identified in the sediments is composed of montmorillonite, illite and vermiculite. In all the samples (except J4), quartz is the main mineral element (between 43% and 52%), which is a usual characteristic of sandy dredged sediments. J4, which has the lowest sand proportion (only 7%, refer to Table 6) is the sample with the lowest quartz content (21.4%, see Table 9) but also the sample with the largest proportion of clay minerals, in accordance with its grain size distribution (13.1% of grain <2.5 μm in Table 6).

The cation exchange capacity is first calculated from the results of the MBV tests using Equation (1) proposed by Cokca and Birand (1993) as:

$$CEC_{MBV} = \frac{100}{W_m} V_{cc} N_{mb} \tag{1}$$

with $N_{mb} = \frac{\text{weight of methylene blue (g)} \times \frac{100-X}{100}}{320}$.

And where CEC is the cation exchange capacity (meq/100 g clay), W_m is the weight of clay specimen (g), V_{cc} is the volume of methylene blue titrant (mL), N_{mb} is the normality of the methylene blue substance (m_{eq}/mL) and X is the moisture content of the methylene blue substance (%). CEC_{MBV} values obtained range between 6 and 24 meq/100 g clay. J4 (24 meq/100 g clay) is the only sample with a CEC_{MBV} value above 20. T1, T2 and T6 have a CEC_{MBV} value below 10, the other samples ranging between 10 and 20 meq/100 g (Table 10). The CEC results calculated using the MBV test method are underestimated compared with the measured CEC results (Table 10).

The sodium adsorption ratio (SAR) is determined using Equation 2 for which the amount of sodium (Na) in relation to the calcium (Ca) and the magnesium (Mg) concentrations in the water recovered from the saturated soil paste must be known. Na, Ca, Mg components are measured using Inductively Coupled Plasma/Atomic Emission Spectrometry (ICP/AES). We write: $SAR = Na / \sqrt{\frac{1}{2}(Ca + Mg)}$ (2)

The concentrations of magnesium and calcium are around 20,000 mg/kg D.M. and 50,000 mg/kg D.M., respectively, with the exception of T3, for which Mg and Ca concentrations are ten times lower. Conversely,

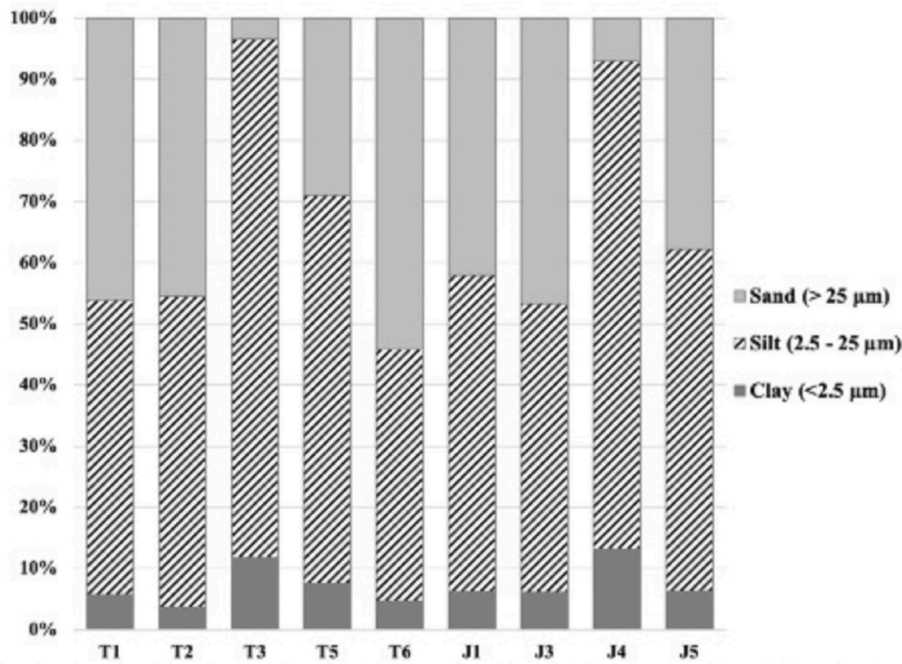


Fig. 5. Graphical representation of Usumacinta River sediment particle size distribution.

Table 7
Geotechnical properties and soil classification.

Sediment	% passing at 80 µm %	MBV	Atterberg Limits			GTR classification
			LL	PL	PI	
T1	75	2.3	39.0	31.0	8.0	A1
T2	42	1.8	33.0	22.9	10.1	A1
T3	89	3.1	49.0	36.7	12.3	A2/A1
T5	85	5.7	40.9	25.7	15.2	A2
T6	62	2.5	55.0	(-)	(-)	A1
J1	98	4.6	52.8	33	19.8	A2
J3	85	3.4	43.5	30.7	12.8	A2
J4	94	7.7	62.0	43.9	18.1	A2
J5	98	4.2	45.6	33.8	11.8	A2

Note: PI = LL - PL, with PI: Plasticity Index; LL: Liquidity Limit; PL: Plasticity Limit.

Table 8
Physicochemical parameters.

Sediment	Organic matter %	pH	Electro-conductivity µS/cm	Carbonates CaCO ₃ %
T1	4.7	8.5	135.7	9.4
T2	3.5	8.6	95.5	5.2
T3	5.0	8.3	70.1	0.9
T5	3.6	8.5	137.2	(-)
T6	3.5	8.6	102.2	7.3
J1	4.5	8.3	308.5	8.4
J3	5.1	8.4	240.0	7.8
J4	6.0	8.6	263.0	4.5
J5	4.9	8.4	52.5	7.2

sample T3 sodium concentration is four times higher than in the other sediment samples. Consequently, SAR value of sample T3 (8.30) is ten times that of the other samples, which ranges between 0.61 and 1.24 (Table 11).

3.5. Microstructural properties

The specific surface area (SSA) is determined first from the Methylene Blue Value (MBV) using the relationship established by Santamarina et al. (2002):

$$SSA_{MBV} = \frac{1}{319.87} * \frac{1}{200} * (0.5N) * A_v * A_{mb} * \frac{1}{10} \quad (3)$$

where 319.87 corresponds to the molecular weight in g/mol of the methylene blue chemical formula, A_v is the Avogadro number, A_{mb} is 130 Å, which corresponds to the area covered by one methylene blue molecule and N is the number of methylene blue increments added to the sediments (Santamarina et al., 2002).

The specific surface values obtained from MBV range between 44 and 189 m²/g as shown in Table 12. T2 exhibits the lowest value for T2 and J4 the highest one. Samples T1, T2, T3, T6 and J3 all have an SSA value below 100. On the other hand, T5 is the only Tenosique sediment sample with a SSA value above 100 (141 m²/g).

Then, specific surface area (SSA), pore size distribution and nitrogen gas (N₂) adsorption isotherms are determined using BET and BJH methods. All the adsorption isotherms present the same pattern with a hysteresis that may be explained by the bottle ink phenomena between aggregates (Dogan et al., 2007). This hysteresis appears when a material has mesopores (between 2 and 50 µm). The hysteresis height reflects the material's affinity with water. It is calculated by subtracting the amount of N₂ adsorbed during desorption and absorption, where the hysteresis is the largest. The volume of N₂ adsorbed by samples T3 and T5 is twice higher than for the three other sediment samples from Tenosique (Fig. 6a). The values of the adsorption isotherm of the Jonuta samples are very similar, with the exception of sample J4 (Fig. 6b). The hysteresis average of all the sediment samples studied is 2.46 cm³/g (Table 13).

The sediments whose hysteresis is high also have larger SSA values. These sediments are the easiest to moisture and the most difficult to dry. Of all the sediments studied, J4 presents the highest affinity with water in relation to its highest hysteresis value (Table 13).

The specific surface area of samples T1, T2 and T6 is approximately 21 m²/g, while samples T3, J1, J3 and J5 have SSA values ranging

Table 9
Mineralogical components (in %).

Sedi- ment	Silicates								Carbonates		Non identified minerals	
	Clays			Mica	Feldspars			Cristo-balite	Quartz			
	Mont-morillonite	Vermi-culite	Illite	Biotite	Ortho-class	Anortho-class	Albite					
T1	2.6	3.1	5.1	3.1	4	5.3	3.2	2.5	43.7	7.1	18.4	1.9
T2	4	2.2	2.2	2.7	3.8	5.9	3.9	3.2	52.0	4.8	14.1	1.2
T5	5.1	3	5.4	2.6	3.1	5.4	3.2	1.7	50.5	3	9.7	7.3
T6	3.4	3.1	2.3	2.8	4	9.2	3.8	2.6	48.7	3.9	14.7	1.5
J1	4.5	2.8	2.3	2.7	3.8	7.1	3.1	2.8	45.2	11.7	12.3	1.7
J3	3.6	2.9	2.6	2.9	4.2	5.9	3	2.9	48.4	5.6	16.5	1.5
J4	10	17.1	6.4	7	5.3	9.6	4.3	1.6	21.4	2.2	10.1	5
J5	3.6	3	3.1	3.4	5	8.3	4.5	2.8	44.6	6.2	14	1.5

Table 10
CEC obtained using the Methylene Blue Value method and direct measurement procedures.

	T1	T2	T3	T5	T6	J1	J3	J4	J5
CEC _{MBV} (m _{eq} /100 g clay)	7	6	10	18	8	14	11	24	13
CEC _{measured} (m _{eq} /100 g clay)	15.5	13.3	19.4	25	18.3	24.5	20.2	35.7	20.7

Table 11
Elementary chemical for Sodium Adsorption Ratio calculation.

	T1	T2	T3	T5	T6	J1	J3	J4	J5
Ca (mg/kg D.M.)	61,600	47,900	3340	39,200	50,600	58,500	48,200	59,800	51,900
Mg (mg/kg D.M.)	22,600	19,100	3540	18,100	21,600	19,000	20,100	15,400	18,900
Na (mg/kg D.M.)	138	111	487	123	135	183	129	241	168
SAR (-)	0.67	0.61	8.30	0.73	0.71	0.93	0.70	1.24	0.89

D.M.: dry mass.

Table 12
SSA obtained using the Methylene Blue Value method.

	T1	T2	T3	T5	T6	J1	J3	J4	J5	average
SSA _{MBV} (m ² /g)	57	44	77	141	63	112	85	189	104	96.9

between 25 and 30 m²/g. Two results differ: T5 and J4, with a specific surface area of 36.2 m²/g, and 54.5 m²/g, respectively (Table 13). So, sample J4 has the highest affinity with water with a hysteresis value 1.6 times higher than the mean hysteresis value, and a SSA value almost twice higher than the mean SSA value.

Fig. 7 displays the pore size distribution of the sediments obtained from the N₂ adsorption isotherm tests and with the BHJ method. The pores smaller than 25 Å predominate. Samples J4 and T5, which have the highest SSA values, are also the sediments with the highest number of micropores smaller than 25 Å.

4. Discussion

4.1. Use of sediments for earthen construction materials

The beneficial uses explored for the Usumacinta River sediments concern earthen materials, including brick production.

The use of sediments can be considered in sustainable construction at a local level and for lightweight buildings. This includes the construction of non-load bearing walls for lightweight buildings for agricultural and craft purposes. The problem raised concerns the determination of the key parameters for the assessment of the suitability of the sediments to meet the requirements for use as bricks. Workability and plasticity are (Tables 5 and 7), here, the two main parameters used to evaluate this suitability of sediment bricks.

Classification of soil properties suitable for brick production is carried out using the Winkler diagram, which is based on the industrial data

relating the particle size of the mixtures to different brick applications (Fig. 8). On each of its three sides, the equilateral triangle gives the percentages of the grain sizes for clay, silt and sand. The sediments studied are placed on the Winkler diagram, using the values summarised in Table 6. The latter has almost 100% of the grains with a particle diameter greater than 25 µm is placed on the lower-right corner of the diagram. The points, however, are located outside the areas identified as appropriate for brick production and the corresponding sediments, therefore, must be reinforced or stabilized before use.

The first beneficial use for the Usumacinta sediments to be examined is terracotta production (crude or reinforced bricks). Some already available diagrams provide some data on the optimal or acceptable suitability of the sediments studied for such use. The Atterberg limits can be plotted on the Casagrande diagram in the plane of the plasticity index as a function of the liquid limit and in the plane of plasticity limit and plasticity index. The Casagrande diagram (Gippini, 1969) in Fig. 9a defines the extrusion areas. The samples located in areas A and B have optimal and adequate characteristics for extrusion. The workability diagram of Bain and Highley (1979) in Fig. 9b defines two workability areas for both brick and pottery production: workability is optimal inside rectangle C and good inside rectangle D (Sena da Fonseca et al., 2013).

Samples T1 and T5 that are located inside area D are, with acceptable shrinkage and therefore, suitable for the production of bricks and pottery. These two samples are located within the A-frame in Fig. 9a, meaning optimal characteristics for extrusion. In this figure, sample T3 is close to the A-frame limit but remains outside the workability area as

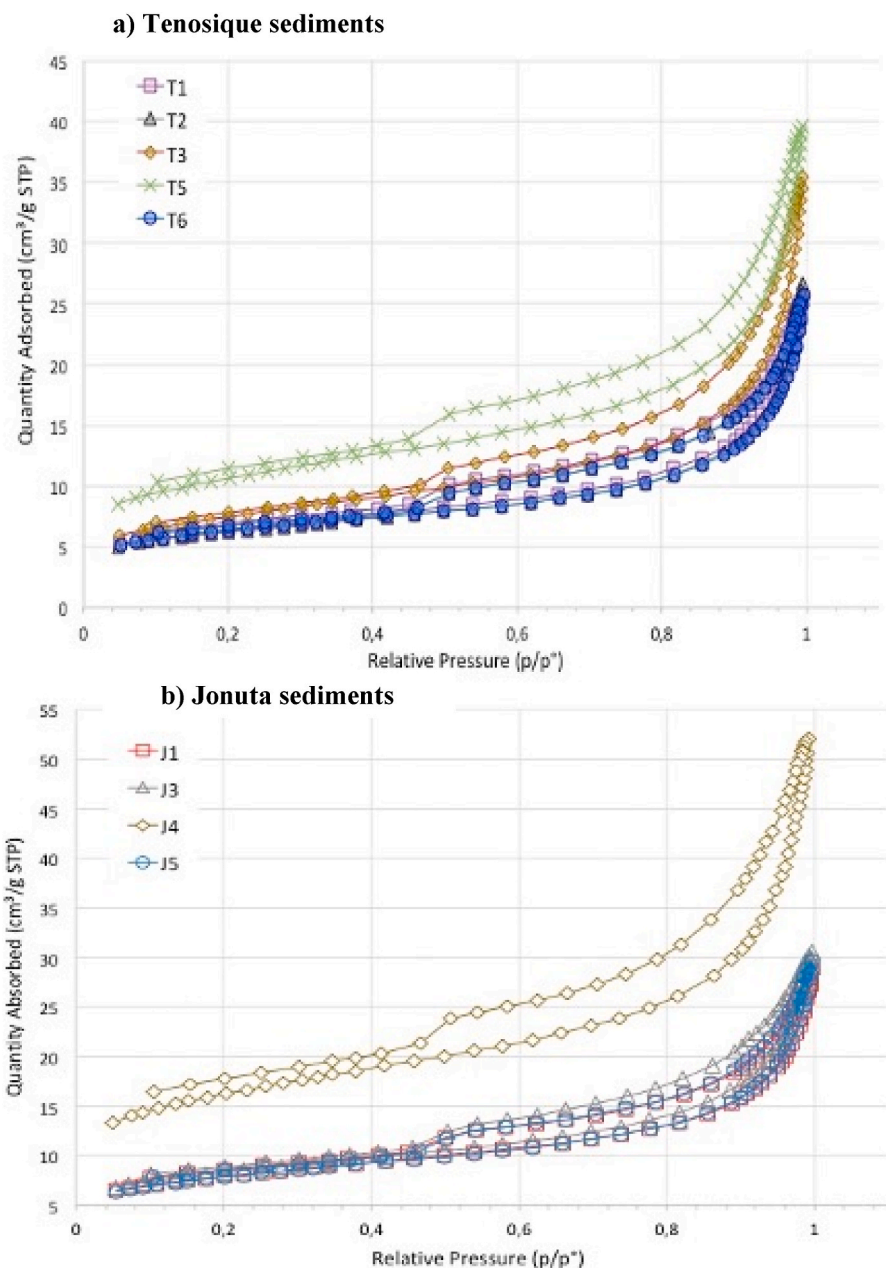


Fig. 6. Adsorption isotherms of: (a) Tenosique and (b) Jonuta sediments.

Table 13

SSA obtained using the BET method.

	T1	T2	T3	T5	T6	J1	J3	J4	J5	Average
Hysteresis (cm ³ /g, STP ^a)	2.26	1.93	1.99	2.75	1.94	2.25	2.73	3.98	2.36	2.46
SSA _{N2} (m ² /g)	21.9	21.14	25.8	36.2	21.3	26.9	28.2	54.5	26.7	29.2

^a STP: standard temperature of 0 °C at 1-atm pressure.

does sample T2 in Fig. 9b. For this reason, these two samples are not suitable for the production of bricks.

On the other hand, the Jonuta sediments when placed in the same extrusion (Fig. 10) and workability (Fig. 10) diagrams are outside areas D and C regarding workability. Although some of them are close to or inside the A-frame (J5), they are suitable in a limited way for the production of bricks, without stabilization or strengthening.

In conclusion, based on Casagrande diagram and workability diagram characteristics, like grain size distribution and Atterberg limits, the

classification of the sediments for their possible use for earthen production are carried out. For Tenosique, T1 and T5 are suitable for bricks because of their intermediate level of plasticity and shrinkage. T2 occurs to be out of chart and T3 close to the threshold values. For Jonuta, almost all of the samples are out of the threshold of plasticity and shrinkage. Only J5 present an acceptable level of plasticity.

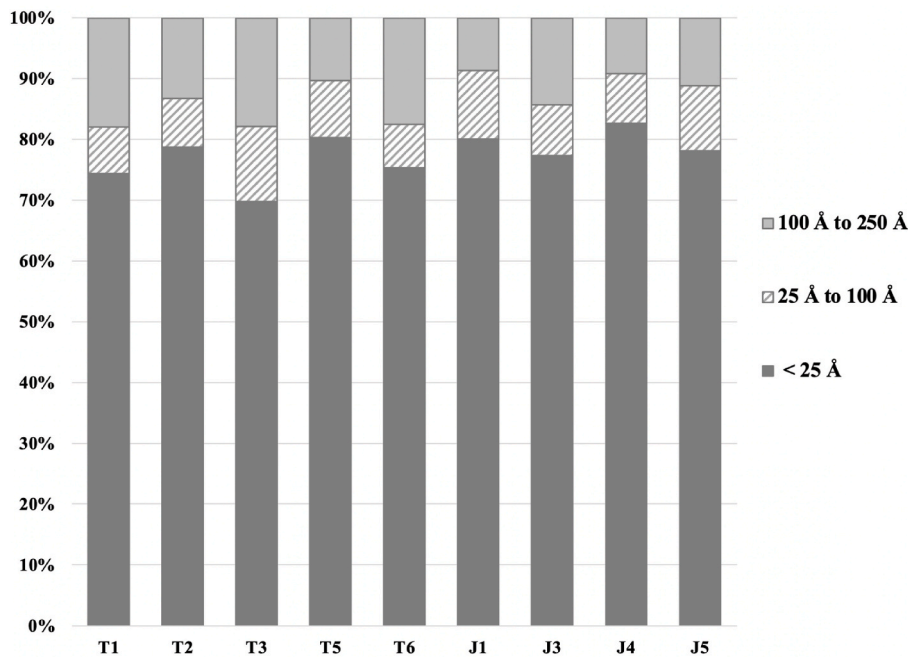


Fig. 7. Graphical representation of pore size distribution for all sediments.

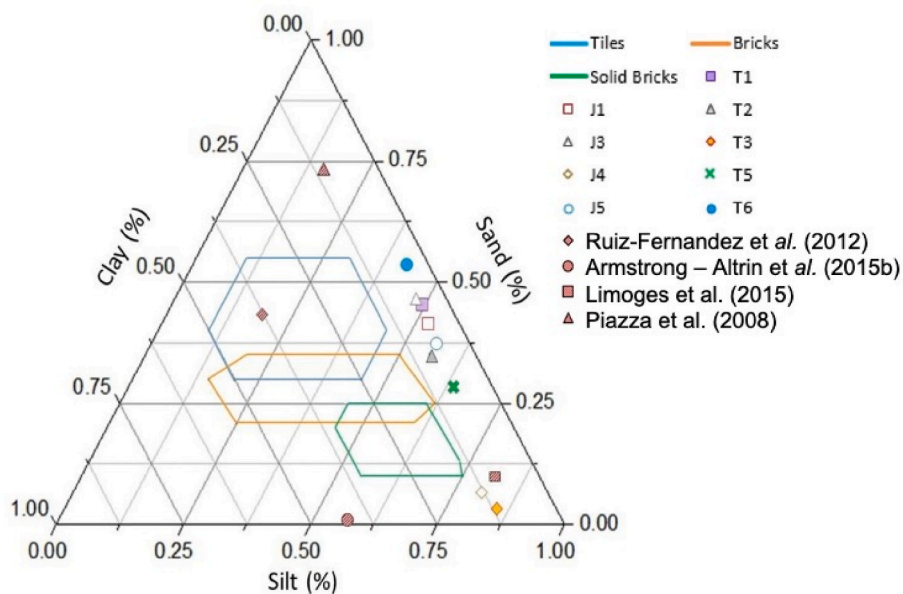


Fig. 8. Classification of the Usumacinta River sediments on Winkler diagram for assessment of their suitability of use as bricks (Ruiz-Fernández et al., 2012; Armstrong-Altrin et al., 2015b; Limoges et al., 2015; Piazza et al., 2008).

4.2. Use of sediments for earthen structures

Another possibility is use for the construction of access and communication pathways around the river (river roads and footpaths, local services, unpaved roads with restricted use) and other earthen structures like dike, dam and barriers. The results obtained for the 80 μ pass, MBV values and Atterberg limits can be used for the GTR classification (Table 7). With six sediment samples classified as A2 and the remainder as A1, the sediments can be used as unpaved road construction material, provided that some solidification/stabilization preliminary steps are carried out. The classification of the soils and their suitability for compaction allow for the discussion about their possible use as road material. Because the envisaged road network is subject to

seasonal flooding and weathering, the sediment strength needs to be known to assess performances.

In order to evaluate the resistance to particle stripping on the surface of earthen roads, the viscosity of the sediments is examined. After cyclones and torrential rains (rain, high winds ...), agricultural and service road surface degradation can occur in the form of surface tension of water current at the air-water interface resulting in surface erosion. Fig. 4a displays the viscosity values measured for the Tenosique sediments. The sediments form two consistent groups: T1-T2 and T3-T5. These two groups differ in their blue value results (Table 5), showing differences in the clay content and water affinity. For T3-T5, water acts as a lubricant and decreases viscosity. The viscosity value hierarchy can also be explained by the carbonate content. The higher the carbonate

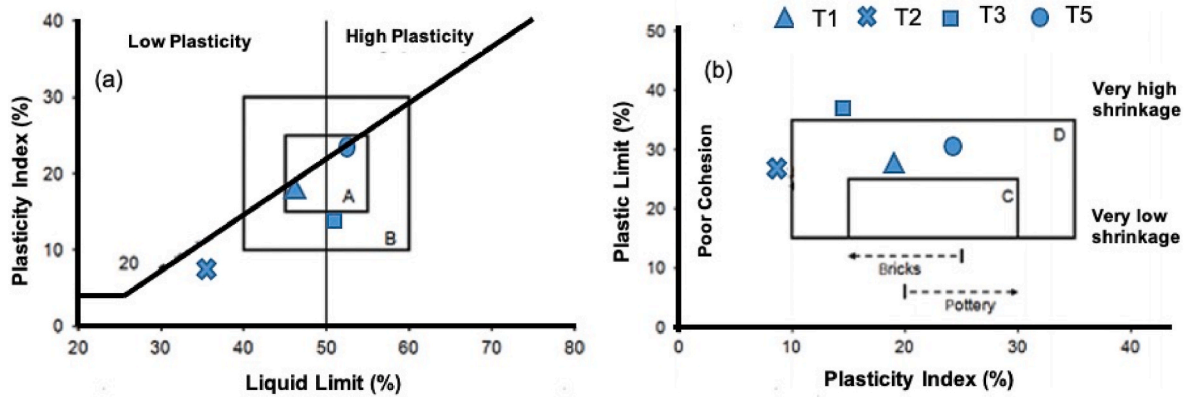


Fig. 9. (a) Casagrande diagram with material extrusion areas, Tenosique samples (Gippini, 1969), (b) Clay workability diagram, Tenosique samples (Bain and Highley, 1979).

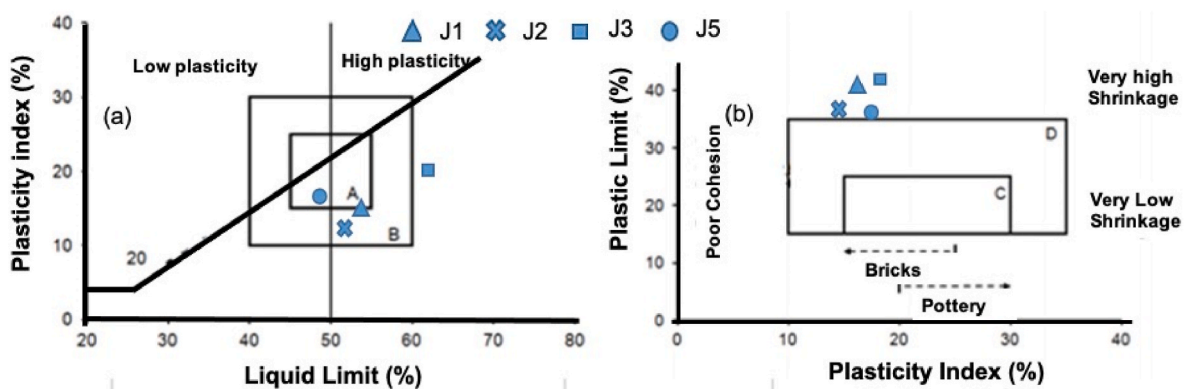


Fig. 10. (a) Casagrande diagram with material extrusion areas, Jonuta samples (Gippini, 1969), (b) Clay workability diagram, Jonuta samples (Bain and Highley, 1979).

content, the higher the sediment resistance and viscosity. Sample T1, for instance, which presents the highest carbonate content (9.4%), exhibits also the highest viscosity value (Table 8 and Fig. 4). Again, simple and quick measurements, like carbonate content, can avoid viscosity measurements, which require specific equipment not easily accessible to local communities.

4.3. Use of sediments in agriculture

The sediment characterisation focuses on spreading of the sediments. The main study parameters to be considered when assessing sediment suitability for agricultural reuse are:

(1) the particle size composition: particle distribution is related to the nature and texture of the soil. Hence the sediment classification according to texture is represented here using a ternary diagram or not, according to clay, silt and sand contents. Soil nature or texture provides information on soil fertility; (2) the organic matter: this is a complex mix of substances and carbon compounds. It has a major role in the soil global behaviour. It ensures soil structure and stability performances, and also provides storage and supply to plants of the nutritive elements needed through mineralisation; (3) the clay-humus complex: it is the actual nutrient reservoir for crops (Anger, 2014). The layered structure of clays gives to the complex a high negative charge. A number of free cations found in the soil solution can thus bind with it (Ca²⁺, K⁺, H⁺, Na²⁺ etc.). To assess the size of this reservoir, the Cation Exchange Capacity is determined; (4) carbonates: limestone, among others, is a soil natural mineral particle composed of either calcium carbonate (CaCO₃) or anthropogenic inputs in the form of basic mineral amendments; (5) the nutrient potential of soils and fertilisers: this potential is

assessed through the analysis of the mineral elements, contained in the clay-humus complex and soil solution, that are necessary for germination and growth of plants.

Soil amendment must have some essential characteristics, like (i) fertility, nutrients and organic matter, (ii) specific surface and (iii) non-salinity of the environment.

4.3.1. Soil fertility with sediments

Soil fertility is characterised by its ability to bind cations with its specific surface, called CEC level. The higher the CEC, the higher the number of cations that can be stored in the soil. This value results from the negative charges at the clay surface and from organic matter content. The CEC level depends on the soil mineralogical characteristics (amount and type of clay) and on the organic matter content. It also depends on the pH: the higher the pH, the higher the CEC. The Usumacinta River sediments have a relatively low CEC level, which corresponds to an organic matter value below 6% (Table 8). Clay amount and nature are factors with great impact on the CEC values. After the specific amount of montmorillonite and vermiculite has been added for all the sediments (Table 9), it should be noted that the larger the amount of clay (montmorillonite plus vermiculite), the higher the CEC value (Table 14). Apart from sample T1, which does not follow this trend, it is worth noting that all the values are very close and that other factors, like feldspar and carbonates, could affect CEC values. In conclusion, CEC is a relevant factor for soil fertility monitoring. Regarding acid soils with low CEC values, soil remediation can be achieved with the addition of lime.

CEC values are estimated using the methylene blue value method, which is a simple and quick measurement tool that does not require sophisticated equipment (Table 14).

Table 14CEC_{measured} classified in increasing order with corresponding CEC_{MBV} and (montmorillonite + vermiculite) percentages.

	T2	T1	T6	T3	J3	J5	J1	T5	J4
CEC _{measured} (meq/100 g clay)	13.3	15.5	18.3	19.4	20.2	20.7	24.5	25	35.7
CEC _{MBV} (meq/100 g clay)	6	7	8	10	11	13	14	18	24
(Montmorillonite + vermiculite) content (%)	6.2	5.7	6.5	–	6.5	6.6	7.3	8.1	27.1

CEC_{MBV} values are underestimated in comparison with the directly measured CEC values (Table 14). However, both methods are highly correlated (Fig. 11) meaning that MBV testing

Is a reliable method for the determination of CEC level because it is easy to apply and does not

Require specific equipment.

4.3.2. Specific surface area of sediments

The Specific Surface Area (SSA) is a parameter, which provides information on the sediment affinity for water and the solid particle developed surface, which is the place where the nutrients for the soils are both exchanged and retained. Two methods can be used to determine the SSA values: direct measurement using nitrogen gas (N₂) adsorption isotherms (SSA_{N2}) and Methylene Blue Value (SSA_{MBV}). The N₂ adsorption method underestimates the SSA values of the soil tests (Tables 11 and 13). Yukselen and Kaya (2006) have made the same observation, which they explain by the fact that the N₂ adsorption method measures only the external surfaces. Other liquid methods can measure both internal and external surfaces. The SSA values will be the same whatever the test method used only for soils having a noninternal surface area (Santamarina et al., 2002). As already mentioned, the SSA_{MBV} values are higher than those obtained using the BET method. This can be accounted for by the fact that, with the second method, the measurements are carried out on blocks of materials with some inaccessible surfaces whereas methylene blue measurements are carried out on suspended materials. However, the SSA values obtained using both methods show a very good correlation (Fig. 12), especially for SSA_{MBV} values higher than 60 m²/g, for which the correlation is high. As Yukselen and Kaya (2008) already mentioned, the high correlation between both methods serves the MBV method, which is simple and requires no heavy equipment.

4.3.3. Sediment salinity

Soil salinity has significant impact on agriculture. SAR is a quantitative indicator, which provides a reasonable estimate of sodicity risks for soils, based on the Na⁺ level. Na⁺ concentrations can have a negative impact on the physical properties of soils causing clay dispersion and

resulting in a reduction of the aggregate stability, a decrease in soil hydraulic conductivity, an increase in the susceptibility to surface sealing and soil erosion problems, the soil compaction, and a decrease in soil aeration (Martínez-Alvarez et al., 2018). Soils with a high sodicity level are not appropriate for agriculture. The United States Soil Salinity Laboratory (USSLS and United States Salinity Laboratory Staff, 1954) has developed a widely used salinity classification system, which considers the total level of salt estimated from the electrical conductivity values and the sodium adsorption ratio to classify the soils as saline, saline-alkaline and alkaline Fig. 13. According to this classification, and results of Tables 8 and 11, the Usumacinta River sediments tested are classified as non-saline, non-alkaline. Alkalisiation and sediment dispersion are limited. The sediments are therefore less sensitive to water and flocculation, which is an advantage for reuse in agriculture.

Again, with the aim of using simple and easily accessible measurement methods, a correlation is established between the SAR values, whose direct measurement requires sophisticated equipment, and the CEC values calculated using the MBV method. With the exception of sample T5, which has a very high CEC_{MBV} value, a good linear correlation is found between SAR and CEC (Fig. 14). Thus, for non-amorphous sediments, SAR can be determined from the CEC values, which are much easier to obtain than the identification of different cations (Ca⁺, Na⁺, Mg⁺) in sediments.

5. Conclusions

The Usumacinta River basin in Mexico, located in a tropical region, is exposed to climate change and human activities with anthropogenic impacts. River sediments are an important part of this ecosystem. This paper is the first to provide the results of the extensive characterisation campaign carried out to determine the rheological, geotechnical, physicochemical, and microstructural properties of the Usumacinta River sediments in five different areas. Carrying out such a study required a large volume of sediment.

The first results highlight the influence of rheology, the trend between clay (MBV) and viscosity is useful for the workability of earthen construction. The clay content influences almost all sediment properties. In spite of a relatively low clay content (montmorillonite, vermiculite), below 11%, the presence of clay, however, appears to affect the microstructural, rheological and physicochemical properties of sediments.

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The present study demonstrates strong correlation between the geotechnical and the physicochemical characteristics. In fact, the higher the clay content appears, the higher the specific surface area (SSA) and the cation exchange capacity (CEC) are, but the lower the viscosity is. The presence of carbonate minerals appears to affect viscosity. Among the different pore size ranges investigated, clay content and micropore range are satisfactorily correlated below 25 Å.

The results also show that SSA values measured using both N₂ adsorption isotherms and MBV methods are highly correlated. The same conclusions apply to the CEC values measured using the direct method

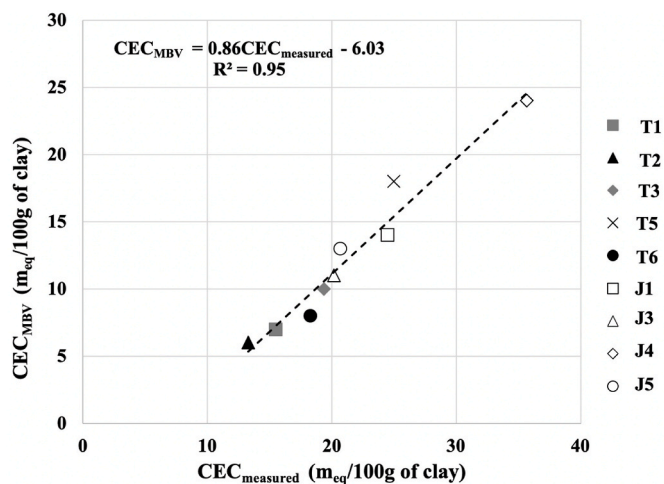


Fig. 11. Correlation between directly measured, and calculated CEC values using MBV method.

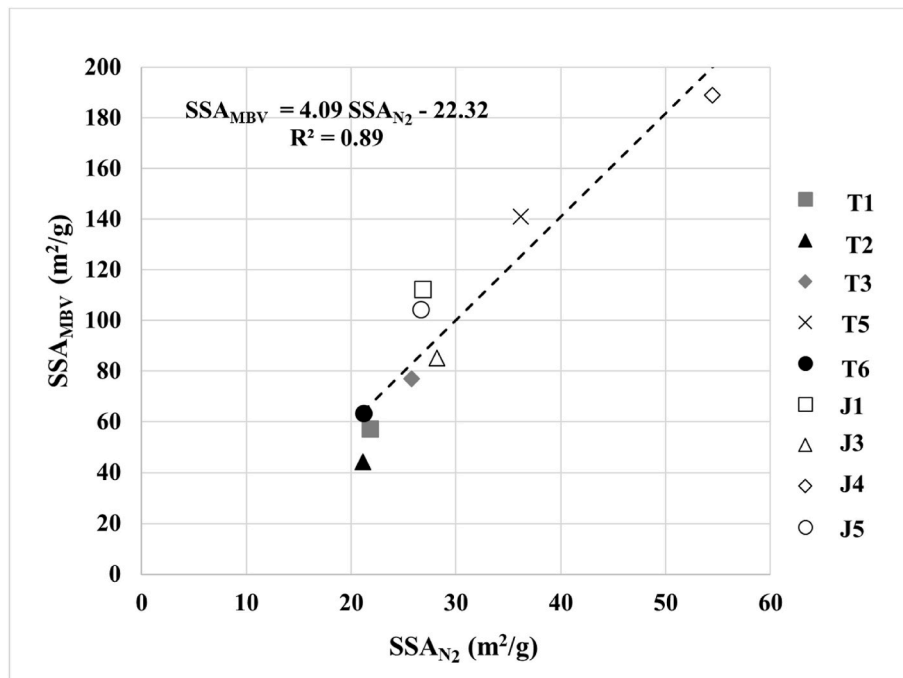


Fig. 12. Correlation between SSA values obtained using N₂ adsorption method and SSA values calculated using MBV method.

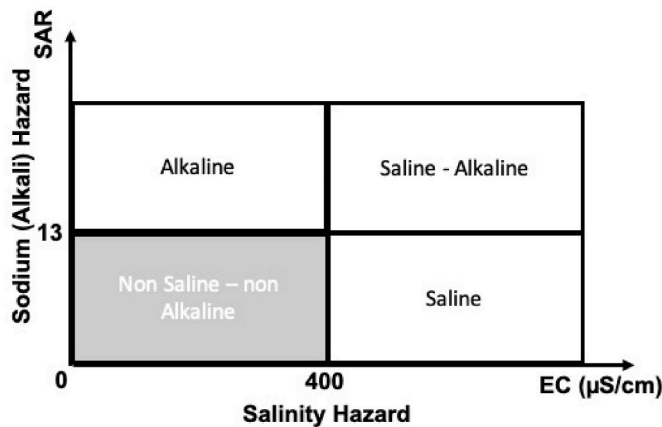


Fig. 13. Salinity/alkalinity/sodicity classification chart from USSLS and United States Salinity Laboratory Staff, 1954 and EC (2012)

or calculated from the MBV results. The MBV method should therefore be encouraged because it does not require expensive equipment and is, in addition, easy to use in laboratories.

Furthermore, correlations between the microstructural and the physicochemical properties are highlighted and show that the sodium adsorption ratio (SAR) and the cation exchange capacity can be obtained from the specific surface area values. The environmental properties obtained prove that, with the exception of nickel, the level of heavy metal pollution in the sediments is low. Using the phytoremediation technique, as reported before, nickel can be removed and then Usumacinta River sediments can be beneficial in earthen construction and in agriculture.

The physicochemical properties like grain size distribution, soil texture (silty, clayey, silty-sand), pH, CaCO₃, organic matter, CEC and SAR are important criteria to study possibilities for the use of the sediments in agriculture. In relation to pH, SAR and electrical conductivity, the sediments are classified as lightly alkaline, non-saline or mildly saline (sample J4) with low sodicity risk. Consequently, the sediments studied suit agriculture use with, however, specific attention to alkaline-

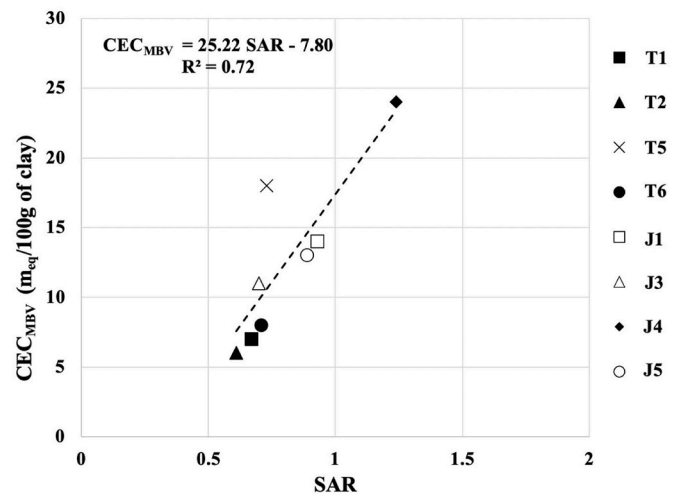


Fig. 14. Correlation between SAR and CEC calculated using the MBV method.

sensitive and sodium-sensitive crops.

In conclusion, this extensive and detailed study of the Usumacinta River sediments is an essential first step toward identifying achievable beneficial uses. Additional on-going studies are currently being conducted to assess the suitability of the sediment for use as construction material for light buildings (Djeran-Maigre et al., 2022b) or unpaved roads (Djeran-Maigre et al., 2022a) and agronomic valorisation to promote riverbank biodiversity and local agriculture (Djeran-Maigre et al., 2022c).

CRediT authorship contribution statement

Irini Djeran-Maigre: Writing – review & editing, Validation, Supervision, Project administration, Methodology, Funding acquisition, Formal analysis, Data curation, Conceptualization. **Andry Razakamantsoa:** Validation, Resources, Methodology, Investigation, Data curation, Conceptualization. **Daniel Levacher:** Writing – review &

editing, Validation, Supervision, Methodology, Investigation, Formal analysis, Conceptualization. **Mazhar Hussain:** Validation, Investigation. **Estelle Delfosse:** Writing – original draft, Formal analysis.

Declaration of competing interest

The authors declare the following financial interests/personal relationships which may be considered as potential competing interests: Irini DJERAN-MAIGRE reports financial support was provided by French National Research Agency. Irini DJERAN-MAIGRE reports was provided by National Council for Science and Technology. No relationship or activity that may be interpreted as a conflict of interest by the reader.

Data availability

The authors do not have permission to share data.

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Abbreviations

MBV	Methylene Blue Value
LL	Liquidity Limit
CEC	Cation exchange Capacity
SSA	Specific Surface Area

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