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Manufacture of sustainable fired shale bricks using sewage sludge as raw material

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Abstract

This paper assessed the use of shale collected from western China and the incorporation of sewage sludge for manufacturing fired shale bricks, which could reduce the environmental impact produced in the mining process of traditional raw materials. Chemical constituents and mineral composition of shale and sewage sludge were characterized by x-ray diffractometer (XRD) and x-ray diffractometer (XRD), the analysis indicated that only the shale was a fluid bloating material, while the sewage sludge could only be used as additive. Fired shale brick specimens containing 0%–30% (wt) of sewage sludge were fired at temperatures ranging from 920 to 950 °C. The tests revealed that with the increase of replacement ratio of sewage sludge, the water content and shrinkage rate increased, whereas plasticity index, bulk density and compressive strength decreased. Comprehensive considerations of the physical and mechanical properties of specimen, the optimum replacement ratio of sewage sludge and sintering temperature were determined to be 15% (wt) and 940 °C, respectively. The tests showed that the highest amounts of micropores, which could improve the thermal insulation properties of the specimen. Through the analysis of the experimental results, it could be concluded that the addition of sewage sludge resulted in the successful manufacture of qualified fired shale bricks.

1. Introduction

In order to reduce the impact on the environment, one of the problems that must be solved in modern society is how to improve resource efficiency. Along with the advancement of urbanization, a lot of resources are used to manufacture building materials, and the building material industry presents enormous potential for the sedimentary and low-grade metamorphic rocks (e.g., clays and tuffs) \([1–4]\) and industrial waste (e.g., water treatment sludge, municipal sludge and tannery sludge) \([5–7]\). These studies indicated that the industrial waste could be used as a partial substitute of sedimentary and low-grade metamorphic rocks in the manufacture of bricks.

Due to the good insulation performance and durability, fired clay brick is widely used as building materials in the world. However, the exploitation of clay has an adverse effect on the environment, while the use of industrial waste in the manufacture of building materials could reduce the consumption of natural resources and the cost of waste treatment. Accordingly, it is interesting to use industrial waste and other materials instead of clay in the manufacture of construction materials.

In China, wall materials account for about 70% of the whole building materials, and fired clay bricks play a leading role in wall materials. The annual production of fired clay bricks is about 600 billion pieces in China, which is equivalent to about 0.47 billion square meters of farmland is destroyed every year \([8]\), so it is necessary to choose a material instead of clay to manufacture fired bricks. The shale, created by dehydration and cementation of clay, is gradually being applied in building materials and constructions, such as the use of shale in...
The chemical composition of sewage sludge showed considerable amount of SiO$_2$. Shale was collected from Hongqiao Sewage Treatment Plant in Shuimogou District, Urumqi City, as the main raw material, while sewage sludge was used as a partial replacement of shale for producing micropores to West Mountain area of Urumqi.

In this study, shale and sewage sludge were analyzed chemically using x-ray fluorescence spectrometer (Bruker Company, Karlsruhe, Germany). The analysis results are shown in table 1. The results revealed that the shale was mainly comprised of SiO$_2$, Al$_2$O$_3$, Fe$_2$O$_3$ and CaO, and the percentage of fluxing agent (Fe$_2$O$_3$, CaO, MgO, K$_2$O and Na$_2$O) reached 11.39%, ensuring the sufficient viscosity of glassy phase during the high temperature sintering process. The chemical composition of sewage sludge showed considerable amount of SiO$_2$ (18.46%), and was associated with minor Al$_2$O$_3$ (6.14%) and, in a small quantity, Fe$_2$O$_3$ (2.39%), CaO (2.40%), MgO (2.38%) and SO$_3$ (2.46%). These results indicated that only the shale was feasible for manufacturing fired bricks, while the sewage sludge could only be used as additive.

2. Materials and methods

2.1. Materials

In this study, shale and sewage sludge (figure 1) were used to manufacture fired bricks. Shale was chosen as the main raw material, while sewage sludge was used as a partial replacement of shale for producing micropores to improve the self-weight and thermal insulation performance of fired shale bricks. The shale was obtained from West Mountain area of Urumqi (Xinjiang, China), and was sieved until it reached a particle size ≤1 mm. Sewage sludge was collected from Hongqiao Sewage Treatment Plant in Shuimogou District, Urumqi City (Xinjiang, China). Considering that the sewage sludge was difficult to be completely dried and needed to be broken after drying process, the sewage sludge was directly used to mix with shale after being compressed dehydration, which would reduce the production cycle and production cost.

2.1.1. Chemical characteristics

Shale and sewage sludge were analyzed chemically using x-ray fluorescence spectrometer (Bruker Company, Karlsruhe, Germany). The analysis results are shown in table 1. The results revealed that the shale was mainly comprised of SiO$_2$, Al$_2$O$_3$, Fe$_2$O$_3$ and CaO, and the percentage of fluxing agent (Fe$_2$O$_3$, CaO, MgO, K$_2$O and Na$_2$O) reached 11.39%, ensuring the sufficient viscosity of glassy phase during the high temperature sintering process. The chemical composition of sewage sludge showed considerable amount of SiO$_2$ (18.46%), and was associated with minor Al$_2$O$_3$ (6.14%) and, in a small quantity, Fe$_2$O$_3$ (2.39%), CaO (2.40%), MgO (2.38%) and SO$_3$ (2.46%). These results indicated that only the shale was feasible for manufacturing fired bricks, while the sewage sludge could only be used as additive.
The fluxing agent could help lower the material’s softening temperature\cite{24}. Due to the fact that the chemical composition of sewage sludge contained relatively greater amounts of CaO, MgO, K\textsubscript{2}O and Na\textsubscript{2}O, which could be used as fluxing agent in the firing process, the addition of sewage sludge might be an effective method to lower the softening temperature during the manufacture of fired shale bricks.

2.1.2. Mineralogical characteristics

The XRD pattern of shale (figure 2(a)) and sewage sludge (figure 2(b)) were obtained through x-ray diffraction (Rigaku Company, Akishima, Japan). The mineralogical composition of shale was composed of quartz (SiO\textsubscript{2}), calcite (CaCO\textsubscript{3}), albite (NaAlSi\textsubscript{3}O\textsubscript{8}), kaolinite (Al\textsubscript{4}Si\textsubscript{4}O\textsubscript{10}(OH)\textsubscript{8}) and illite (KA\textsubscript{1}3(SiAl)\textsubscript{4}O\textsubscript{10}(OH)\textsubscript{2}nH\textsubscript{2}O).

The diffraction pattern of sewage sludge presented as main quartz (SiO\textsubscript{2}), illite (KA\textsubscript{1}3(SiAl)\textsubscript{4}O\textsubscript{10}(OH)\textsubscript{2}nH\textsubscript{2}O), albite (NaAlSi\textsubscript{3}O\textsubscript{8}) and chlorite (Mg\textsubscript{3}Si\textsubscript{4}O\textsubscript{10}(OH)\textsubscript{2}Mg\textsubscript{3}(OH)\textsubscript{6}).

2.1.3. Thermal characteristics

The thermal behavior of the shale and sewage sludge is given in figure 3. For shale, an endothermic valley, attributed to the loss of moisture, was observed at approximately temperature 100 °C. As the temperature increased, the DSC curve was smoother, showing a trend of endothermic (400 °C–1000 °C). During this period, the crystal transformation of quartz occurred, the hydroxyl of illite and kaolinite escaped, the lattice was destroyed and the amorphous state was formed. The dehydration and decomposition of illite also occurred at this stage, and the iron in the illite lattice escaped in the form of hematite. The carbonate and various impurities decomposed and the crystal structure was destroyed. There was a large endothermic valley at temperatures between 1100 °C–1200 °C, indicating that the shale began to melt, the liquid phase was produced in large quantities, and the eutectic components in the billet absorbed a lot of heat during melting.

For sewage sludge, an endothermic valley appeared at the temperature 100 °C, which was caused by the evaporation of a large amount of free water. At temperatures between 300 °C–550 °C, the exothermic effect of sewage sludge was observed. At 500 °C temperature small exothermic spike was observed, related to the burning of organic matter. There was an endothermic valley at temperatures between 650 °C–750 °C, which was caused by the decomposition of minerals. Further exothermic effect was observed at temperatures between 950 °C–1050 °C, which was related to exothermic crystallization of non-static substances.

2.2. Preparation of samples

2.2.1. Mix proportions

In this study, sewage sludge was used to manufacture fired shale brick as additive. In order to investigate the optimum proportion of the additive, different replacement ratios of sewage sludge are given in table 2.
As presented in the table, the specimens are identified as mS-nSS, where S and SS denote shale and sewage sludge, respectively. In addition, m refers to the proportion of shale and n denotes the replacement ratio of sewage sludge. For instance, 85S-15SS indicates that the replacement ratio of sewage sludge is 15%.

**Figure 2.** XRD pattern of (a) shale and (b) sewage sludge.

**Figure 3.** Differential scanning calorimetry (DSC) of shale and sewage sludge.
2.2.2. Sintering

In order to determine the sintering temperature of the fired shale brick using sewage sludge as additive accurately, the optimum sintering temperature (OST) of shale should be investigated. The sintering temperature, heating rate and soaking time of brick samples for the sintering tests are given in Table 3.

The calorific value of sewage sludge was higher, which helped to reduce the sintering temperature of brick. Therefore, the sintering temperature of specimen should be adjusted based on the replacement ratio of sewage sludge, as given in Table 4. The sintering process of fired shale brick with sewage sludge additive was still in accordance with the requirements of Table 3.

2.3. Methods

2.3.1. Water content and plasticity index test

It was experimentally proved that the effect of water content on the mixture properties should be considered [25]. According to Chinese code GB/T 50123–2019 [26], liquid limit (LL) and plastic limit (PL) experiments were carried out in this paper to study the influence of water content on mixtures of shale and sewage sludge physically.

Put the sample into a bowl. A 76 g cone was gently placed on the surface of the mixture and sunk into the mixture under the action of its self-weight. When the cone sunk into the mixture to a depth of 10 mm after 15 s, the water content was determined to be the liquid limit value.

For plastic limit experiment, ball-shaped mixture was rolled to the shape of cylindrical pasta. If cracks appeared when the diameter of cylinder was about 3 mm, the water content of the mixture was established as the plastic limit value.

The plastic performance of the mixture was expressed by plasticity index (PI), which was the difference of the LL and PL, as given in equation (1):

\[ PI = LL - PL \] (1)

2.3.2. Shrinkage rate test

Based on ASTM standard C326–2009 [27], drying shrinkage rate and firing shrinkage rate were determined using equation (2)–(3):

Table 2. Mix proportion of the brick samples.

| Sample       | Shale | Sewage sludge |
|--------------|-------|---------------|
| 100S         | 100   | 0             |
| 95S-5SS      | 95    | 5             |
| 90S-10SS     | 90    | 10            |
| 85S-15SS     | 85    | 15            |
| 80S-20SS     | 80    | 20            |
| 75S-25SS     | 75    | 25            |
| 70S-30SS     | 70    | 30            |

Table 3. The temperature, heating rate and soaking time.

| Samples            | Temperature range (°C) | Heating rate (°C min⁻¹) | Soaking time (min) |
|--------------------|------------------------|-------------------------|-------------------|
| Brick specimens    | 0–400                  | 4                       | 100               |
|                    | 400–600                | 1                       | 200               |
|                    | 600–900                | 3                       | 100               |
| 900–Sintering temp | 1                      | 50                      |
| Sintering temp     | 0                      | 180                     |

Table 4. The sintering temperature of bricks with different replacement ratios of sewage sludge.

| Mix proportion | 100S | 95S-5SS | 90S-10SS | 85S-15SS | 80S-20SS | 75S-25SS | 70S-30SS |
|----------------|------|---------|----------|----------|----------|----------|----------|
| Sintering temp | OST  | OST-10  | OST-10   | OST-20   | OST-20   | OST-20   | OST-30   |
\[
\text{Drying shrinkage rate (\%) } = \frac{(L_p - L_d)}{L_p} \quad (2)
\]
\[
\text{Firing shrinkage rate (\%) } = \frac{(L_p - L_f)}{L_p} \quad (3)
\]

where \(L_p\) is the plastic length of the green brick, \(L_d\) is the dry length of green brick, \(L_f\) is the length of the fired brick.

2.3.3. Bulk density test

The bulk density was determined according to ASTM C134–95 [28], and was calculated using equation (4):

\[
B = \left( \frac{d}{l \times w \times t} \right)
\]

in which \(B\) is the bulk density (kg m\(^{-3}\)), \(d\), \(l\), \(w\) and \(t\) are dry weight (kg), length (m), width (m) and thickness (m) of the specimen, respectively.

2.3.4. Water absorption test

The water absorption of the brick was measured as specified by ASTM C67–14 [29]. The dry, cooled specimens were submerged in clean water at 15.5 °C–30 °C for 24 h. In this test, the specimens were removed and wiped off the surface water. The complete weighing of the specimens should be within 5 min after removing the specimens from the bath. The calculation of the water absorption was given as follows:

\[
\text{Water absorption (\%) } = \frac{100(W_i - W_d)}{W_d}
\]

where \(W_i\) and \(W_d\) are the dry weight and saturated weight of the specimens, respectively.

2.3.5. Compressive strength test

Operations were carried out to assess the mechanical performance of fired bricks. The specimens were tested as denoted by Chinese code GB/T 2542–2012 [30]. Processes were carried out using compression testing machine connected to the computer. The compressive strength value was calculated using the following equation (equation (6)):

\[
P = \frac{F}{A}
\]

where \(P\) is the compressive strength of the bricks (MPa), \(F\) is the maximum load of the testing machine (N), \(A\) is the average of the areas of the upper and lower bearing surfaces of the brick (mm\(^2\)).

2.3.6. Coefficient of thermal conductivity test

Bricks were usually used as raw materials of the exterior walls of buildings, thus the thermal insulation properties should be determined to improve the building efficiency. The thermal conductivity coefficient of brick was tested by infrared thermal imager (Infratest Electronics CO., Ltd, Shanghai, China) as denoted in Chinese code GB/T 10295–2008 [31]. The coefficient of thermal conductivity was obtained from equation (7):

\[
\lambda = \frac{t_w - t_0}{(t_i - t_0)R_w} \cdot d
\]

in which \(t_i\) is the temperature of incubator (K), \(t_0\) is ambient temperature (K), \(t_w\) is external surface temperature of brick (K), \(R_w\) is heat transfer resistance of the external surface of brick (m\(^{-2}\)·K W\(^{-1}\)), and \(d\) is the thickness of the brick (m).

3. Results and discussions

The optimum sintering temperature of shale was determined firstly. Then in order to promote the industrialized production of fired shale bricks using sewage sludge as additive, the water content, plasticity index and drying shrinkage rate of green brick samples, the firing shrinkage rate, bulk density, compressive strength, and thermal conductivity of fired brick samples were tested. Obvious defects, such as cracking and deformation, were observed in the brick samples with 30% (wt) of sewage sludge. Therefore, the performance tests were mainly carried out for brick samples whose replacement ratios of sewage sludge were lower than or equal to 25%.

3.1. The optimum sintering temperature of shale

In order to obtain the optimum sintering temperature of shale, the sintering temperatures were determined to be 800 °C, 850 °C, 900 °C, 950 °C, 1000 °C, 1050 °C and 1100 °C through the analysis of thermal characteristics of shale (figure 3). The cured specimens are given in figure 4. During the firing process, there were no fissures and efflorescence defects could be observed in the specimens. The color of the brick samples was light orange, and the surface of the samples was rough and dull at the temperature 800 °C. Following the increasing of sintering temperature, the color of brick samples tended to be darker.
The water absorption (WA) and firing shrinkage (FS) of brick samples are given in figure 5. Based on Chinese code GB 26538–2011 [24], the water absorption of fired bricks should not be larger than 20.0%, indicating that the minimum sintering temperature was about 850 °C. The firing shrinkage of brick samples was −0.56 and the water absorption increased significantly at the sintering temperature 1100 °C, showing that the brick samples were overburned. Therefore, the highest sintering temperature was approximately 1050 °C.

Figure 6 gives the XRD pattern of shale at different sintering temperatures. The contents of quartz and albite in the brick samples decreased as the sintering temperature increased. Liquid phase appeared at the contact part of albite and quartz at the temperature 900 °C, and the feldspar and quartz content decreased. Following the increase of sintering temperatures, quartz content decreased and part of the quartz crystals were melt in the glass phase of the samples, and mulita would be formed because of the reaction between the quartz and alumina. Finally, a large amount of glass phase was formed at 1050 °C, indicating that the sintering temperature should not be higher than 1050 °C.

According to the study of He [32] and figures 4–6, the optimum sintering temperature of shale was calculated from the equation:

$$ T = T_s + (T_c - T_s) / 2 $$

(8)

Where $T_s$ is initial sintering temperature and equals to 850 °C, $T_c$ is melting temperature and equals to 1050 °C.

It could be obtained that the optimum sintering temperature of shale used in this paper was about 950 °C.
3.2. Performance of fired shale bricks with sewage sludge

3.2.1. Effects of sewage sludge content on water content and plasticity index of green bricks

The water content and the plasticity index of green brick were considered as important factors for the shaping of bricks. Figure 7 shows the measured results. The water content of green brick tended to increase with the increase of sewage sludge content, while the increase of sewage sludge content led to the decrease in plasticity index. Results showed that when the sewage sludge content increased from 0% to 25%, the water content increased from 19.00% to 24.21%, the plasticity index decreased from 12.22% to 7.02%. Following the increase of sewage sludge content, the forming of green bricks became very difficult, thus the maximum sewage sludge content was determined to be 25%. This is because:

- The organic fibers in sewage sludge absorbed a large amount of water, which reduced the free water in the shale-water two-phase system and weakened the attraction between adjacent particles of the shale, thus reducing the forming ability of the mixture.

- The excessive moisture of the sewage sludge affected the mixing uniformity of the sewage sludge and shale significantly. Therefore, following the increase of sewage sludge content, the plasticity index of the green brick decreased.

Figure 6. XRD pattern of shale under different sintering temperatures.

Figure 7. Effects of sewage sludge content on water content and plasticity index of green bricks.
3.2.2. Effects of sewage sludge content on the drying shrinkage rate and firing shrinkage rate

The drying shrinkage rate and firing shrinkage rate affected the manufacture of the production mold and the outer dimensions of the specimen. Figure 8 presents the test results for shrinkage rate. The highest shrinkage rate was observed in specimens with 15% content of sewage sludge additive. In this case, the drying shrinkage rate and firing shrinkage rate increased 16.6% and 58.8%, respectively. This was due to the fact that the water content required for brick forming increased with the increase of replacement ratio of sewage sludge, and the evaporation of water led to the shrinkage of brick during the drying and sintering process.

3.2.3. Effects of sewage sludge content on bulk density and compressive strength of brick

Smaller bulk density of brick helped to reduce the building self-weight and construction cost, and adequate compressive strength of brick was a necessary condition for its application in engineering. The test results of bulk density and compressive strength of bricks are given in Figure 9. The bulk density produced with 0% sewage sludge exhibited a value of 1770 kg m$^{-3}$, this value reduced to 1230 kg m$^{-3}$ at the 25% replacement ratio. This revealed that the high loss on ignition of sewage sludge resulted in a large number of pores after firing, which effectively reduced the bulk density of bricks.

The compressive strength of the brick decreased rapidly with the increase of sewage sludge content. The compressive strength was 13.95 MPa for the 0% replacement ratio, and when the sewage sludge content was 25%, the compressive strength decreased to 3.00 MPa. This situation was due to the reason that the number of pores increased with the increase of sewage sludge content, which reduced the cross-sectional stress area of the brick.

According to Chinese code GB 50003–2011 [33], the compressive strength of fired bricks should not be less than 5.0 MPa, while the compressive strength was 4.42 MPa for the 20% replacement ratio. Therefore the
replacement ratio of sewage sludge should not be more than 15%. Considering that the other parameters satisfied the requirements of manufacturing and application of fired brick, the optimum replacement ratio of sewage sludge was determined to be 15%.

3.3. Microstructure of fired shale bricks
The microstructure of the fired shale bricks (figure 10) was studied by SEM (TESCAN Company, Brno, Czech). Compared to the specimen without sewage sludge, the microstructure’s view of the ceramic body with 15% of sewage sludge additive showed that the density of the microstructure was smaller and the amount of micropores was larger. High porosity was also confirmed by the chemical characteristic obtained earlier in table 1. The earlier investigations of physical and mechanical performance of specimens also confirmed the same results: for the 0% and 15% replacement ratio of sewage sludge, density strength decreased from 1770 to 1380 kg m\(^{-3}\), compressive strength decreased from 13.95 to 8.23 MPa (figure 9).

3.4. Thermal conductivity of fired shale bricks
The testing equipment and the test results are given in figures 11 and 12. The thermal conductivity coefficient of fired shale brick was 0.45 W (m K\(^{-1}\)) for the 0% replacement ratio, 0.19 W (m K\(^{-1}\)) for 15% replacement ratio of sewage sludge. The addition of sewage sludge generated more pores inside the brick, which could block the transfer of heat and improve the thermal performance of the brick. Compared with fired clay brick (\(\lambda = 0.81\) [34]), the thermal conductivity coefficient of the fired shale brick decreases obviously with or without the addition of sewage sludge.
4. Conclusions

The effect of different content of sewage sludge on the properties of green or fired shale bricks were examined experimentally. It was found that the physical, mechanical and thermal performances as well as microstructure of the bricks were affected apparently by the additive of sewage sludge. The main observations are summarized as follows:

1. According to the analysis of XRF, shale (large amount of SiO₂ and Al₂O₃) was a fit bloating material for manufacturing fired bricks independently, while the sewage sludge could only be used as additive to improve the properties of the bricks.

2. After the introduction of fired shale bricks without the sewage sludge additive at 800 °C–1100 °C temperature, the ceramic body with the following parameters was created: the firing shrinkage 0.51%–(−1.16)% and water absorption 22.05%–13.86%. Taking into account of the XRD and thermal characteristics, the optimum sintering temperature of shale was determined to be 950 °C.

3. After the introduction of 0%–30% of sewage sludge additive into shale mixture and the burning of the bricks at 920 °C–950 °C temperature, the following parameters of green or fired bricks was created: the water content 19.00%–24.21%, plasticity index 12.22%–7.02%, drying shrinkage 4.22%–5.15%, firing shrinkage 0.97%–1.36%, bulk density 1770–1260 kg m⁻³ and compressive strength 13.95–3.00 MPa. Considering the requirements of manufacture and engineering application of bricks, the optimum replacement of sewage was determined to be 15%.

4. As could been seen from the SEM images of the bricks, the microstructure of the brick was disrupted by the sewage sludge. The microstructure of reference specimen was denser than the microstructure of specimen containing 15% of sewage sludge, the results could also be seen from the results of the physical and mechanical tests.

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Data availability statement

The data generated and/or analysed during the current study are not publicly available for legal/ethical reasons but are available from the corresponding author on reasonable request.
Conflict of interest statement

We declare that we do not have any commercial or associative interest that represents a conflict of interest in connection with the work submitted.

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