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RESEARCH ARTICLE

A study on the oil-based drilling cutting pyrolysis residue resource utilization by the exploration and development of shale gas

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Abstract Based on the requirement of national energy conservation and environmental protection, attention has been given to building an environment-friendly and resource-saving society. Shale gas oil-based drilling cutting pyrolysis residues (ODPRs) have been used as the main research object to developing new technology which can convert the residues into a harmless and recyclable material. Using the test data of ODPR, we analyze the development prospect in the building material industry and provide a scheme to utilize this particular solid-waste efficiently. Theoretically speaking, the ODPR resource utilization such as admixture of cement, making sintered brick, and non-fired brick, by the exploration and development of Fuling shale gas is feasible.

Keywords Shale gas · ODPR · Recycling · Building materials · Feasibility

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Introduction

With the rapid development of China's economy, energy demand has been constantly increasing, and shale gas, as new type resources of energy, has become one of the major motivations in the economic development of China. In the course of exploration and development of shale gas, we use oil-based drilling fluid of multi-interval fracturing technology in horizontal well for gas discovery, and then, oil-based drillings circulate the ground which include oil-based cuttings and a portion of oil-based mud. The pre-processes of oil-based drilling fluid and cuttings are presented in Fig. 1. All the processes are mainly through the vibrating screen system, which makes the oil-based drilling cuttings available. In one well, about 250-m^3 oil-based drilling cuttings would be generated. Because it is a kind of oily solid waste, pyrolysis procedures are used to ensure the safe discharge. The oil rate of oil-based residues will be controlled within 0.3%, which can guarantee solidification treatment (Control standards for pollutants in sludges from agricultural use 1984).

Add hardening agent such as cement in ODPR to convert it into solids with certain strength. In this way, the contamination of ODPR can basically be solved for a short period of time. Technically, through solidification (Ball et al. 2012; Kogbara et al. 2013, 2016; Kogbara 2014), the absolute majority of contaminants in the ODPR are mixed in the solidified blocks, so effective treatment can be achieved. However, this process only mixes the pollutants in the solidified blocks, rather than completely counteracts them. When buried underground for a long time, the solidified blocks under physical, chemical, and biological actions will undergo a series of variations, and thus result in secondary pollution (Leonard and Stegemann 2010; Antemir et al. 2010; Zhang et al. 2016). From Fig. 2, we can see that solidifying processes of ODPR is very tedious. There are

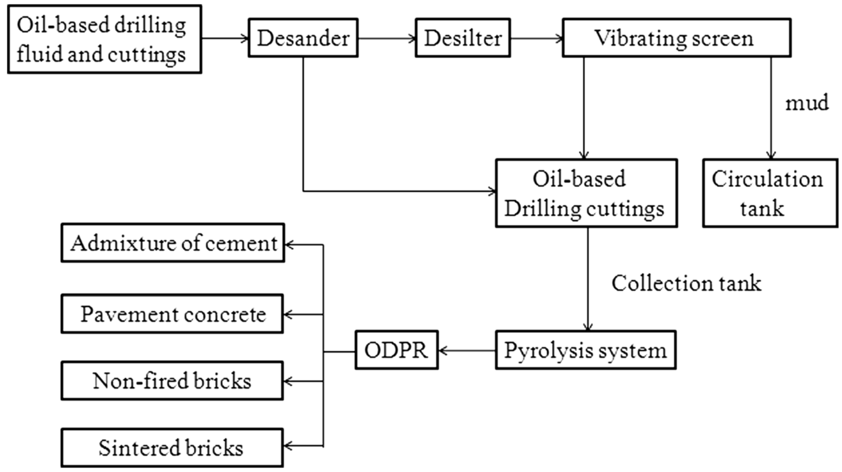

Fig. 1 Pre-processes of oil-based drilling cuttings and comprehensive utilization of ODPR

mainly through the first and second mixed with agent, then solidified in a consolidation tank. In addition, there are also some problems, for example, large area occupied and high cost during building. What is worse, if construction is not well controlled, there will be risks in environmental pollution. So, the ODPRs' safe and environmental dispose and resource recycling by the exploration and development of shale gas are in urgent need.

Therefore, the feasibility of ODPR used as a replacement material for producing building materials was investigated. We analyzed the mineral composition, particle size distribution, chemical composition, thermal analysis, etc., of ODPR. The technical mechanism of the various methods such as the admixture of cement (Bernardo et al. 2007; Mostavi et al. 2015; Al-Otoom 2006), making sintered brick (Li et al. 2011), non-fired brick (Tuncan et al. 2000; Lirong et al. 2015), and sintered brick, will be discussed as well. Based on the actual practice, we summarized the development prospect throughout the building material industry and provided insights into the proper utilization of ODPR generated by the shale gas industry in our country.

Experiments

Treatment processes of oil-based drillings

In the course of exploration and development of shale gas, about 250-m^3 oil-based drillings would be generated in a well. Oil-based drilling is a kind of oily solid waste, so it has to be pre-processed to ensure safe disposal. Besides, the drillings, usually dark black, contain a certain amount of diesel and different kinds of crushed rocks. Figs. 1, 2, 3, and 4 are the treatment process drawing and comprehensive utilization of ODPR. It is clear that the pyrolysis technique is the final method for disposal of oil-based drillings which through the high-temperature furnace, the oil rate of oil-based drilling residues will be controlled within 0.3% so that resource utilization can be applied.

Raw materials

The cement used in this study was commercial ordinary Portland cement 42.5 (P.O. 42.5) from Chongqing Huaxin Cement Co., Ltd. Oil-base drilling cutting pyrolysis residues (ODPRs) was

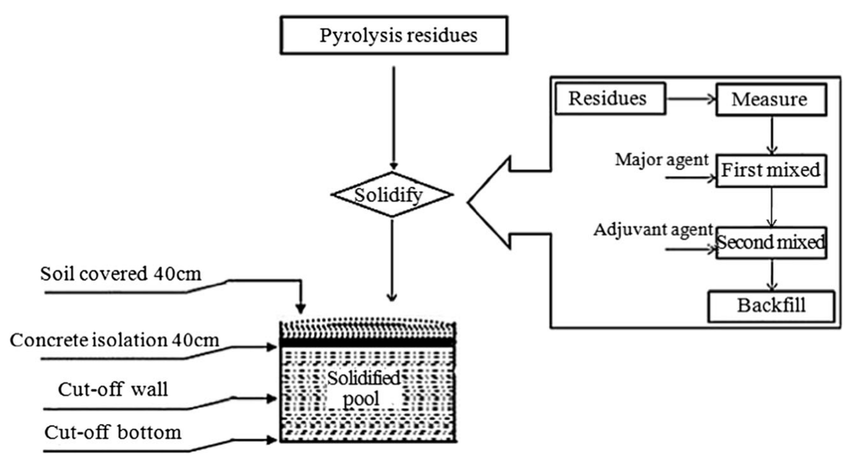

Fig. 2 Solidify processes of ODPR

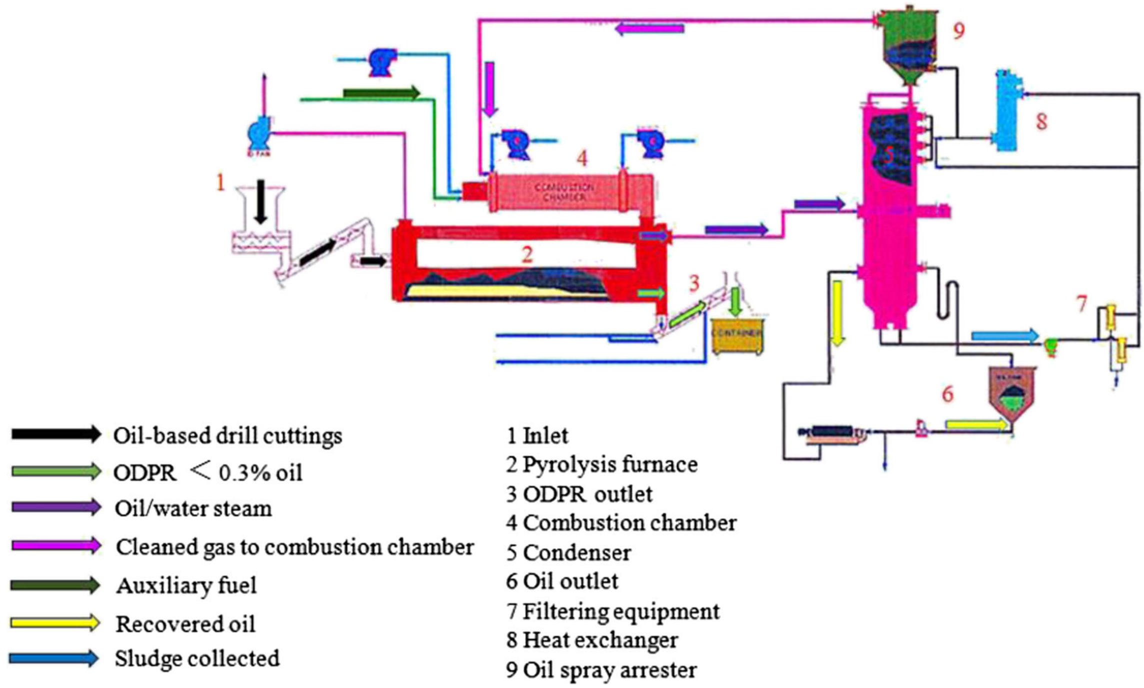

Fig. 3 Treatment processes of oil-based drillings

provided by Chongqing Fuling Shale Gas Exploration and Development Company, SINOPEC (China); fly ash and clay were provided by a power plant in Chongqing City (China). The chemical compositions of them are presented in Table 1.

Sample preparation

The pozzolanic activity of ODPR was measured by four methods (strength activity index test, crystalline analysis test,

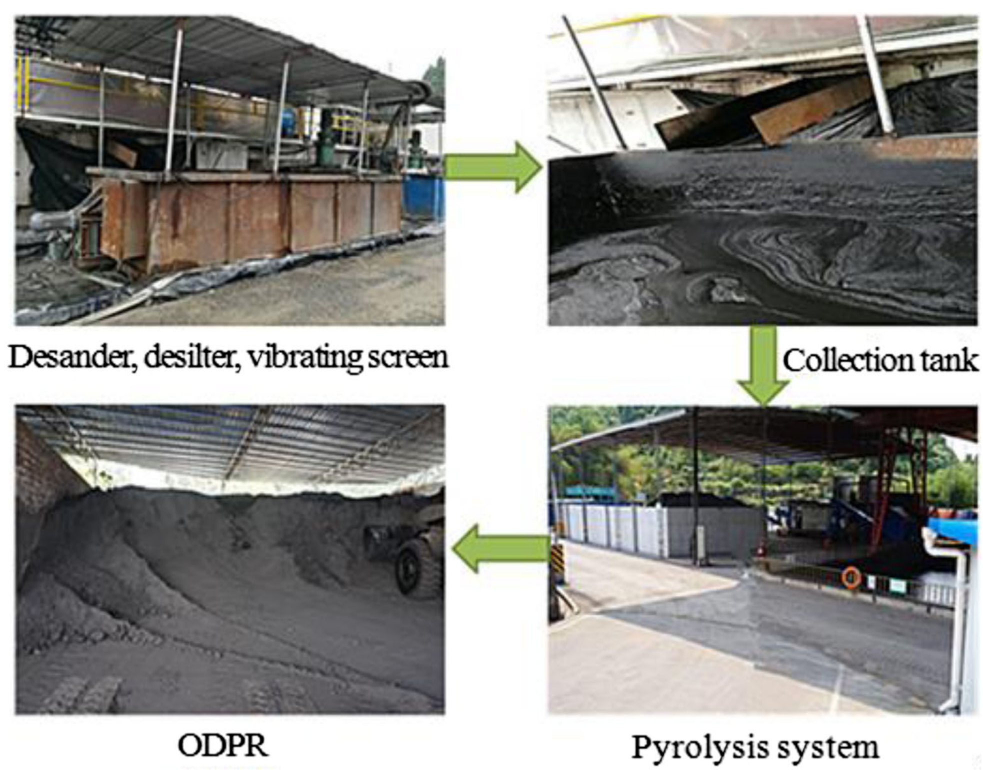

Fig. 4 Treatment processes of oil-based drillings

Table 1 Mixture ratio of pozzolanic material–$Ca(OH)_2$–H_2O system

ODPR content	Quick lime content	Water to binder ratio	Curing methods
50%	50%	9:1	60 °C constant temperature, 3 days

thermal analysis test, and bound water analysis test). A brief description of these methods is presented here.

Strength activity index

The compressive strength was assessed on mortar cubes (40 × 40 × 160 mm^3) made with standard sand (1:3) and constant water to binder ratio (*w*/*b*) of 0.50. Control mortar cubes were prepared using a normal Portland cement (PC) with a chemical composition reported in Table 2. The blended cement was composed by 30% *w*/*w* of ODPR and 70% *w*/*w* of Portland cement. Complementary, a pozzolan sample was prepared by using 30% *w*/*w* of ODPR as replacement of PC. The mortar was mixed in a planetary orbital mixer for 5 min, and the specimens were molded and compacted, which followed the standard procedure. De-molding was conducted after maintained in a curing box under (20 ± 1) °C and 95% relative humidity for (24 ± 2) h. At 28th day, the compressive strength (three cubes were tested at each age) was determined. Specimen molding and compressive strength test were operated ISO-679:1989. The results reported in this study are the average of three tests for mechanical properties. The compressive test was conducted by using the 3000-kN compression testing machine. Finally, the strength activity index (strength activity index (SAI) = A/B × 100%) was calculated as the ratio of the compressive strength of blended cement mortar (A) to the strength of the Portland cement mortar (B) at the same age, as percentage.

Crystalline analysis

MDI Jade 5.0 can be used to directly analyze the crystalline of ODPR's X-ray diffraction (XRD) test results. Then, the amorphous content of ODPR would be gotten, which can be represented by the pozzolanic activity.

Table 2 Chemical composition percent by mass

Materials	SiO_2	Al_2O_3	CaO	SO_3	MgO	Fe_2O_3	Loss
Cement	22.07	4.98	61.15	2.17	3.62	3.14	3.59
ODPR	55.42	7.90	7.12	0.97	1.58	2.07	8.44
Clay	57.76	19.38	0.35	–	0.5	8.66	9.68
Fly ash	45.7	18.6	20.4	2.80	0.98	5.98	6.99

Fig. 5 Appearance of ODPR

Thermal gravimetry and differential thermal analysis

The pozzolanic activity of the ODPR samples was determined according to unreacted calcium hydroxide by using thermal analysis (Netzsch STA 449 C). A paste with an appropriate concentration was made from a mixture of 50% ODPR and 50% quick lime, poured in a cylindrical container, and made completely sealed and airtight. The samples were treated in a dryer at 60 °C temperature for 3 days, and the control group was treated in a dryer at 25 °C temperature for 3 days. Afterwards, the samples were pulverized. The powders were exposed to thermal treatment up to 900 °C at a heating rate of 20 °C/min, and their DTG and TG curves were recorded. In the thermal analysis curves of thermal gravimetry and differential thermal analysis (TG-DTA), the temperature of the dehydroxylation of $Ca(OH)_2$ can be scoped. The higher the mass loss is, the more content of $Ca(OH)_2$ in the hydration products is, and the worse the pozzolanic activity of ODPR is. The samples' mixture ratio of pozzolanic material–$Ca(OH)_2$–H_2O system is listed in Table 1.

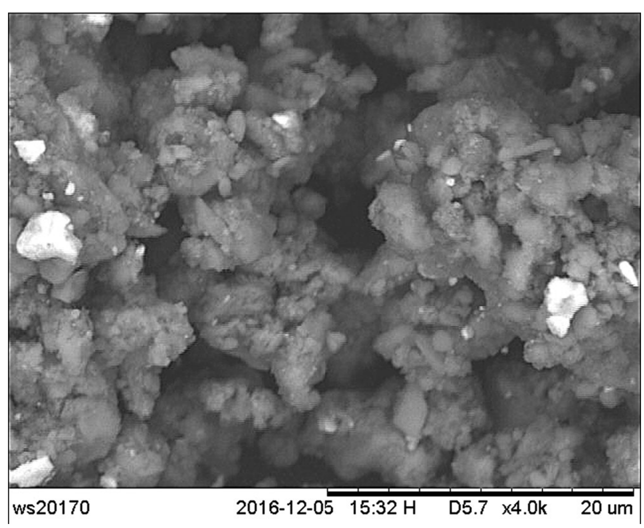

Fig. 6 SEM pattern of ODPR

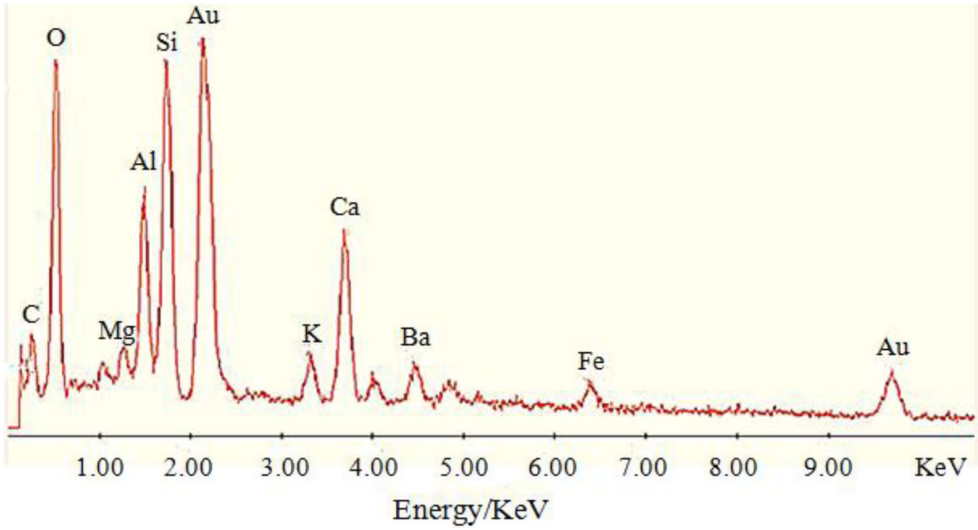

Fig. 7 Energy-dispersive X-ray spectroscopy (EDX) analysis of ODPR

Bound water analysis

A sample of 1000 g of hydrated power was accurately weighed out, which pre-dried to constant weight in 60 °C. Then, the powder is put in a crucible calcine to constant weight in 975 °C in muffle furnace, and cooled to constant temperature in a dryer. Finally, the bound water ratio $W = (C_1 - C_2)/C_2 - W_x/(1 - W_x)$ was calculated as the ratio of the bound water, where C_1 is the quality of hydrated power which is pre-dried to a constant weight in 60 °C, C_2 is the quality of 975 °C calcine, and W_x is the loss on ignition of unhydrated power. The samples' mixture ratio of pozzolanic material–$Ca(OH)_2$–H_2O system is listed in Table 1.

Testing methods

Chemical composition

Chemical analyses of raw materials were determined by the XRF (Axios-Advanced) techniques.

Particle size distributions

The particle size distributions (PSDs) were measured in ethyl alcohol suspension, which was carried out using a laser particle sizer (MASTERSIZER 2000, Malvern Ltd).

XRD analysis

The crystalline minerals of ODPR were identified, using XRD measurement in a X'Pert PRO diffractometer (PANalytical) with Cu Ka radiation and a position-sensitive detector. The accelerating voltage was 35 kV and the current was 60 mA. Diffraction peaks on the XRD spectrum were detected by software package X'Pert HighScore and Plus MDI Jade 5.0.

Scanning electron microscopy and energy-dispersive X-ray spectroscopy

Microstructure of the samples was analyzed with a scanning electron microscope (SEM; ASTEREO SCAN440, Leica Cambridge, Ltd.), which was used to investigate the morphology of ODPR particles.

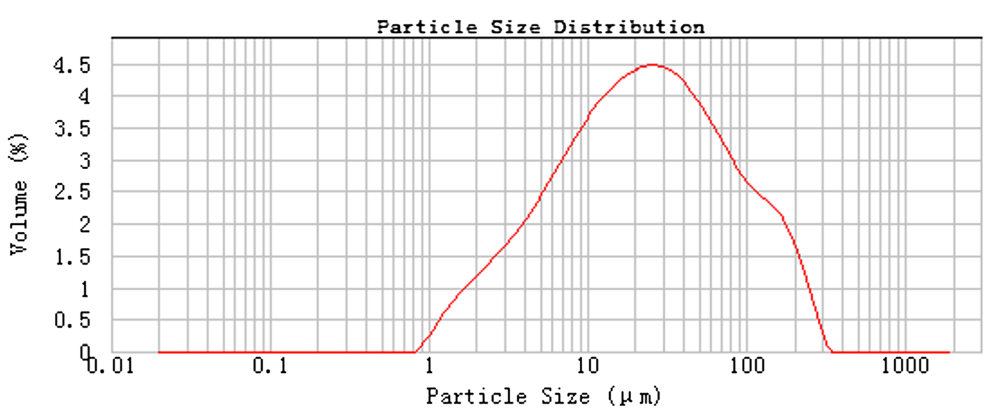

Fig. 8 Particle size distribution of ODPR

Table 3 Results of index of activity measurements

Test items	Loss	SO_3	Index of activity (H_{28}) = R_1/R_2
ODPR	8.44	0.97	71.5

Energy-dispersive X-ray spectroscopy (EDX) was analyzed with the aid of INCA x-sight Oxford IETEM100.

Thermal analysis

Thermal behavior of the ODPR including TG-DTA was examined by using analyzer (Netzsch STA 449 C). The samples were heated at a constant rate of 20 °C/min from ambient temperature to 900 °C.

Results and discussion

Physical properties of ODPR

Appearance, SEM, particle size distribution, and EDX of the ODPR are shown in Figs. 5, 6, 7, and 8.

According to Figs. 5, 6, and 7, we can observe that the ODPR is gray and arrives as a powder; it also contains Ca, Si, and Al. The appearance presents without rules and a state of laxity shape. The particle size distribution is shown in Fig. 8, pitch diameter (0.5) is 22.8 μm, and particle size distribution is unimodal and wide. The d_{max} of all ODPR is less than 0.2 mm; it makes them a priori convenient for a usage as sand and mineral addition substitute.

Chemical composition of ODPR

In order to compare and analyze the feasibility of ODPR resource utilization, the cement, ODPR, clay, and fly ash's chemical compositions are also presented in Table 2.

From Table 2, it can be concluded that the ODPR contains active CaO, SiO_2, and Al_2O_3, which are in accordance with the EDX analysis. It has self-cementitious property, which can be used as a new cementing material. The mechanism of self-cementing is based on two reactions: generation of calcium silicate hydrate (C–S–H) which by the reaction of active SiO_2 with CaO and generation of ettringite (AFt) which by the reaction of active Al_2O_3 with CaO (M.M. Radwan et al. 2013; Havlica et al. 2004; Ramos et al. 2014).

Activity of ODPR

The index of activity of ODPR

The results of strength activity index are presented in Table 3.

Table 3 shows that the index of activity of ODPR is 71.5%, greater than 65%; it indicates that ODPR has a certain pozzolanic activity. The loss on ignition and SO_3 content, i.e., 8.44 and 0.97%, respectively, meet the requirements of mineral admixture of cement, making sintered brick, non-fired brick, and sintered brick. Generally, the loss on ignition is below 10.0% and SO_3 content is 3.5%. So, the erection condition of a new cementing material has been already possessed.

X-ray diffraction pattern analysis

Figure 9 shows the randomly chosen three samples of XRD pattern experiment data.

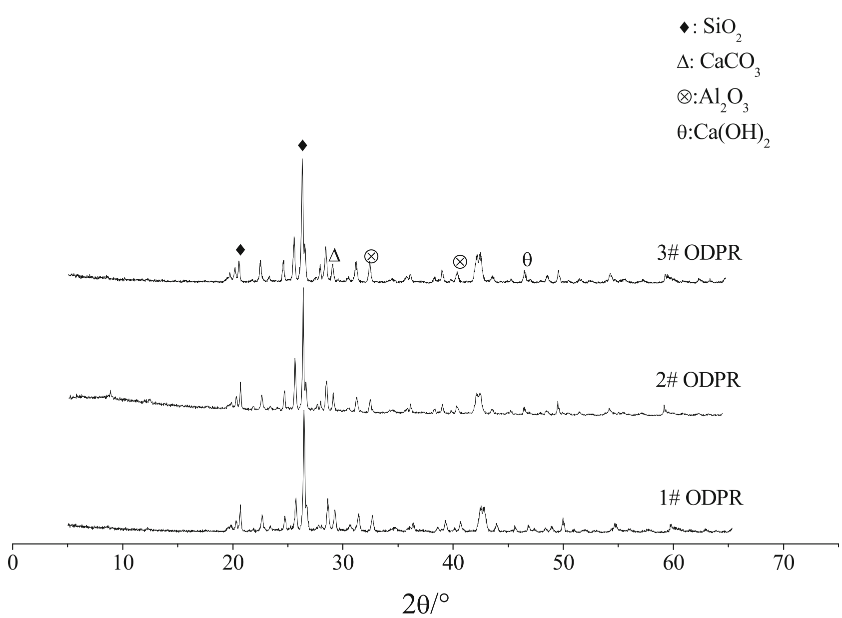

Fig. 9 XRD patterns of ODPR

Table 4 XRD results of amorphous phase content

No.	Amorphous phase content (wt.%)	Mineral
ODPR-1	33.41	Limestone, quartz, hematite
ODPR-2	31.18	Limestone, quartz, hematite
ODPR-3	32.73	Limestone, quartz, hematite

The XRD patterns of ODPR show the main minerals, including limestone, quartz, and hematite. Studies (Wang et al. 2014; Sheng et al. 2007; Fortes et al. 2016) have shown that α-SiO_2 of clay minerals have high stability of the amorphous structure; therefore, ODPR contains a certain amount of α-SiO_2 and Al_2O_3. So, its CaO content enables the formation of the CaO–Al_2O_3–SO_3 system. This self-cementitious property makes the residues suitable as a new cementing material.

Using MDI Jade 5.0 directly analyze the crystalline of ODPR's XRD test results. So, the amorphous contents of random test of ODPR are shown in Table 4. We can clearly see that the average value of amorphous content of ODPR is about 32%, which has a certain pozzolanic activity.

Thermal analysis

The thermal behavior of ODPR samples are presented in Figs. 10 and 11. The most significant changes according to TG and DTA analysis for starting samples are summarized below. The DTA pattern of ODPR (Figs. 10 and 11) showed an endothermic peak in the temperature range of 400–450 °C, and the weight-loss ratio of control group has reach about 20%, but the ODPR is only about 10%. That is referable to the de-hydroxylation of $Ca(OH)_2$, which from the area of endothermic can reflect the pozzolanic activity. This value of $Ca(OH)_2$ and H_2O obtains the amount of unreacted calcium

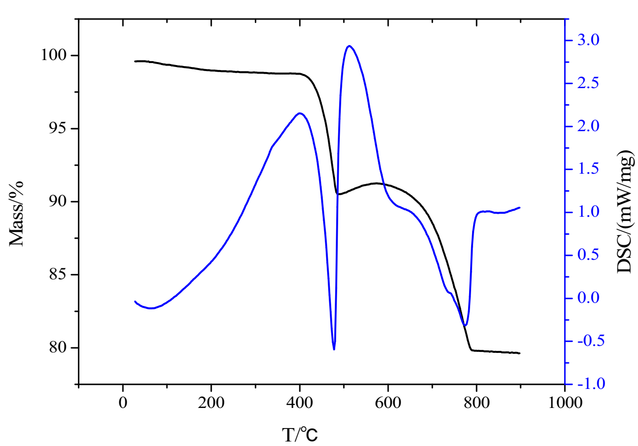

Fig. 10 Thermal analysis of ODPR–$Ca(OH)_2$–H_2O under 25 °C (control group)

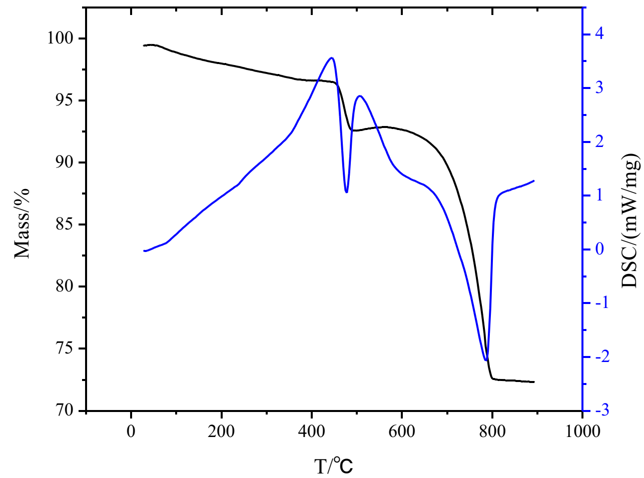

Fig. 11 Thermal analysis of ODPR–$Ca(OH)_2$–H_2O under 60 °C

hydroxide well. So, it can show that the ODPR has higher level of pozzolanic activity.

Bound water analysis

With the system of pozzolanic material–$Ca(OH)_2$–H_2O which reacts with the active SiO_2, it generates certain amount of gel C–S–H. By calcining the methods of 975 °C, we can measure the hydration rate of pozzolanic material. The higher the value of W is, the more amount of gel C–S–H is, and the stronger the pozzolanic activity of ODPR is. According to Table 5, it is clear that the average value of hydration rate of ODPR–$Ca(OH)_2$–H_2O under 60 °C is about 0.2, which is higher than the control group. So, from Figs. 9, 10, and 11 and Tables 3, 4, and 5, the ODPR has higher level of pozzolanic activity which can resource utilization.

Comprehensive utilization of ODPR

Admixture of cement

(1) Theoretical basis of the admixture of cement

The process flow sheet and production practice of the admixture of cement are given in Figs. 12 and 13. It is clear that

Table 5 Results of bound water content

No.	Bound water content (W)
ODPR–$Ca(OH)_2$–H_2O under 25 °C—control group	0.12
ODPR–$Ca(OH)_2$–H_2O under 60 °C—#1	0.21
ODPR–$Ca(OH)_2$–H_2O under 60 °C—#2	0.17
ODPR–$Ca(OH)_2$–H_2O under 60 °C—#3	0.19

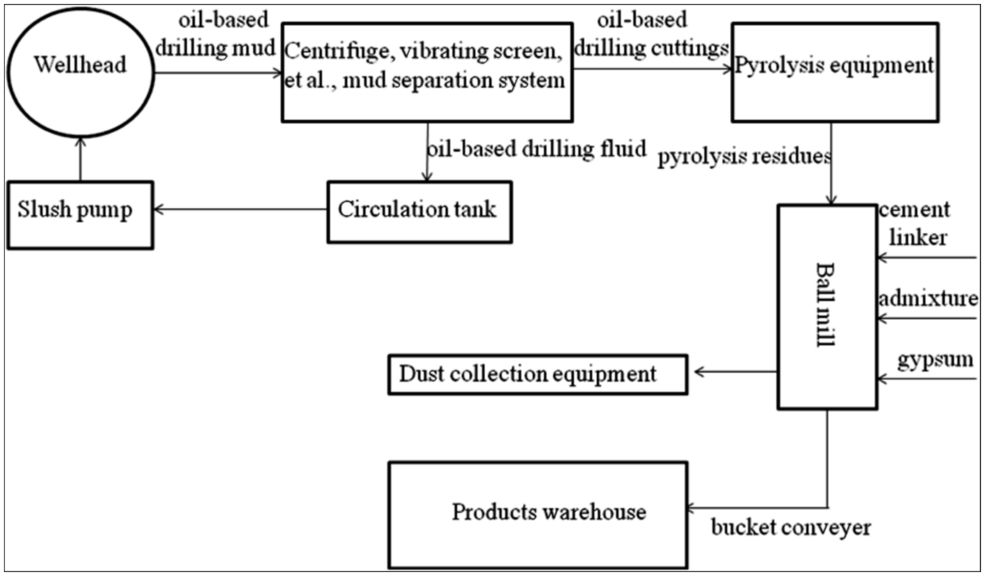

Fig. 12 Process flow sheet of the admixture of cement

while passing through an innocent treatment of pyrolysis processing, and then piping to ball mill with cement linker and other admixtures, the product which is transferred by the bucket elevator is repeatedly grabbed to product warehouse.

The mineral composition of ODPR is similar to clay and fly ash. It is similar to a siliceous material which can be used as a new cementing material (Bernardo et al. 2007; Ramos et al. 2014). ODPR contains high volume of SiO_2 and Al_2O_3, and mostly in amorphous form. $Ca(OH)_2$ comes only from the hydration of cement and react with the active SiO_2 and Al_2O_3; it will generate certain amount of fibrous AFt and gel C–S–H (Wang et al. 2014; Sheng et al. 2007; Fortes et al. 2016). The crystals cross with each other, then form a chain structure which is mainly responsible for hardening of whole system (Radwan et al. 2013). Also, the process flow sheet and production practice of the admixture of cement are feasible. In conclusion, the activity of ODPR is the same as other pozzolanic materials.

(2) Major index of admixture of cement quality control

A primary observation was that during the tests, greater water was demanded when ODPRs were equal amount instead of 30% cement. So, the cement performance of ODPR becomes important during the process of resource utilization.

Admixture of pavement concrete

The process flow sheet and production practice of pavement concrete are given in Figs. 14 and 15. It can be concluded that

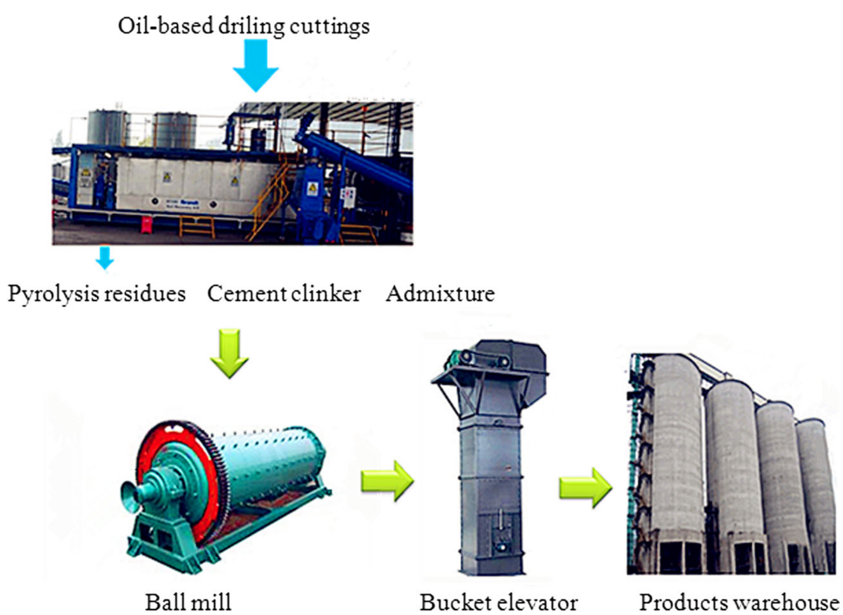

Fig. 13 Production practice of the admixture of cement

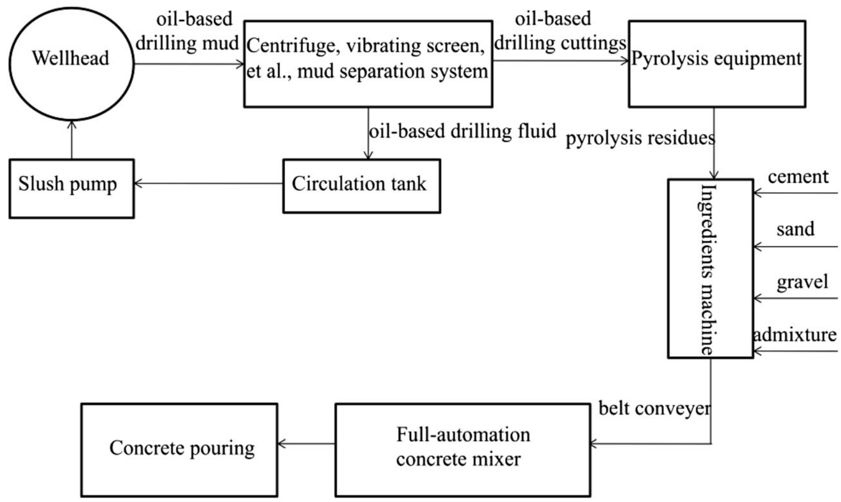

Fig. 14 Process flow sheet of the admixture of pavement concrete

while passing through an innocent treatment of pyrolysis processing, and piping to main mixer with cement, sand, gravel, and other admixtures which were mixed for about 1 min, then the water and water-reducing agent were added and mixed for approximately 1 min slowly. Finally, the cementitious materials were mixed for about 1–2 min. The ready-mixed concrete is transported by the concrete transfer car to the construction sites to pouring.

Chinese standard "Specification for Construction and Acceptance of Interlocking Block Pavement" CJJ79–98 requires that the compressive strength of the street and residential area pavement concrete should be no less than 25 MPa. When the concrete compressive strength is within C25~C55, the material used on the road surface will not influence the road structure (Shackel 1992). It is generally known that the pavement cushion concrete should use C10~C15 range and pavement concrete should use C20~C25 range.

The ODPR is a reactive pozzolanic material that improves the microstructure of the interfacial transition zone (ITZ) between the cement paste and the aggregate in high-performance concrete. The mechanism is based on two reactions: generation of calcium silicate hydrate (C–S–H) which by the reaction of active SiO_2 with CaO and generation of ettringite (AFt) which by the reaction of active Al_2O_3 with gypsum and CaO. So, the compressive strength of concrete can be increased. Besides, it greatly decreases the average pore radius of the concrete. On the other hand, choosing reasonable formula also can improve the gradation of whole system. The compressive strength not only increases but also becomes much denser in the system and improves the durability of

Fig. 15 Production practice of the admixture of pavement concrete

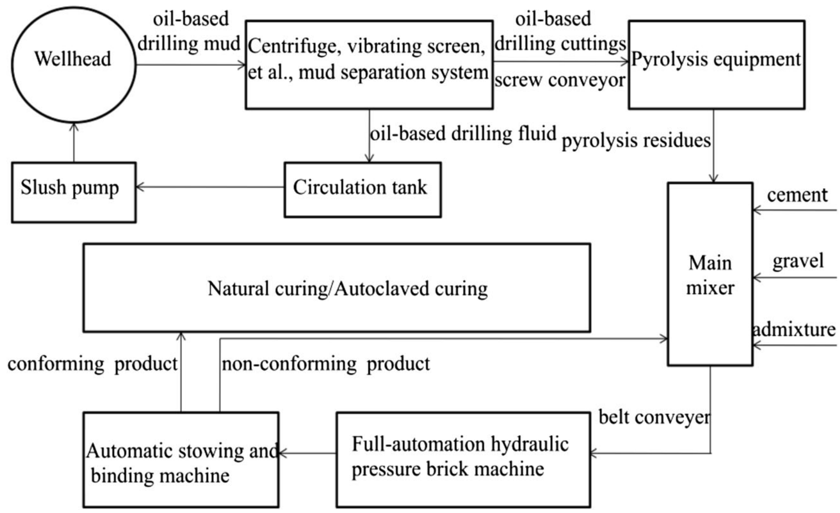

Fig. 16 Process flow sheet of the admixture of non-fired bricks

concrete (Irha et al. 2015; Smadi and Haddad 2003). Also, the process flow sheet and production practice of the admixture of concrete are feasible. So, the ODPR can be used as the admixture of pavement concrete.

Admixture of non-fired bricks

(1) Process flow sheet

The process flow sheet and production practice of non-fired bricks are given in Figs. 16 and 17. It is known that while passing through an innocent treatment of pyrolysis processing, and piping to main mixer with cement, sand, water, and other admixtures which were mixed for about 3–5 min, then through the full-automation hydraulic pressure brick machine compression molding, the non-fired bricks are produced.

(2) Theoretical basis of autoclaved bricks

Generally speaking, steam pressure curing is a versatile method of utilizing waste materials such as ODPR for the manufacture of autoclaved bricks. The hydration mechanism of products by steam pressure curing technology is a comprehensive hydrothermal synthesis reaction with the main composition of siliceous material and calcium material which can form SiO_2–CaO–Al_2O_3–H_2O system (Wang et al. 2014). In the process of autoclave curing, polymerization degree has

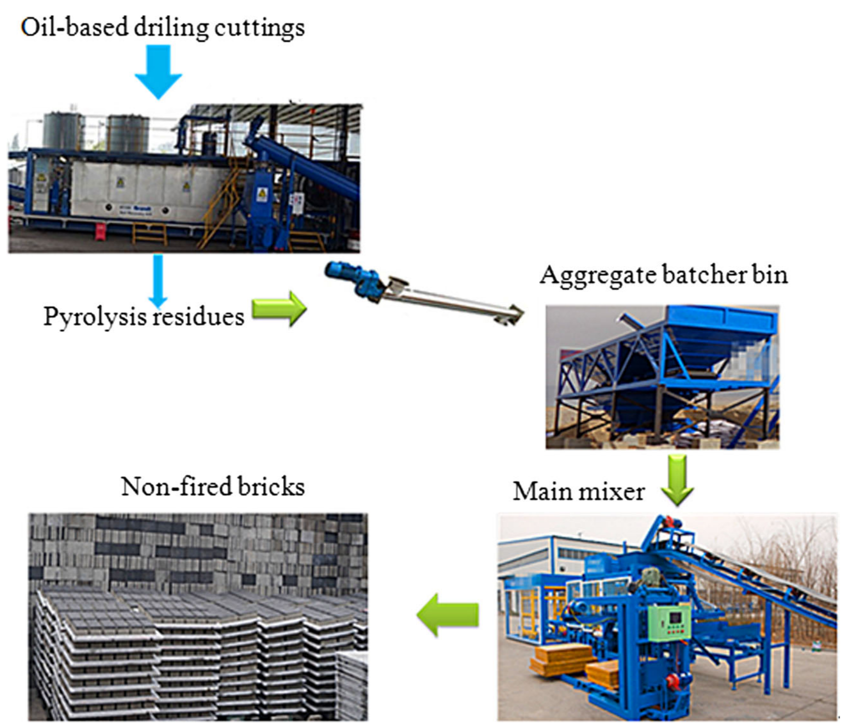

Fig. 17 Production practice of the admixture of non-fired bricks

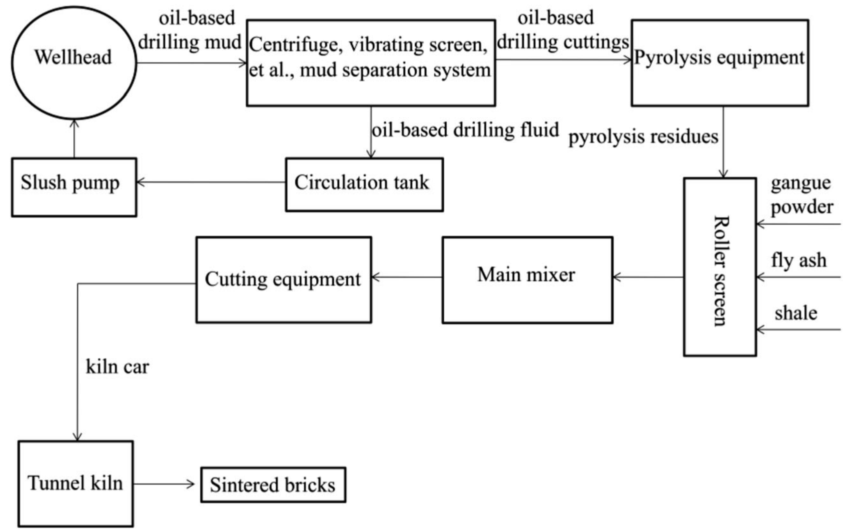

Fig. 18 Process flow sheet of the admixture of sintered bricks

been becoming more and more rigorous of the macromolecular structure of silicon-oxygen tetrahedron. Better crystallinity and hardness of calcium silicate hydrate (C–S–H) was generated, then transformed into tobermorite to follow the autoclaved curing. The essential reasons are that the dissolving properties of ODPR are in accordance with crystallinity of ettringite and C–S–H which increased in microstructure.

According to Figs. 7 and 8, it indicates that the ODPR is a calcareous and aluminum material and specific surface area is greater than the quartz sand, because absorbed water in the ODPR will be released and will contribute to accelerate the hydration of the cement under the pressure-steam condition (Wang et al. 2016). Therefore, admixing of the certain ODPR can efficiently enhance the performance of mixture (Zhou et al. 2014).

(3) Theoretical basis of non-autoclaved and non-fired bricks

Although it has been determined that the factors affect strength formation, cement content plays an important role. The EDX spectrum shows that the main elements of the ODPR were Ca, Si, and Al, respectively. So, a gross judgment is made that generation of the short stick shape C–S–H gel, and the other hand, AFt by the reaction of active Al_2O_3 with gypsum and CaO. Besides, the filler effect (Tuncan et al. 2000) of ODPR should not be ignored, which can be the micro-aggregate filling of whole system, and makes internal structure become much denser (Lee et al. 2013; Martins et al. 2014; Ganjian et al. 2015).

The main equipment of the production process is automatic hydraulic press for bricks. The high-pressure extrusion forming makes internal structure become less poriferous and fewer harmful pores. So, the strength of bricks was improved. In conclusion, the process flow sheet

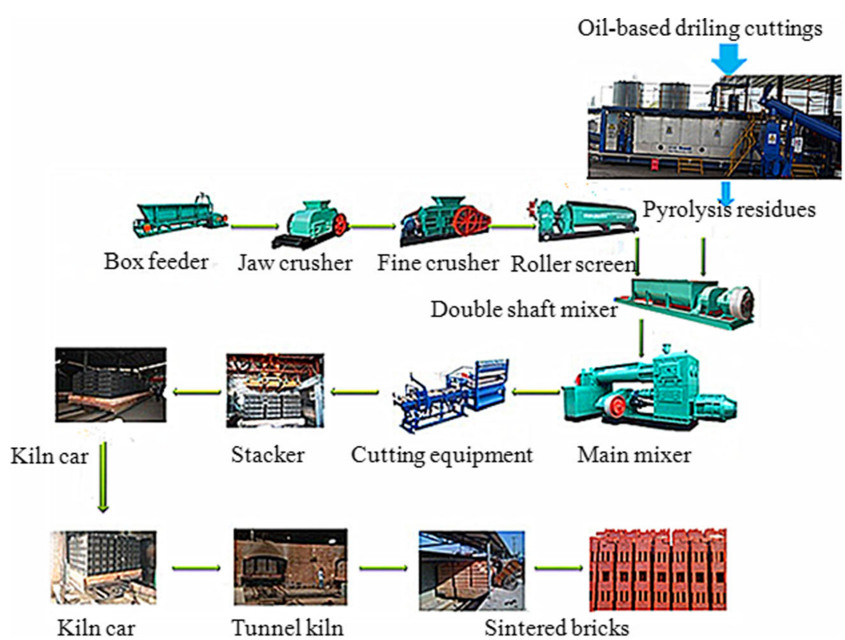

Fig. 19 Production practice of the admixture of sintered bricks

and production practice of the admixture of non-fired bricks are feasible.

Admixture of sintered bricks

The process flow sheet and production practice of sintered bricks are given in Figs. 18 and 19. It can be known that while passing through an innocent treatment of pyrolysis processing, and piping to main mixer with gangue powder, fly ash, water, and other admixtures which were mixed for about 3–5 min, then through the full-automation vacuum hydraulic pressure brick machine extrusion forming, the consequently green body are produced. Finally, the sintered bricks adopt the advanced tunnel kiln technology, and the high-temperature burn came into being.

The chemical components of ODPR and clay are shown in Table 1. It can be seen that iron, silicon, and aluminum were the main compositions for ODPR. Because manufacture sintered bricks with clay has been a mature technology, the similar compositions between ODPR and clay indicated that preparing bricks with these two materials was possible.

With 350 °C, adsorbed water evaporated and organic compounds burned. The constitution water was released from illite at 350–700 °C. Between 700 and 950 °C, the decomposition of some crystals happened such as the decomposition of calcite. When the temperature reached 950 °C, molten materials would fill these little interspaces and cause shrinkage, being the flux effect predominant over the decomposition of gaseous components (He et al. 2012). Because it maximally realized comprehensive utilization of solid waste, there were many alkaline substances (Eliche-Quesada et al. 2015; Yoo et al. 2016) in ODPR, which not only can lower the sintering temperature to save energy but also can utilize large amount of ODPR, turning solid waste into resource. In conclusion, the process flow sheet and production practice of the admixture of sintered bricks are feasible.

Conclusions

This study has demonstrated a feasibility of using oil-based drilling cutting pyrolysis residues. The results obtained in the study can be summarized as the followings:

(1) ODPR has a certain pozzolanic activity.
(2) Theoretically speaking, the ODPR resource utilization such as admixture of cement, making sintered brick, none-fired brick, etc., by the exploration and development of Fuling shale gas is feasible.
(3) Oil-based residue resource utilization not only achieves energy-saving and emission reduction but also enhances both environmental relief and economic benefits.

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