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Performance of sewage sludge reuse in the manufacturing of fired bricks

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ABSTRACT

BACKGROUND AND OBJECTIVES: The disposal of sludge from wastewater treatment plants into the natural environment represents a major danger to the environment and human health. The use of urban sludge as raw material in the manufacture of clay bricks not only reduces the amount of sludge but also transforms it into useful materials. This paper studies the physicochemical, geotechnical, mineralogical, mechanical, and environmental characterizations of earth bricks with the objective of evaluating the performance of the sewage sludge and eventually studying its consistency with natural clay to produce brick samples. The aim of the study is to evaluate the properties of bricks made from sewage sludge, innovating an effective elimination of the urban sludge.
METHODS: Measurements of sludge heavy metal concentrations are made with an inductively coupled plasma on a 63-micrometer fine particle fraction. Organic matter performed by the Walkley-Black assay and the loss on ignition method for comparison. Volatile matter, total nitrogen, moisture content, dryness, pH, methylene blue assay, and carbonate rate were determined using a 2-millimeter Bernard calcimeter by volumetric method. Density, plasticity, liquidity index, and pore distribution were determined using a mercury porosimeter, and the specific surface and granulometric analysis have been established. Mineralogical characterization of sludge by X-ray diffraction, and X-ray fluorescence has been provided for comparative analysis with natural clay. The brick samples were then shaped, dried, fired at 930 Celsius degree, and qualified by tests, including linear and mass shrinkage, porosity, water absorption, density, compressive strength, X-ray diffraction, and leaching.
FINDINGS: The analyses by X-ray fluorescence and X-ray diffraction of the sludge revealed the presence of mineral constituents, including calcite, silica, kaolinite, and dolomite and shows an important amount of silicon dioxide (31.6 percent) and aluminum oxide (11.5 percent). The sludge was classified as fine with a silty-clay character, with a plasticity index of 54.63 percent, and was therefore very plastic, and had trace element concentrations below the authorized standards. A sample containing 10 percent of sludge gives a strength of 25.9 megapascal which is close to that of the control brick. The bulk density analysis of brick sample 1 was 1.57 grams per cubic centimeter and classifies it as a light building material. Above 20 percent of samples substitution, their strength becomes less reliable, due to the large amount of organic matter that burns during firing which creates pores in the finished product.
CONCLUSION: These results suggest that thermal processes limit the leaching of metals and are practically inaccessible to the ecosystem, so the brick cannot harm the environment. As the sludge content increases, the final structure no longer conforms to the control sample. This incorporation of natural clay and sludge allows to obtain a lighter brick than the conventional one. The study suggests that a 10 percent incorporation of sludge allows the production of efficient bricks and reinforces the potential of this valorization technique, which efficiently contributes to the accomplishment of sustainable waste management objectives. By helping reduce waste produced in very large quantities, this study contributes to the protection of the environment and human health.

NUMBER OF REFERENCES	NUMBER OF FIGURES	NUMBER OF TABLES
62	6	3

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INTRODUCTION

Around the world, debate over environmental issues is an on-going concern. With economic, social, and environmental pressures, the need for natural resources increases over time. Large amounts of waste are generated daily through a multitude of manufacturing processes in various industrial, mining, sanitary, building, and agricultural sectors. The use of recycling processes shows a thoughtful way to protect natural resources and the environment (Martínez-García *et al.,* 2012; Nandi *et al*., 2015). The treatment of wastewater consumed by humans on an industrial and domestic scale inevitably generates significant amounts of sludge, which must be disposed of. Their incorporation as a raw material for the manufacture of clay bricks supports the idea that this sludge can be widely considered as a resource rather than a waste. In the perspective of saving energy and reducing carbon dioxide (CO_2) emissions, the construction industries are looking for efficient, resistant materials with less environmental load while being compatible with the concept of sustainable development. (Rao-Meda *et al*., 2020). Sewage sludge is strongly conditioned by the source of the collected wastewater (Werther and Ogada, 1999; Jarde *et al*., 2003; Singh *et al*., 2004). It is generated at various stages of decontamination of this wastewater altered by different human activities. This sludge is considered a semi-solid and heterogeneous residue, a set of organic and inorganic components, a multi-phase medium of bacteria, and a microbial biomass containing pathogens, nutrients (nitrogen and phosphorus), heavy metals, and a large amount of water 50-60 percent (%) (Zat *et al*., 2021; LeBlanc *et al*., 2009; Chang *et al*., 2020; Yang *et al*., 2015). The sludge in the plant undergoes several treatment processes, including thickening, dewatering, and stabilization through different physical and chemical processes (Liu *et al*., 2013). At this level, the digested sludge is generated, although the moisture content and organic matter content remain high. In Morocco, by 2030, the volume of treated wastewater will reach 900 cubic millimeter per year (mm^3/y), (MAPM, 2011), necessarily generating significant quantities of sludge, with 89,965 cubic meter (m^3) as an annual total in 2014 (PNA, 2008). This pushes the country to engage and initiate the practices of the circular economy by developing any channel of recycling and recovery of waste. Strategies for the treatment of sewage sludge should be considered and implemented so that the process of wastewater treatment takes on its full significance (Moulato *et al.,* 2020), avoiding any contamination of soil, plants, and water. The estimated quantity of sludge worldwide is 45 million tons in 2017 (Zhang *et al.,* 2017). In Europe, it is estimated to be 13.5 million tons in 2020 (Mininni *et al*., 2015). There are several treatment technologies to transform plant sludge into a non-toxic form that respects the ecological balance. These include their uses as resources for fertilizers and bioflocculants (Shi *et al.,* 2017), the production of methane-rich biogas through anaerobic digestion (hydrolysis, methanogenesis) (Collet *et al*., 2017) and the employment of sludge as fuel in thermal processes such as incineration, pyrolysis, and gasification for electricity and heat productions (Abuşoğlu *et al.,* 2017). The energy efficiency of heat recovery systems can reach 37.1% (Quan *et al.,* 2022). The disposal of sludge should be controlled, the moisture content should be less than 60 % and adding solidifiers (magnesium salts, lime, coal ash, aged garbage) is favored. However, this method of disposal has largely demonstrated the most significant environmental impacts. Moreover, its storage requires the occupation of large areas of land, which causes the emission of greenhouse gases. In Japan, 97% of the sludge undergoes reuse in various energy and agricultural sectors and 50% is directed toward recycling it into construction materials (Guangyin and Youcai, 2017). Energy efficiency in the building sector concerns researchers to produce a sustainable ecological material promoting the reduction of energy (Saidi *et al.,* 2019; Limami *et al* 2021). Incorporating 25% of sludge from the plants in the production of bricks can indeed reduce 48.6% of energy consumption during the firing process (Mohajerani *et al.,* 2019). Clay bricks are the most widely used building materials in civil engineering structures (Murmu and Patel, 2018), due to their mechanical and thermal insulation properties (Limami *et al.,* 2019). The high silica (SiO_2) content in sewage sludge improves the physical properties of the brick, which makes this recovery route more environmentally friendly and conservative. A high content of ferric oxide (Fe_2O_3) can be observed in the sludge of the wastewater treatment plant (WWTP), which represents an advantage to the production of bricks and the saving of iron ore in the manufacturing process (Montero *et al.,* 2009; Zhang *et al.,* 2016). Heavy metals are immobilized in the brick without influencing the environmental and technical characteristics of the final product (Chang *et al.,* 2020; Cusidó and Cremades, 2012; Kadir *et al.,* 2017). Indeed, after sintering, the heavy metals present in the sewage sludge were solidified (Yang *et al.,* 2021) and their leaching is limited. This represents a potential advantage that makes this wastewater treatment plant sludge valorization pathway relevant. Prior to incorporating the sludge in the production of the bricks, it is important to conduct a comparative analysis of the natural clay with the sludge from the WWTP to analyze the possible consistency between the two materials. The amount of sludge to be added, the temperature, and the firing time are major factors that condition the quality of the brick to be designed in terms of strength, bulk density, and linear shrinkage (Rao-Meda *et al.,* 2020; Lin and Weng, 2001). Indeed, the temperature increase from 950°C (Celsius degree) to 1150°C leads to the increase of the bulk density and compacts the particles inside the brick through the formation of crystalline and liquid phases (Luo *et al.,* 2020). Several researchers have strongly endorsed this recovery pathway as it eliminates some of the costly and energy-intensive steps in WWTP sludge disposal and reduces the carbon footprint following the incorporation of a minimum amount of 15% of sludge (Mohajerani *et al.,* 2019). An addition of 6% of liming sludge revealed better technical properties and characteristics (27.5 megapascal for compressive strength, 2.18 grams per cubic centimeter for bulk density, and 10.46% water absorption) (Anik-Hasan *et al.,* 2022). Studies have evaluated additives as raw materials to increase the amount of sludge incorporated, which can be as high as 40% through mixing the sludge with amorphous rice husk ash and Sodium carbonate (Na_2CO_3) (Yang *et al.,* 2021). Paper processing residues (Sutcu and Akkurt, 2009) and glass waste (Dondi *et al.,* 2009) were also evaluated for possible incorporation into bricks. The technique of using waste in the construction industry reduces the need for raw materials and decreases environmental pollution. This recovery generates job opportunities, thus contributing to socio-economic development (Soni *et al.,* 2022). Indeed, the dimensions of sustainable development concept is related to economic, social, and environmental efficiency through better waste management (Łęgowik-Świącik, 2019). A management system must be unique for each area according to its dynamic variables (Weekes *et al.,* 2021). In recent years, several studies have assessed WWTP sludge as raw materials in brick production (Areias *et al.,* 2020; Erdogmus *et al.,* 2021; Wu *et al.,* 2022). In this study, the objective is to testify the feasibility of incorporating the sludge from the Boukhalef plant located southwest of the city of Tangier in the manufacture of clay bricks. The WWTP in question treats urban and industrial waste from the TFZ (tangier free zone), the industrial zone of Gzenaya, and the large neighborhoods in proximity. A chemical, physical, geotechnical, and mineralogical characterization of the WWTP sludge was conducted to compare its composition with that of the natural clay. The sludge was used as a partial substitute for the clay used in the formulation of the laboratory-scale brick samples. To determine the optimum substitution, qualification tests of the different brick samples, including linear drying and firing shrinkage, loss on ignition (mass shrinkage), porosity, water absorption, compressive strength, density, X-ray diffraction, and leaching tests were considered. These measurements were determined and compared to existing standards of the Association Française de Normalisation (AFNOR). The objective is to provide decision makers concerned with the management of sludge from WWTPs in Morocco the relevant elements constituting an identity card of the sludge from the plant of Boukhalef in Tangier for a choice of an effective treatment method. Indeed, the production of an ecological, low energy demand, and resistant building material from sludge reduces the harmful effects of pollution and supports the strategies of circular economy and environmental protection. This study was conducted at the Faculty of Sciences and Techniques of Tangier in Morocco in 2021. It aims to evaluate the properties of bricks made from waste, approving the efficient ability of the urban sludge to be used as raw material for the manufacture of a building element, with improved thermal performance, good quality, and mechanical strength according to the authorized standards, and reduce energy consumption and establish sustainability, thus innovating an effective elimination of urban sludge, which is daily and inevitably produced, by exploiting its physico-chemical properties for the manufacture of a widely usable material for construction, putting in place an environmental and socio-economic approach.

MATERIALS AND METHODS

Characteristics of sewage sludge

The WWTP of Boukhalef in Tangier treats 11,000 m^3/day of industrial and domestic wastewater and rainwater as effluents, with a tertiary treatment of 5,000 cubic meter per day (m^3/day). The sewage sludge used in our experiment was produced in the purification station of Boukhalef; it is dark gray in color, displayed in a fresh solid form, with an unpleasant odor. This semi-solid was exploited as an additive after dehydration and storage in special skips collected using a stainless-steel shovel and preserved in closed containers in order minimize any contact with air. Sludge samples were also stored at a temperature of 4°C to minimize any bacterial activity that could modify its properties. The sludge was analyzed according to the standards and used as a raw material by partial substitution of clay for the manufacture of bricks. It has 71% water content and undergoes initial pre-treatment through air drying. Representative samples were then homogenized, dried in a steam bath at 40°C for 48 hours. The sludge is sieved and conserved to obtain a constant mass, eliminate impurities, reduce the organic fraction, and guarantee a balanced humidity before performing the various analyses. To evaluate the suitability of this sludge to be incorporated in the composition of the brick, the complete characterization of the most relevant parameters must be performed first. X-ray diffraction (XRD) allowed to identify the crystalline phases of the sewage sludge and their sizes using a Spinner model apparatus with a copper anticathode and generator with a voltage and consumption of 35 kilovolt (KV) 30 milliampere (mA). A scan from 4° (degree) to 84° of the 2 ϴ (theta angle) was performed in each case at 0.02° per second. X-ray fluorescence (XRF) spectrometry also allowed to quantify and qualify the chemical elements present in the sludge such as silica (SiO_2), calcium oxide (CaO), ferric oxide (Fe_2O_3), alumina (Al_2O_3), titanium dioxide (TiO_2), magnesium oxide (MgO), sulfur trioxide (SO_3), phosphorus pentoxide (P_2O_5), and sodium oxide (Na_2O). It is important to know and monitor the concentrations of heavy metals in the sewage sludge in order to determine its pollution level. This analysis is performed with an Inductively Coupled Plasma (ICP) Atomic Emission Spectrometer on a fine particle fraction of 63 Micrometer (µm) which contains the highest concentration of metals. The sample transmitted to the plasma undergoes various stages of decomposition, atomization, and ionization. The intensity of the lines emitted by the atoms and ions generated is measured. The granular distribution of fine grains with a diameter lower than 80 (µm) was measured through laser granulometry to determine the proportion of the various solid constituents of a soil according to their size. The specific surface of the sludge was measured after thermal treatment at 105°C using the Brunauer–Emmett–Teller method of physical adsorption of gas molecules on a solid surface as well as the methylene blue method, which measures the fineness of the grind material. In order to adjust the moisture content of the sludge and the clay for an adequate consistency and plasticity for extrusion, it is necessary to measure the plasticity index (Pi) by the Atterberg limits, which represents the range of water content in which the sludge is in the plastic state. The limits of liquidity (LL) and limits of plasticity (PL), Pi = LL – PL, have been determined with a cone penetrometer according to the French standard (NF) P94-052-1, and NF P94-051. To have access to the distribution of the volumes and sizes of the sludge pores, the mercury porosimeter measures them through the diameter of the pore entrance. Basic chemical characteristics were determined; pH to estimate the neutralization potential of the sludge, dryness, moisture and electrical conductivity a quick way to assess the salinity of the organic substrates. The volatile matter is measured by the weight lost from the sample after calcination for 2 hours at 525°C to determine the organic components that can be found in gaseous form in the atmosphere. The percentage of total nitrogen dissolved in the sludge was determined, including organic and ammoniacal, which allows to quantify the sludge level pollution. The carbonate content was measured on a 2 millimeter (mm) sieved sludge by the action of hydrochloric acid (HCl) on the calcium carbonate ($CaCO_3$) in the sludge; a high carbonate content makes the sludge alkaline and favors all fixation modes. The organic matter characteristic of sludge was determined by the “Walkley-Black” dosage method and directly through the loss on ignition (LOI) method after thermal treatment at 500°C for 16 hours. This study will allow us to know if it has common properties with natural

Fig. 1: Samples of fired test bricks

clay so as to produce bricks of the same quality as the conventional ones.

Comparison of raw materials

The annual production of bricks requires significant amounts of clay (3.13 billion cubic meter of clay soil) (Mohajerani *et al.,* 2019). This means that adding a substitute such as sewage sludge to the clay could be a practical and sustainable alternative, reinforcing the concept of the circular economy through the management of both resources and waste. The variable composition of clay allows its incorporation into several types of waste (Liew *et al.,* 2004). Before mixing the sludge with natural clay, a comparative analysis of the two components must be conducted. The control clay was provided from the brick factory, Al Andalous, located in the region of the city of Tangier. X-ray diffraction and X-ray fluorescence indicate the presence of existing mineralogical phases of the clay, and the chemical composition of major elements. The contents of organic matter and calcite were measured and compared. The granulometric distribution analyzed by laser granulometry determines the size of particles smaller than 80 (µm) (Agostini, 2006) present in the clay. This factor can affect the drying and firing properties and the formation characteristics of the clay. The specific surface is considered in the comparative analysis of the clay and the sludge. It is influenced by the parameters indicated before and the shape and surface state of the grains. The plasticity limit, Atterberg liquidity, and plasticity index were examined and compared.

Production of the sample mixture

The preparation of cylindrical bricks, 30 millimeter (mm) in diameter and 30 millimeter (mm) in length, was carried out at the laboratory scale, and eight different mixtures were obtained: Indicator, Brick1 (B1), Brick2 (B2), Brick3 (B3), Brick3.5 (B3.5), Brick4 (B4), Brick5 (B5), and Brick6 (B6). Based on the results previously obtained and to have a better performance at the level of extrusion, the quantity of water used for the preparation of the samples of bricks varies between 21% and 30.5%. For the reference brick, the quantity is only 10%. The mixtures of sludge with clay were made with a substitution rate of 0%, 10%, 20%, 30%, 35%, 40%, 50%, and 60%. The brick samples prepared at different rates were compared with the reference sample while using the existing standards. Initially the sludge was dried at 105°C and crushed using a ceramic ball mill and then sieved through 2 (mm) before being combined with the brickyard clay according to the different substitutions proposed. Using a mixer, the two materials were homogenized in dry conditions before being moistened and extruded, powered by an electric box. It continuously manufactures the products by transporting the material from the circular entry section (feeding hopper) to the rectangular exit. Drying is then conducted to eliminate the water contained in the shaped products, under ambient conditions and with precision to obtain a shape without surface or volume defects while avoiding cracks. The test bricks do not have their true qualities yet. It is necessary to allow them to acquire their mechanical strength, stability, and resistance to weathering and to subject them to firing at elevated temperatures. The samples made are subjected to a system of firing of up to 930°C in a tunnel kiln maintained for 48 hours. The shape of the final sintered bricks obtained after cooling to room temperature is shown in Fig. 1.

Characterization of bricks

The fired and unfired bricks underwent a series of experiments, including qualification tests and evaluation of the characteristics, and the behavior of materials produced for use in construction. The linear drying shrinkage was measured as the ratio between the difference in length before and after drying and the initial length (Demir, 2008; Mezencevova *et al.,* 2012). The lengths of the samples were measured with a caliper. As for the linear firing, it is related to the sintering process. It is measured to determine the action of heat on the clay material, which causes physical and chemical phenomena and creates a firing shrinkage. The porosity of bricks was measured, and it is one of the most relevant and important parameters in the characterization of the brick. It directly influences the thermal mechanical properties of the material, including strength, absorption, and permeability. The water absorption has also been examined; it determines the ability of the brick to absorb water, it is proportional to the porosity, and it has a close relationship with the density. The degree of water absorption is a function of the size and distribution of pores in the brick, and its durability and strength are strongly influenced by this parameter (Esmeray and Atıs, 2019). Compressive strength represents the maximum stress allowed by a material subjected to a crushing load, expressed in megapascal (MPa) and is considered as a good indicator of the quality of fired materials. Scanning electron microscopy (SEM) images were taken to identify the differences in the pore and crack structure of the different brick samples. XRD analysis was considered to study the mineralogical transformations of the control brick compared with the other samples with different substitution rates. The leaching test is performed to evaluate the potential of pollutant release to the outside (natural) environment. The fired bricks are crushed, ground, and stirred in water for 24 h with a Liquid/Solid (L/S) ratio equal to 10 according to the procedure described in the NF European norm (EN) 12457, 2002. After filtration, the leachates are analyzed in the laboratory using the ICP technique.

RESULTS AND DISCUSSION

Analyses applied to raw materials

The results of the examination of the mineralogical phases present in the raw materials used for the manufacture of bricks at the laboratory scale are demonstrated in Figs. 2 and 3. XRD reveals the presence of the spectra of mineral constituents, including calcite ($CaCO_3$), silica (SiO_2),kaolinite ($Al_2Si_2O_5(OH)_4$), and Dolomite ($CaMg(CO_3)_2$) in the WWTP sludge. The observed mineral composition within the sewage sludge is similar to that of natural clay used by the brickwork. XRF shows that the sludge is mainly composed of silicon dioxide (SiO_2) and aluminum Oxide (Al_2O_3) with respective percentages of 31.6% and 11.5%. It also contains low contents of 5.75% CaO, 3.73% Fe_2O_3, 2.09%MgO, 2.71% P_2O_5, 1.93% SO_3, and other elements in smaller quantities. The predominant oxides in common with the clay are silicon dioxide, aluminum Oxide, iron oxide and CaO with respective percentages of 58.1%, 16.9%, 3.73%, and 5.38%. The combination of 15% sludge with shale revealed very comparable results with a value of 25.60% SiO_2, 12.0% Al_2O_3, and 5.13% Fe_2O_3 (Wu *et al.,* 2022). Al_2O_3 is related to plasticity, and its content in the studied sludge is within the allowed limit. The proportion of SiO_2 of the WWTP is lower than that of clay but still significant. The content of Fe_2O_3 allows the sludge to be considered as clay with a medium content of coloring oxide. Calcium oxide does not exceed the upper limit tolerated by the standards; its presence is due to the quantities of lime used in the sludge dewatering process in the WWTP. The mineral and chemical composition of sludge and clay is beneficial for the sintering and solidification of the final product. There is chemical and mineralogical affinity between the sewage sludge and the clay body (8.83% Al_2O_2, 14.26% SiO_2, and 7.78% Fe_2O_2) (Areias *et al.,* 2020). Among the relevant parameters in the analysis of the possible coherence between clay and sludge is the <80 m particle size of both materials. The median particle diameter 50 (D50)%

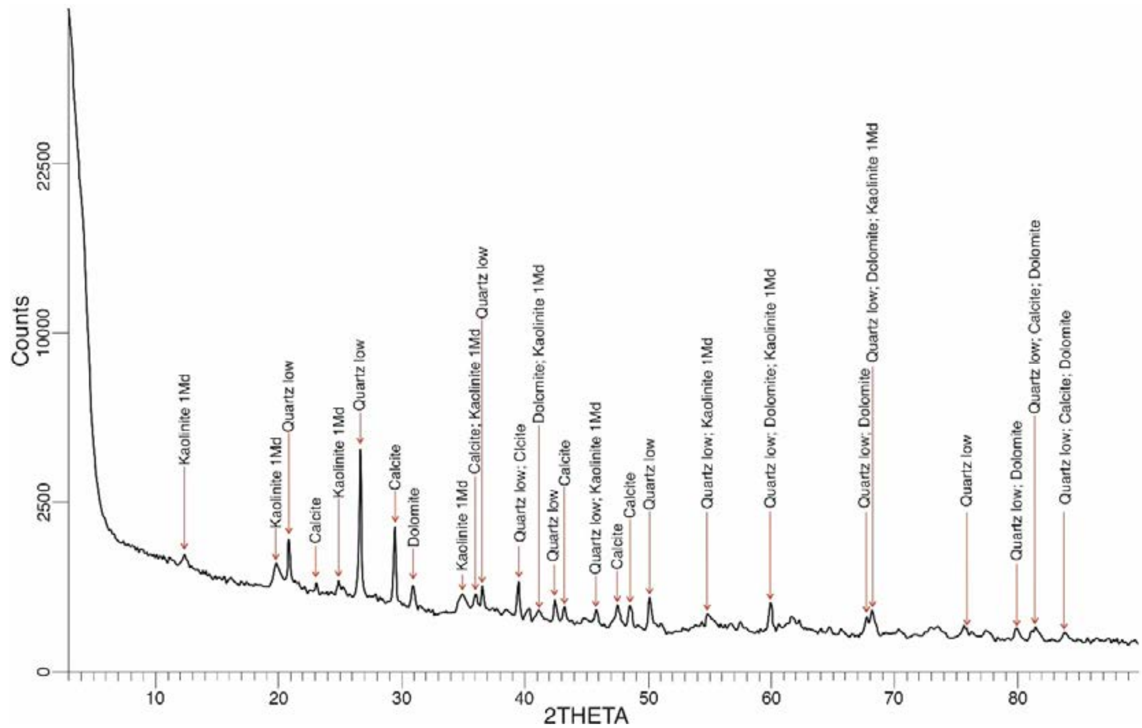

Fig. 2: X-ray diffraction of the studied sludge

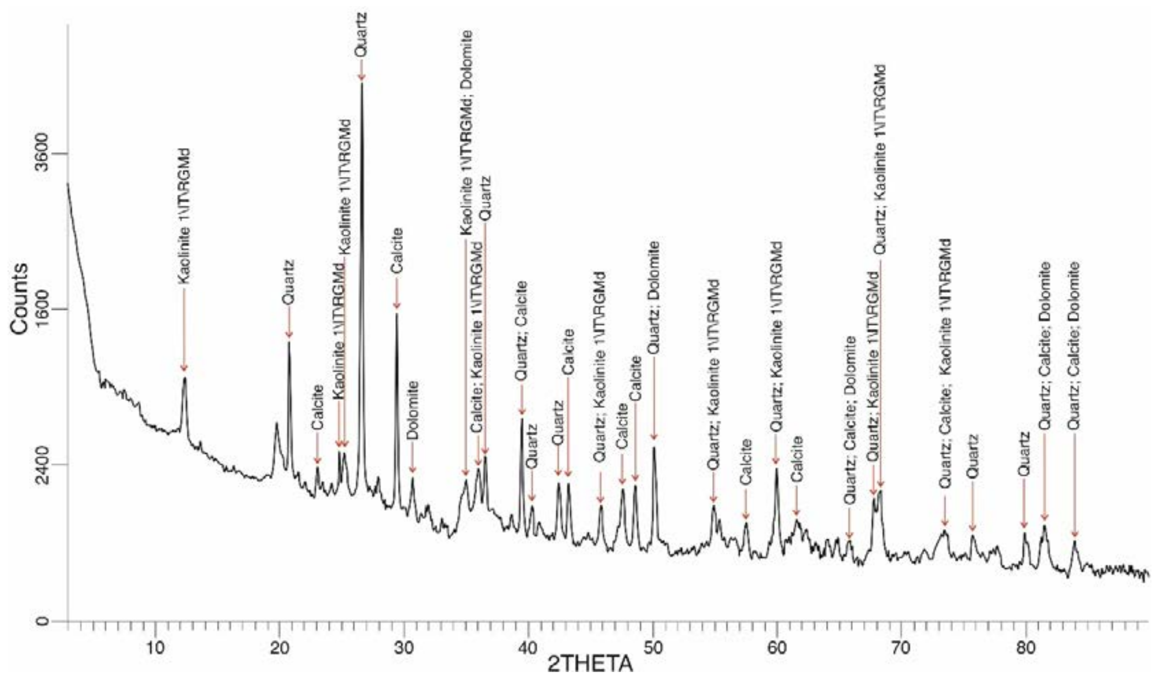

Fig. 3: X-ray diffraction of natural clay

Table 1: The used wastewater treatment plant sludge properties and composition

Parameters	WWTP Sludge properties
pH value	8,32
Moisture content (40°C)	51,57%
Density	1.69 gram per cubic centimeter (g/cm^3)
Electrical conductivity	3,34 millisiemens per centimeter (ms/cm)
Volatile matter	39,67%
Carbonate content	7,97%
COD/BOD	1,8 milligrams of oxygen per liter (mgO_2/L)
Total nitrogen	0,845%
Organic matter	32.5%
Atterberg Liquid limit (%)	114,730%
Atterberg Plastic limit (%)	60,100%
Cadmium (Cd)	2,14 milligram per kilogram (mg/kg)
Chromium (Cr)	366,48 (mg/kg)
Copper (Cu)	2,14 (mg/kg)
Lead (Pb)	79,18 (mg/kg)
Nickel (Ni)	20,33 (mg/kg)
Zinc (Zn)	411,96 (mg/kg)
Loss on ignition	37,5%

of the clay and the sludge studied by laser particle sizing shows a very similar particle distribution of 30.9 µm and 26.669 µm, respectively. The <63 µm fraction of the sludge is 98.95%, with a methylene blue value between 2.5 and 6. These results explain that the sludge is thin, with a clayey silty character, is of average plasticity, and with a total specific surface of 83.72 square meter per gram (m^2/g). The specific surface of the clay, 148.60 m^2/g, is much higher than that of the WWTP sludge, which agrees with granulometric analysis. The values of the Atterberg limits of the Al Andalusian clay and the sludge show that they are classified as very plastic materials (Pi > 40). The Pi of the sludge is 54.630% and that of the clay is 63.172%.

The sludge can be incorporated into the formation of bricks without losing its plastic behavior. The sludge of the studied WWTP is richer in organic matter than the natural clay; it contains 32.50% as opposed to 3.36% for the clay. This result is due to its nature as an activated sludge. The calcite contents for both materials are very comparable (8% for the clay and 7.97% for the sludge). Nevertheless, the high content of organic matter leads to an increase in porosity and a low adhesion force, which influences the thermal and acoustic properties of the final product through the condensation of porosities (Cusidó *et al.*, 2003). Indeed, its presence in the sludge reduces the energy demand considerably during the firing process of the brick (Ukwatta and Mohajerani, 2017) by releasing its calorific value during combustion. The results show that the sludge is rich in organic matter since it is an activated sludge. This high content is also demonstrated by the high percentage of loss on ignition (39.99%). The sewage sludge has a high porosity of 48.7%. Nevertheless, the process of sintering the bricks completes the crystallization of the material through the increase of temperatures, which can reduce the porosity of the final product (Wang *et al.*, 2019). Table 1 shows the results of basic chemical and physical parameters measured for sludge, and those of the heavy metal determination to evaluate the polluting potential of the sludge studied. The analyzed sample presents concentrations in trace elements lower than the standards retained and authorized for their valorization in agriculture,

Table 2: Values of the plasticity index and specific surface of the realized mixtures

Substitutions	Plasticity index %	Specific surface (m^2/g)
Indicator	63,172	115,115
B1 (10%)	115,593	98,371
B2 (20%)	115,425	94,185
B3 (30%)	87,492	89,999
B3.5 (35%)	81,580	87,906
B4 (40%)	77,048	73,255
B5 (50%)	69,677	66,976
B6 (60%)	66,977	62,79

which encourages using this sludge in a building material without any prior treatment. Certainly, it is not possible to conclude that there is a total absence of metallic pollution in the sludge. Some may be higher than others but still in accordance with the standards (Kadir *et al*., 2017). The nature of sludge was determined by pH as defined in the NF X 31-103. The pH value demonstrates that the sludge is of a basic nature. The sludge is composed of water and dry matter and is generally characterized by high water content. Surface water content represents total moisture and it is determined by the drying of samples at 40°C and 105°C until the mass is constant according to NF X 31-101 and NF X 31-102, respectively.

Mixture plasticity and specific surface

The plasticity allows to mold, shape, and keep a constant volume brick shape during drying and firing. The measurement of the mixture plasticity provides a first insight into its quality and determines its consistency. In the literature, it has not been determined yet how the sludge affects the plasticity and consistency of the mix during extrusion (Zat *et al.,* 2021). The Pi and workability of the mixture decreases as the substitution rate of clay by sludge increases. Indeed, the plasticity limit is inversely proportional to the amount of sludge added in the mixture (Weng *et al.,* 2003). The complete permeation of the necessary added water is a factor that controls the plasticity of the sludge and clay and avoids structural defects during the process. The purpose of the Atterberg limit measurements is to identify the water content in the mixture. As the sludge content increases, so does the water requirement. An increase in the addition of water are due to the presence of big amounts of organic matters in sludge. The organic substances in soil have a high absorptive capacity of water (Demir, 2008). The results obtained reveal very interesting Pi percentages (115.59%–66.97%) for the different substitution rates. The clay minerals present in the clay ensure its cohesion and ability to undergo deformation hence the high percentages of plasticity in the control sample as well as B1 and B2. The specific surface of the mixture of the two raw materials was carried out according to the standard NF P94-068 to measure the total surface (external and internal). This parameter also decreases with the increase of the substitution rate, as shown in Table 2. Plasticity represents an important characteristic of the bricks. Inadequate plasticity can result in extrusion failure and development of heterogeneities in the brick body, which can result in weak mechanical properties (Mezencevova *et al.,* 2012).

Analyses of brick samples

After characterization of sludge and clay, the tests have been performed for brick sample manufacturing. Different amounts of sludge, 0% (indicator), B1 (10%), B2 (20%), B3 (30%), B3.5 (35%), B4 (40%), B5 (50%), and B6 (60%) were chosen. Prior to forming bricks, raw materials (natural clay and sludge studied) were dried, crushed, sieved, and mixed well in the dry state after adding water (10%, 21.5% 24.5%, 25.0%, 27.0%, 27.5%, 30.0%, and 30.5%). All the parameters and tests carried out were based on the existing standards. To evaluate the behavior of the bricks formed to be applied in construction, it is necessary to apply qualification and characteristics tests.

Linear shrinkage to drying and linear shrinkage to firing:

During the drying process, the water contained in the mixture is extracted from the clay materials. Tensions within the material occur, and deformation or cracks may appear. The linear drying shrinkage

(B1 8.19%, Indicator 8%) depends mainly on the clay mineralogical composition, the texture of the paste shaping, the amount of water used, and the percentage of the degreasing material. The results presented in Fig. 4a and b show that the shrinkage amplitude of the brick samples was affected by the moisture content of the raw mix, which is related to the rate of substitution. Indeed, this parameter varies according to the content of sludge added to the mixture; when the substitution rate increases, the water content increases, as well as the drying shrinkage. The linear shrinkage on drying takes a value of 8% and increases to 13.6% and 26.5% by adding about 5%–10% of sludge (Zat *et al.,* 2021), which is relatively comparable to the results of the study. The action of heat on the clay material causes physical and chemical phenomena and creates a shrinkage on firing, which also increases with the substitution rate of the sludge. The linear firing shrinkage (B1 10.22%, Indicator 10%) is mainly related to the sintering phenomenon, which in turn depends on the particle size of the sludge. Being finer than the clay, the sludge responds better to sintering, which makes the shrinkage more important. Indeed, the fraction of organic matter contained in the sludge has a significant influence on the linear shrinkage of the brick.

Heat loss

In the light of the obtained results, Fig. 4c shows that the heat loss percentage of fired test bricks on ignition increases in the same direction with the increase of the substitution rate at 10.59% and 12.48% for the indicator and B1, respectively. This is due to the decomposition of calcite and the removal of organic matter present in the sludge during the firing of the bricks. The loss on ignition varies with the curing temperature of the material and depends on the organic matter contents of the sludge. It is related to clay mineral dehydroxylation, oxidation of organic matter, carbonate decomposition, sulfides, and hydroxides. Upon the addition of sludge in the mixture, the weight loss apparently increased because of the loss of contribution of organic matter in the sludge. Furthermore, the brick weight loss on ignition also depends on the inorganic substances in both clay and sludge being burnt off during the firing process (Luo *et al.,* 2020).

Porosity

This parameter directly influences the mechanical strength, water absorption, and permeability of bricks (Samara, 2007). It is important to determine the porosity of bricks for the indication of their quality, durability, as well as for dimensional stability to obtain a final product without defects. The occurrence of open pores on the bricks significantly affects its mechanical performance (Hegazy, *et al.,* 2011). The addition of the studied sludge leads to an increase in the porosity of the bricks compared with the control sample as well as for all proportions suggested (Fig. 4d). This is attributed to the large amount of organic matter present in the sludge, which after undergoing thermal destruction during cooking at 930°C, creates pores. The presence of carbonates is also a factor contributing to the formation of pores, which after decomposition releases carbon dioxide and accentuates the phenomenon in question. Samples B1 and B2 have a respective porosity of 20.6% and 22.97% compared with the control value of 16.88%. Studies show that elements composed of SiO_2 have a high porosity percentage and a lower mechanical strength (Singh *et al.,* 2018). Incorporation of 5%–20% sludge by weight of clay can decrease around 6% porosity in the brick specimen (Amin *et al.,* 2022). Nevertheless, the pores present in the brick improve the thermal insulation properties of the material (Mohajerani *et al.* 2019). To achieve adequate porosity, the addition of 10%–20% WWTP sludge is most appropriate.

Water absorption

According to the study, the coefficient of water absorption of the bricks increases with the addition of the sludge proportions (Fig. 4e). Indeed, the addition of biosolids in the mixtures increases the porosity of the bricks, which generates a more important water absorption. It can be deduced that the size and distribution of pores in the brick define the degree of the material and its capacity of water intrusion. This indicator of brick strength is strongly related to the temperature reached during sintering; the water absorption increases with the sludge content and with the decrease of the firing temperature, which directly affects the durability of the brick (Weng *et al.,* 2003). Amounts higher than 15% might surpass the maximum of 22% for water absorption (Areias

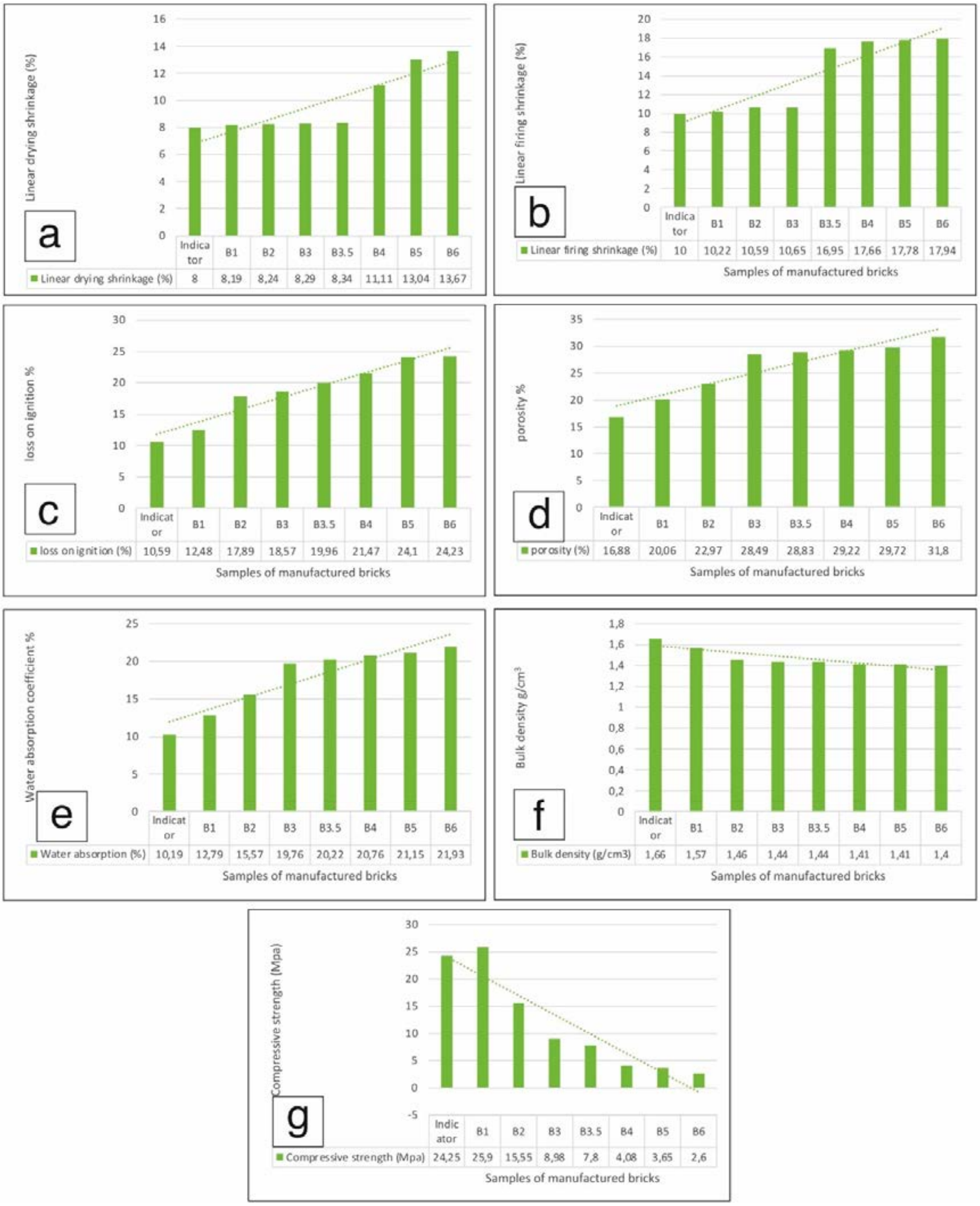

Fig. 4: Qualification tests of the different substitutions of the prepared sludge–clay bricks, B1(10%), B2(20%), B3(30%), B3.5(35%), B4(40%), B5(50%), and B6(60%) compared with the control sample. (a) linear drying shrinkage (%), (b) linear firing shrinkage (%), (c) loss on ignition (%), (d) porosity (%), (e) water absorption coefficient (%), (f) bulk density (g/cm^3), and (g) compressive strength (MPa).

et al., 2022). The water absorption coefficients of tested bricks B1 and B2 do not exceed 12.79% and are remarkably close to the indicator clay sample (10.19%).

Bulk density

As shown in Fig. 4f, the bulk density decreases with increasing sewage sludge rate. These results are consistent with the values of porosity and water absorption. The addition of the sludge makes the brick porous and less dense due to the combustion of organic matter (Frar et al., 2014). Nevertheless, the value of sample B1 (1.57 g/cm^3) does not exceed the recommended margin of 1.75 g/cm^3 and is very comparable to the indicator sample (1.66 g/cm^3). A value of 1.70 g/cm^3 bulk density was mentioned by adding 25% water treatment sludge and increases up to 2.05 g/cm^3 by reaching 100% (Erdogmus et al., 2021). As the temperature increases, the bulk density of the bricks in the sludge–clay mixture increases. The effect of the sludge can be observed through water absorption, porosity, and density, which are closely related. Although the bulk density remains approximately constant from sample B3.5, the sludge additions increase the porosity and water absorption. The temperature increases from 950°C to 1150°C, which can cause the elevation of bulk density and compact the particles inside the brick through the formation of the crystalline and liquid phases (Luo et al., 2020).

Compressive strength

Among the analyses elaborated to define qualification of the produced bricks, the most reliable data are obtained during the pressure resistance tests. All the samples were assessed for pressure after curing. The smallest resistances, 3.65 and 2.6 MPa are recorded for the highest sludge additives. The values in Fig. 4g show that the brick containing 10% (B1) sludge gives a strength of 25.9 MPa, which is close to that of the control brick at 24.25 MPa. Above 20% (B2) substitution, the strength of WWTP sludge bricks becomes less reliable 15.55 MPa due to the large amount of organic matter that burns during firing and creates pores in the finished product. This makes the brick more porous, lighter, and less strong. The release of gases and the thermal decomposition of the organic matter make the brick brittle. The compressive strength of brick samples with 10 % dried sludge and 10 % incinerated sludge could reach up to 70 % and 98 % of the value for the normal clay bricks, respectively (Chang et al., 2020). The strength of the bricks increases with the firing temperature. If the duration is between 2h and 8h, the desired strength can be obtained (Murmu and Patel, 2018). With increasing temperature, the compressive strength is improved by refining the pore structure. This parameter reached 34.4 MPa in a study that combined 15% sludge with 84% shale (Wu et al., 2022). Compressive strength increased remarkably (27.50 MPa) in a study incorporating 6% of liming sludge (Anik-Hasan et al., 2022).

Morphological images by SEM

The morphological analysis of the different samples shows the presence of pores. The comparisons have been made based on the control sample (100% clay). The pore distribution becomes more pronounced from a substitution rate of 20%. The resulting microphotographs corresponds to the different substitution rates proposed in the study. As shown in Fig. 5, the samples hardened at 930°C, and as the sludge content increased, the final structure did not conform to the control sample. Indeed, the texture of the bricks has changed considerably due to the significant condensation of the pores and the growth of their size and distribution. A decrease in the crystalline mineral fraction, including SiO_2 present in the clay, occurs with the addition of sludge, which creates open pores and decreases the mechanical strength of the brick (Limami et al., 2019; Hegazy et al., 2011). An excellent quality brick can be obtained from a mixture of clay (90%) and sludge (10%) dried at 105°C and sintered at 930°C.

XRD

Since the strengths of the control and B1 sample are very comparable, the study focused on analyzing the mineralogical transformations of these two brick samples using XRD analysis. The spectra in Fig. 6 shows the appearance of hematite (Fe_2O_3) and the persistence of quartz (SiO_2) being the main crystalline phase for the two bricks manufactured (Indicator and B1) with minor modifications with respect to the other constituents.

Brick leaching (Environmental study):

Table 3 shows that the concentrations of heavy

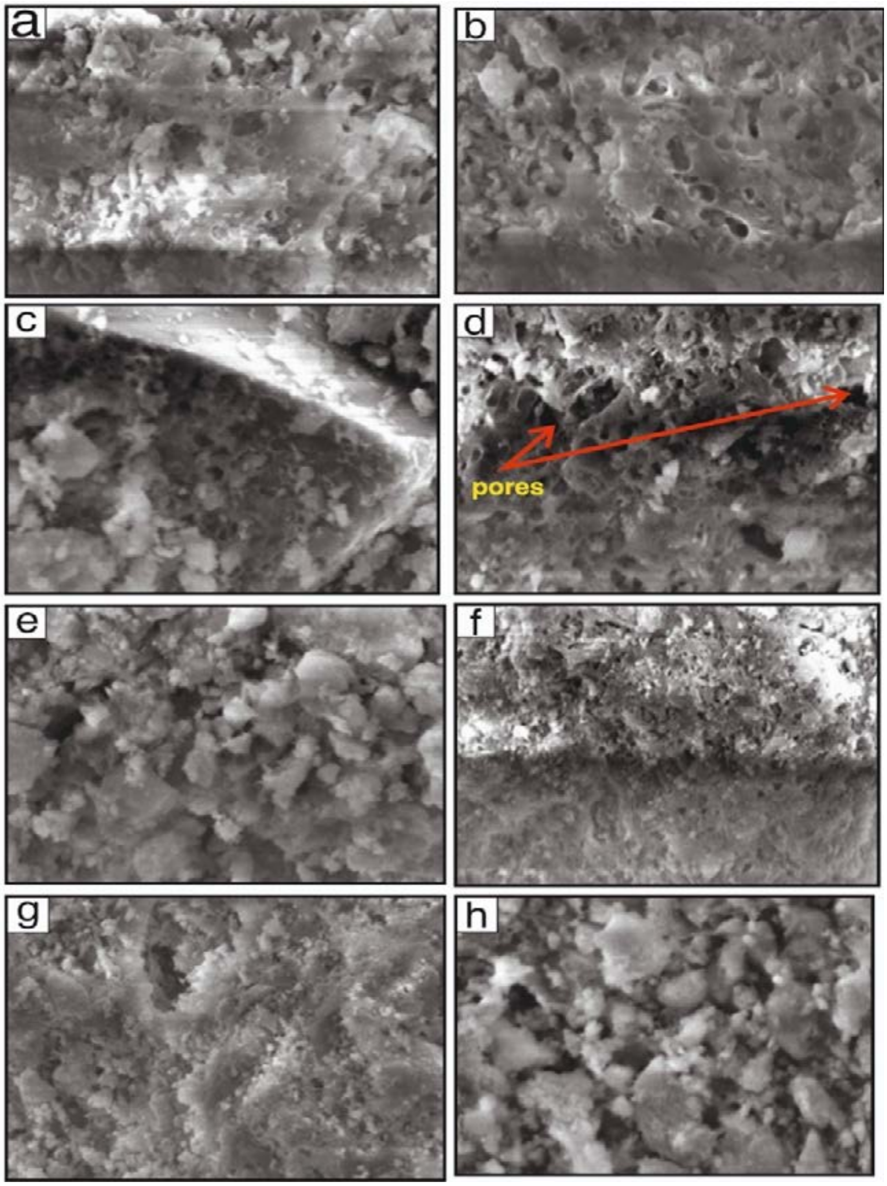

Fig. 5: Scanning electron microscopy images of fired bricks: a (indicator), b (B1), c (B2), d (B3), e (B3,5), f (B4), g (B5), and h (B6)

metals are below the permissible limit values for landfills. During the sintering process, most of the metal contaminants were fixed in the new minerals and converted into stable compounds and immobilized in the brick matrix during firing (Bernstein *et al.,* 2002). Thermal processes make the metal less leachable, thus the brick cannot harm the environment or human health (Wang *et al.,* 2012). In addition to the stabilization and limitation of heavy metal leaching through the solidification of bricks, dewatering of sludge is not mandatory, leading to the saving of energy (Świerczek *et al.,* 2018; Mladenovič *et al.,* 2017). According to other studies, metal leaching is insignificant for fired bricks incorporating sludge (Anik-Hasan *et al.,* 2022; Kulkarni *et al.,* 2019).

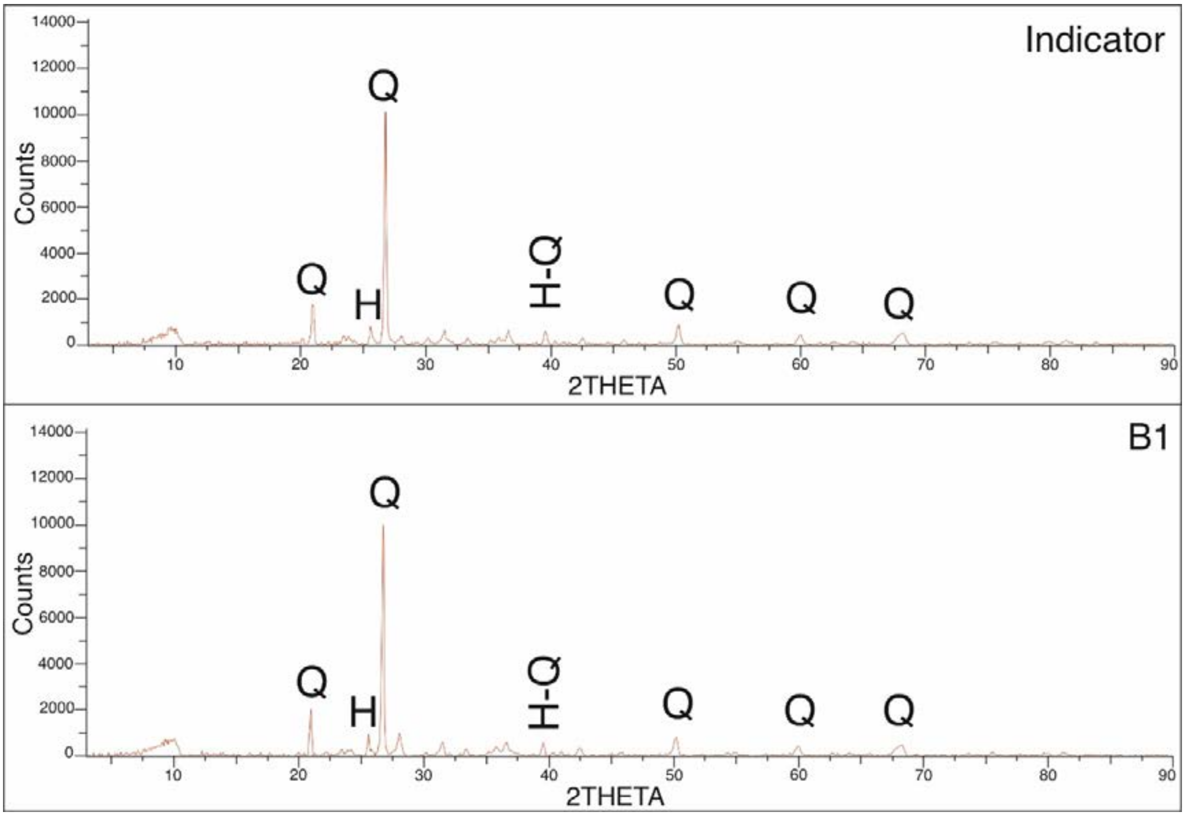

Fig. 6: X-ray diffractograms of the indicator brick sample containing 100% clay, and the B1 sample with 10% sewage sludge.

Table 3: Trace element concentrations milligram per liter (mg/L) in leachate from fired test bricks.

Brick samples	Arsenic	Cadmium	Chromium	Copper	Nickel	Lead	Zinc
B1 (10%)	≤0,01	≤0,005	0,075	0,04	0,037	≤0,005	≤0,008
B2 (20%)	≤0,01	≤0,005	0,085	0,04	0,038	≤0,005	≤0,008
B3 (30%)	≤0,01	≤0,005	0,013	0,05	≤0,005	≤0,005	≤0,008
B3.5 (35%)	≤0,01	≤0,005	0,138	0,05	≤0,005	≤0,005	≤0,008
B4 (40%)	≤0,01	≤0,005	0,077	0,04	0,028	≤0,005	≤0,008
B5 (50%)	≤0,01	≤0,005	0,106	0,04	0,026	≤0,005	≤0,008
B6 (60%)	≤0,01	≤0,005	0,099	0,05	0,026	≤0,005	≤0,008
Level 1[(a)]	0,5	0,04	0,5	2	0,4	0,5	4
Level 2[(b)]	2	1	10	50	10	10	50

[(a)] Limit values for waste in landfills for inert waste L/S=10.
[(b)] Limit values for waste in non-hazardous waste landfills L/S=10

CONCLUSION

The results from the present study support the idea that the WWTP sludge of Boukhalef in Tangier northern Morocco is suitable to be valorized into a construction material. The chemical composition, pollutant potential, physical, environmental, geotechnical characterization, and the mineralogical parameters indicate the feasibility of the incorporation of the sludge with clay as raw material in the production of clay bricks by extrusion, and give a global idea about the studied sludge. Indeed, the chemical (% major elements) and mineralogical composition of the sludge and those of the clay used for the manufacture of construction products are consistent and comparable and in compliance with the standards. The values found reported that the sludge has an assorted composition, notably SiO_2 with a concentration of 31.6%, 5.75% CaO, 3.73% Fe_2O_3, and traces of other elements. The WWTP sludge can be considered as a potentially interesting addition to the manufacture of bricks. The Pi results show that the sludge has a highly plastic nature (Pi > 40), while the clay is classified as plastic clay (15 < Pi < 40). Based on the methylene blue values, sludge studies are classified as a silty–clayey soil, while clay is classified as a clay soil. These empirical tests provide essential data on whether the sludge is plastic, can form a binding paste, is impermeable, and is compact like clay. The production of cylindrical bricks was conducted for mixtures of sludge and clay with different substitution rates. After sintering, the fine mineral particles increased and are bound together to form a compact product. According to the results obtained, the porosity, water absorption, and the bulk density are dependent on the substitution rate of the sludge. Firing time, temperature, and the proportion of sludge added are the factors that initially determine the quality of the building brick. The organic content of the sludge has a significant effect on the strength and performance of the material. The optimum substitution rate of clay in the brick formulation by sludge was evaluated by means of physical and mechanical tests. These obtained results ensure that the mechanical performance of the samples is similar to that of the reference brick for a substitution rate of 10% (B1), and from 20% (B2) of substitution, the compressive strengths of the sludge bricks decreased. This can be attributed to the high presence of organic matter in the sludge, which increases with the substitution rate; this burns during the firing process due to thermal destruction and tends to be depleted and create pores in the finished product. These increase the humidity of the brick and weaken its strength. As a result, the brick becomes more porous, exceptionally light, and less resistant. The average results of the tests of the resistance of the brick are situated between 25.9 MPa and 15.55 MPa for an incorporation not exceeding 20%. The morphology of the brick samples is unsuitable and not consistent with the control product except for B1 containing 10% of sludge, which is remarkably similar to that of the reference sample. Based on the most noteworthy results, an addition of 10% sewage sludge to 90% natural clay was selected as the best choice for this incorporation. Other reviews have selected between 10% and 15% sludge to be incorporated or even more, depending on the nature of the biosolid used and the addition of further additives such as shale, recycled glass, sludge ash, and wood. The leaching test showed exceptionally low concentrations of trace metals in the brick leachates confirming the immobilization of these elements in the ceramic matrix during firing, which represents a potential environmental benefit. These results demonstrate that the sludge is not a hazardous waste, noting the absence of metal pollution, which encourages using this sludge in a building material without any prior treatment. The WWTP sludge is a promising source of local raw material for the fired brick manufacturing sector. The incorporation of natural clay and sludge allows to obtain a lighter brick than the conventional one. This study shows an economically and ecologically innovative method for the elimination of sludge by recycling it into ceramic materials. In Morocco, bricks are often used in the construction of buildings, and the sludge is mostly landfilled or used for agricultural spreading, which are the least favorable pathways. The purpose is to evaluate the production of bricks with waste additive, innovating an effective way of urban sludge elimination, exploiting its properties for the manufacture of a widely usable element for construction. This technique of station's sludge valorization in an excellent quality brick proves to be interesting for an operational and clean choice of elimination contributing to the management of a waste inevitably produced, putting in place an environmental and socio-economic approach meeting the requirements of sustainable development and circular economy.

AUTHOR CONTRIBUTIONS

K. Moulato as the corresponding author, contributed to the conceptualization of the article, prepared all the figures, worked on the interpretation of results, wrote the original version, participated in the analysis of the raw materials used as well as in the manufacture of the bricks in the laboratory, and performed the data analysis. M. Ammari provided the necessary resources, adopted the methodology that was followed, and supervised the execution of the work and the final article. L. Ben Allal revised and edited the paper, made the final visualization, and gave the final suggestions.

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CONFLICT OF INTEREST

The author declares that there is no conflict of interests regarding the publication of this manuscript. In addition, ethical issues, including plagiarism, informed consent, misconduct, data fabrication and/or falsification, double publication and/or submission, and redundancy have been completely observed by the authors.

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ABBREVIATIONS

θ	Theta angle
<	Inferior
>	Superior
°C	Degree Celsius
%	Percent
µm	Micrometer
AFNOR	Association française de normalisation
Al_2O_3	Alumina
$Al_2Si_2O_5(OH)_4$	Kaolinite
As	Arsenic
B.E.T	Brunauer–Emmett–Teller
B1	Brick sample with 10% sludge
B2	Brick sample with 20% sludge
B3	Brick sample with 30% sludge
B3.5	Brick sample with 35% sludge
B4	Brick sample with 40% sludge
B5	Brick sample with 50% sludge
B6	Brick sample with 60% sludge
$CaCO_3$	Calcium carbonate
$CaMg(CO_3)_2$	Dolomite
CaO	Calcium oxide
Cd	Cadmium
CO_2	Carbon dioxide
COD	Chemical oxygen demand
BOD	Biochemical oxygen demand
Cr	Chromium
Cu	Copper
EDX	Energy dispersive X-ray
EN	European Norm
Fe_2O_3	Ferric oxide
g/cm^3	Gram per cubic centimeter
HCl	Hydrogen chloride
ICP-AES	Inductively coupled plasma atomic emission spectrometer
K_2O	Potassium oxide
KV	Kilovolt
L/S	Liquid/ solid

LL	Liquidity limit
Loi	Loss on ignition
mA	Milliampere
MgO	Magnesium oxide
Mg/L	Milligram per liter
mgO_2/L	Milligrams of oxygen per liter
mg/kg	Milligram per kilogram
$mm^3/year$	Cubic millimeter per year
m^3/day	Cubic meter per day
m^3/m	Cubic meter per meter
mm	Millimeter
ms/cm	Millisiemens per centimeter
m^2/g	Meter square per gram
Mn	Manganese
MnO_2	Manganese dioxide
MPa	Megapascal
Na_2O	Sodium oxide
Na_2CO_3	Sodium carbonate
NF-P	Norme Française - Bâtiment, génie civil
Ni	Nickel
Pb	Lead
PL	Plasticity limit
P_2O_5	Phosphorus pentoxide
pH	Potential of hydrogen
Pi	Plasticity index
SEM	Scanning electron microscopy
SiO_2	Silicon dioxide
SO_3	Sulfur trioxide
TFZ	Tangier Free Zone
TiO_2	Titanium dioxide
WWTP	Wastewater Treatment Plant
XRD	X-ray Diffraction
XRF	X-ray fluorescence
Zn	Zinc

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