A novel ecological sand stabilization material largely utilizing coal refuse: mechanical properties, microstructure and environmental friendly performance

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Abstract

An ecological sand stabilization material largely utilizing coal refuse was developed in this research, and its mechanical properties, hydration products and environmental friendly performance were investigated. The optimal design for the sand stabilization material contained 5.75% fly ash, 5% calcium silicate slag, 10% BFS, 2.5% cement, 1.75% gypsum, 50% coal gangue and 25% aeolian sand. This blended system yields excellent mechanical properties that the compressive strength at 28 days exceeds 10 MPa. The main hydration products of the sand stabilization material are ettringite and C-S-H gel, which play an important role in promoting the densification of the paste structure and the strength development of the sand stabilization material. The leaching toxicity test results indicate that the developed sand stabilization material can consolidate heavy metal ions in wastes and is

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environmentally acceptable. This study provides an effective way for the use of coal refuse and a new method to improve the ecological environment of the desert.

Key words: Coal refuse, Sand stabilization material, Hydration products, Environmental friendly performance, Aeolian sand

1. Introduction

China is a country with coal resources as its main energy. The amount of mining consumption in China has increased year by year due to the extensive application of coal. The production and utilization of coal has produced a lot of coal refuse such as fly ash, coal gangue and flue gas desulfurization(FGD). Fly ash is one of the coal combustion products composed of the fine particles that are driven out of the boiler with the flue gases. Although fly ash is a complex industrial waste whose chemical and mineralogical composition is widely different, fly ash in China mainly contains six oxides named SiO_2, Al_2O_3, FeO, Fe_2O_3, CaO, and TiO_2. The output of fly ash in China was 569.5 million tons in 2015 and the stockpile amount of fly ash in China is over 10 billion tons[1]. Fly ash has been widely utilized in building materials, construction engineering, transport, agriculture, chemical and metallurgical industries [2-6]. Coal gangue is a complex industrial solid waste discharged from the process of coal mining and washing, accounting for approximately 10% of coal production [7]. The accumulative stockpile of coal gangue has reached 3.8 billion tons and it is estimated that over 500 million tons of this waste is impounded annually in China [8].

The storage of coal refuse has caused serious environmental problems such as soil contamination and groundwater pollution. In modern society, the general trend of using coal refuse is to find alternative ways for their utilization (i.e. as raw materials for the production of blended cements, Portland cement clinker, ceramics and road construction etc.) to eliminate cost of disposal and avoid soil contamination[9].

The area of deserted land in China is the largest among the world, especially in Inner Mongolia province and Sinkiang province. The deserted land and the potential ones cover 60 percent of the regional area in Inner Mongolia and are increasing 670,000 hectares per year. It is necessary to control the desertification because desertification has been threatening national ecological security and social economic development directly. There are large amount of coal refuse produced in Inner Mongolia. It is known that coal refuse contain amounts of inorganic ingredients, such as oxides of silicon, aluminum, calcium, which can be used as raw materials of building materials. Nevertheless, the market of building materials is limited in Inner Mongolia, and the freight is a great limit if building material products are transported to other places. Since there are large area of deserted land in Inner Mongolia, using coal refuse to produce sand stabilization material can not only control the desertification, but also reduce the environmental pollution through decreasing the accretion of solid wastes.

The methods of sand fixation in China mainly include mechanical sand barriers, vegetal sand fixation, and chemical sand fixation [10]. Mechanical sand barrier is usually set on desert to decrease wind speed and change direction of deflation

[11].Vegetal sand fixation improves the ecological environment of desert by planting vegetations, which is the most common technology in desert control because of its advantages of persistence, high efficiency and stability [12]. Chemical sand fixation uses chemical methods and chemical materials to construct sand fixation layer in desert, which can prevent eolation and maintain moisture of sand [11]. Methods mentioned above can’t meet the requirements of low cost, high efficiency, simple construction and good biocompatibility at the same time. Therefore, it is necessary to use comprehensive sand fixation.

Chemical sand-fixing technology has been widely investigated during past hundred years, and over 100 kinds of chemical materials were developed[13].An assumption that using fly ash as the main material to synthetize sand stabilization material was put forward in 1990s[14]. The solidfying sand materials with the fly ash and bentonite by polyacrylate polymerizing has been investigated and the compressive strength, water absorbing capacity and water holding capacity of the solidfying sand materials were the best when fly ash was 60%, bentonite was 32% and water glass was 16%. The production cost of the solidifying sand material is 3.6 yuan per kilogram, indicating the good feasibility and economic efficiency[15].

Cement based chemical sand stabilization materials were investigated widely in the world. Cementitious sand stabilization material is sprayed on the sand surface and a consolidated layer is formed after hardening. When sand is fixed, vegetation is planted to reinvent desert. A new design principle was brought up that some kinds of polymers can modify cement mortar to prepare polymer modified cementitious sand-fixing material with high water/cement ratio and high cement/sand ratio. The new polymer cement-based sand stabilization material was compounded with the optimal proportion that cement, sand, asphalt emulsion and sodium polyacrylate superabsorbent was 1:6:0.05:0.05. The durability results showed that this sand-fixing material had good aging resistance, water resistance, freeze-thaw stability and weathering capabilities. A technology was put forward that using cement-soil based material to synthetize cement-based plant-growing fixing-sand materials. This new sand-fixing material has a good ability to control and release water and fertilizer in a long time, which is beneficial to the growth of plant in dry arid-semi-arid regions lack of water and fertilizer. To solve the problem of high alkalinity of cement-based material, low-alkali plan was made. Not only is pH of soil neutral, but also NH^{4+} in soil changes to NO^{3-} after ammoniation [16]. Apolymercement-based sand-fixing material was investigated with cement mortar as its substrate and modified by adding a cationic emulsified asphalt and sodium polyacrylate resin. The environmental friendly and low cost product has good characteristics of water retention, water absorption, durability, resistance to freeze-thaw stability, resistance to wind erosion and weather resistance [17]. Cement mortar has a strong function of water absorption and retention if mixed with polyurethane and sodium polyacrylate of 5% of cementeach, which is beneficial to providing sufficient water for plant growth [18].

An ecological sand stabilization material largely utilizing coal refuse was developed in this research, and its mechanical properties, hydration products and environmental friendly performance were investigated. This sand stabilization material controls the desertification through preventing sand from moving by the consolidation layer and planting vegetation such as energy grass inside the consolidation material. After soil improvement, the sand stabilization material can decompose gradually, which can reduce the human impact on sand environment.

2. Experimental

2.1 Materials

Calcium silicate slag, fly ash, coal gangue and FGD were obtained from Inner Mongolia Datang International Renewable Resources Development Co. Ltd. Granulated blast furnace slag (BFS) was offered by Tangshan steel plant and cement was supplied by Inner Mongolia Mengxi cement plant. The chemical composition (analyzed by X-ray fluorescence spectrometer) and specific surface area (tested by full automation instrument testing specific surface) of raw materials are presented in Table 1. The mineralogical phases (determined by X-Ray diffractometer) of raw materials are showed in Fig. 1.

Table 1 Chemical composition and specific surface area of raw materials

Oxides(%)	Calcium silicate slag	Fly ash	BFS	Clinker	Gypsum
CaO	55.35	3.61	36.32	51.54	46.21
SiO_2	31.08	48.08	36.96	26.99	3.50
Al_2O_3	5.97	40.50	10.20	10.40	1.69
Fe_2O_3	3.64	2.96	0.98	4.24	1.19
MgO	3.61	0.24	7.52	2.50	1.42
TiO_2	2.36	2.37	4.28	1.05	0.19
Na_2O	1.81	-	0.29	0.08	0.20

K_2O	0.36	0.36	0.55	1.02	0.31
SrO	0.23	0.29	0.14	0.11	0.08
SO_3	0.23	0.30	1.57	1.85	43.15
ZrO_2	0.20	0.20	-	-	-
MnO	-	-	0.95	0.09	-
P_2O_5	-	0.18	-	-	-
F	-	-	-	-	1.99
specific surface area(m^2/kg)	797.81	575.73	483.81	468.55	668.72

Fig. 1 Mineralogical phases of calcium silicate slag, fly ash, BFS and cement

2.2 Experimental procedure

Fly ash, calcium silicate slag, BFS were dried at 105 °C in an oven while gypsum was dried at 50 °C. BFS was ground in a laboratory ball mill for 90 minutes, while other raw materials were ground for 20 to 30 minutes. The coal gangue was sieved to two kinds of diameter (1-2mm and 0.5-1mm). The diameter of aeolian sand (obtained from desert in Inner Mongolia) is below 0.5mm. Fly ash, BFS, calcium silicate slag, cement and gypsum were mixed in appropriate proportions. Mortar specimens in size of 40 mm × 40 mm × 160 mm were prepared with a certain water/cement (W/C) ratio and cement/sand (C/S) ratio of 1:3. They were cured in a moist cabinet at 95% humidity and 20◦C for 24 h, and then demoulded and transferred to an isothermal curing cabinet at the humidity and temperature mentioned above.

The mortar specimens of sand stabilization material were prepared with a water/cement ratio of 0.35 and molded in size of 20 mm × 20 mm × 20 mm. Theywere cured in the same conditions as mentioned above. The hydration of the pulverized and sieved specimens was terminated by alcohol drenching at the desired testing time, and then dried at 60◦C in a vacuum oven for further characterization [19].

2.3 Testing conditions

1. The chemical analysis was performed with the X-ray fluorescence (XRF-1800) analyzer.
2. XRD analysis was carried out using M21X X-ray diffractometer with CuKα radiation, a voltage of 40 KV, a current of 200mA and 2θ scanning ranging between 5° and 90°.
3. The specific surface area was tested using FBT-9 full automation instrument according to Chinese Standard GB/T8074-2008[20].
4. Mechanical tests were performed according to Chinese Standard GB/T 17671-1999 [21].
5. The leaching toxicity test was carried out according to the solid waste-extraction procedure for leaching toxicity -horizontal vibration method

(Chinese Standard HJ 557-2010) [22].

6. The concentrations of heavy metals were analyzed using OPTIMA 7000DV ICP optical emission spectrometer.
7. TG-DSC analysis was performed on Netzsch STA 449C Thermal analyzer. Dry N_2 gas was used as a stripping gas. The heating rate was 10°C/min, and the samples were heated from 20 to 1000°C.
8. SEM observation was carried out on JSM-6701F scanning electron microscope equipped with Be4-U92 energy spectrum detector for composition analysis of the hydration products.
9. FTIR analysis was performed using a Spectrum GX PerkinElmer Fourier transform infrared spectrometer.

3. Results and discussion

3.1 Mechanical properties of sand stabilization material

3.1.1 Influence of water/cement ratio on sand-fixing

Fig. 2 shows the effect of water/cement ratio on the compressive strength of the sand stabilization material with different water/cement ratio (0.65, 0.7, 0.75, 0.8 and 0.85). The compressive strength of investigated sand stabilization material decreases with the decrease of water cement ratio when water cement ratio is above 0.7 while it increases with water cement ratio increase when water cement ratio is below 0.7. However, the strength values of the sand stabilization material with 0.85 water cement ratio at the age of 7 and 28 days are higher than that with 0.65 W/C ratios. It is possibly because coal gangue and other raw materials have high water absorption. Cementitious material has low fluidity with low water cement ratio, making bonding force between cementitious material and aggregate small. Raw materials of sand stabilization material absorb a lot of water because of high water absorption when water cement ratio is below 0.7, which makes actual water cement ratio decrease. The water absorbing quantity of cementitious materials and aggregate is almost saturable when water cement ratio is 0.7. Compactness of sand stabilization material decreases with more water, which makes compressive strength decrease.

Fig.2 Influence of water/cement ratio on sand-fixing

3.1.2 Influence of cement content on sand-fixing

As showed in Fig.3, it is obviously that compressive strength increases with the increase of cement content. The values of compressive strength at the age of 3 days are more than 3 MPa except that of1.25% cement content. The value differences of compressive strength at different agesamong2.5%,3.75% and 5%cement content and are not more than 1MPa, which is considered as the small difference correspondingly. Especially at the age of 28 days, the value of compressive strength with 3.75% cement content is equal to that with 5%. The result shows that in this system, BFS is also a main factor influencing sand-fixing effect. For saving energy and source, we are supposed to use cement as little as possible.

Fig. 3Influence of cement content on sand-fixing effect

The compressive strength is an important parameter used to evaluate the sand-fixing property. The designed proportions are listed in Table 2. At the age of 3 days, the flexural strength and compressive strength of sand stabilization material have reached values of 1.55 and 6.4 MPa respectively, while the compressive strength value of sand stabilization material have reached 12.4 MPa at the age of 28 days. Although the mixing amount of cement was very low (2.5%), and the mixing amount of coal refuse was high (62.5%),all the strength values of the investigated sand stabilization material at the age of 3, 7 and 28 days were significantly higher than that of international requirements (compressive strength of the sand fixation layer is more than 3MPa).

It can be inferred from the mechanical properties that it is feasible to use coal refuse to produce sand stabilization material. This produced material can not only consume large quantities of coal-based solid waste, but also save a lot of natural resources. It decreases the producing cost of sand stabilization, which possesses important environmental and economic significances.

Table 2 Designed proportion of sand stabilization material (%)

Fly ash	calcium slag	silicate	BFS	cement	Gypsum	Coal gangue	Aeolian sand	water/cement ratio
5.75	5		10	2.5	1.75	50	25	0.75

3.2 Micro-structural analysis of the sand stabilization material pastes

3.2.1 XRD analysis

To analyze the hydration products of sand stabilization material, the pastes were tested by XRD. The XRD patterns of the sand stabilization material pastes hydrated at different ages are presented in Fig. 4.It can be seen that the diffraction peaks of ettringite ($Ca_6Al_2(SO_4)_3(OH)_{12}\cdot 26H_2O$), $Ca(OH)_2$ and C–S–H phases appeared in all samples obtained during hydration. The diffraction peaks of C-S-H is weaker than ettringite because the cement content is very low. In early hydration process of the sand stabilization material pastes, C_3S, C_2S in cement and silicon-rich phase of BFS vitreous disintegrate rapidly, and free state of Si^{4+} is formed, which makes concentration of $[H_3SiO_4]^-$ in liquid increase gradually.$[H_3SiO_4]^-$ combines with Ca^{2+}

to generate C-S-H at a certain concentration. BFS is a material with potential cementitious, which can be activated in alkaline environment. BFS has low polymerization degree of $[SiO_4]$ and $[AlO_4]$, which can quickly participate in the reaction in the early hydration. The generated product of BFS is beneficial for the development of early strength. Moreover, fly ash particles play a role in reducing water and making the construct more compact.

It is likely that in early hydration process of the sand stabilization material pastes, the raw materials such as fly ash, calcium silicate slag, BFS, cement, and gypsum were beneficial to form $Ca(OH)_2$and ettringite. Besides, it is noted that the peaks of $Ca(OH)_2$diminished whereas those of C–S–H gel increased, especially at the age of 28 days. Thus, it can be concluded that the dominant hydration products of C–S–H gel and ettringite are principally responsible for the strength development of sand stabilization material in early hydration process. Part of the C_2S hasn't hydrated completely in the early hydration process, especially at 3 the age of days, providing continuous dynamic for the growth of strength.

Fig. 4 XRD patterns of hydrated sand stabilization material pastes

3.2.2 TG-DSC analysis

The TG-DSC results for the hardened sand stabilization material paste are shown in Fig. 5. The first endothermic peak at about 70–200 °C is mainly due to the dehydration of C–S–H gel and the decomposition of ettringite ($Ca_6Al_2(SO_4)_3(OH)_{12}\cdot 26H_2O$). The mass losses at different ages of hydrated sand-fixing material pastes at about 70-200°C are 3.82%, 3.88% and 4.51% respectively, which can reflect the content of hydration products at different curing ages indirectly. In addition, growing trend of the content of these two main hydration products ensured the development of compressive strength of sand-fixing material. The second endothermic peak located at about 400–470°C is attributed to the decomposition of $Ca(OH)_2$. The mass losses of hydrated pastes at 3d, 7d and 28d at about 400-475°C are 0.43%, 0.46% and 0.43% respectively, showing that the content of $Ca(OH)_2$ increased at first and then reduced. This is because in the hydration process, first hydration reaction generated $Ca(OH)_2$, while the second hydration reaction consumed $Ca(OH)_2$. The content of $Ca(OH)_2$ in sand stabilization material pastes would decrease when the amount of $Ca(OH)_2$ consumed was more than that of $Ca(OH)_2$ generated,. The third endothermic peak can be observed at about 650–750°C, which is associated to the decomposition of $CaCO_3$.

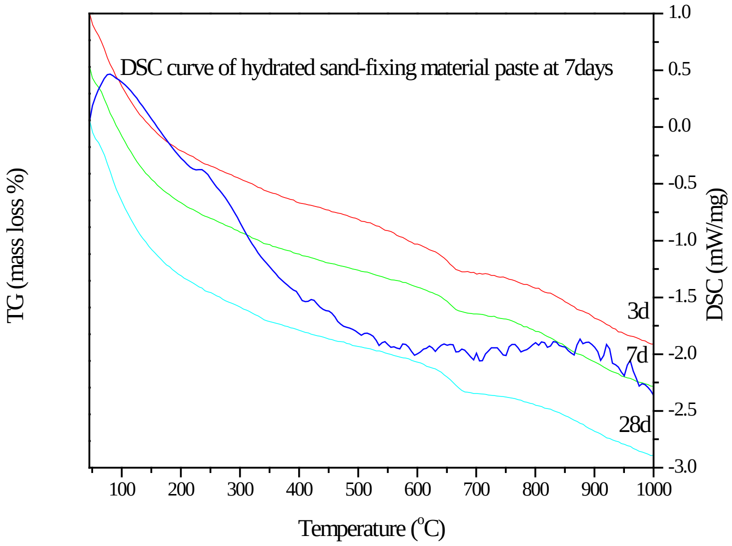


Fig. 5 TG-DSC curves for hydrated sand-fixing material pastes

3.2.3FTIR analysis

The FTIR spectra of the hardened sand stabilization material pastes hydrated at different ages (3d, 7d, 28d) are shown in Fig. 6. The big band around 3452cm^{-1} is related to Si-OH stretching vibration, while the band around 1640cm^{-1} is related to H_2O bending vibration. Free water participates into the hydration and gradually transforms into crystal water with the increase of the hydration period. The absorption peak around 1482cm^{-1} is the characteristic absorption band of CO_3^{2-}. The band around 1425cm^{-1} is one of the absorption peak of C-S-H gel. It is noted that the absorption peak around 975cm^{-1} indicates asymmetric stretching vibration of Si-O-Si in C-S-H gel. The absorption peak around 975cm^{-1}becomes stronger with the increase of the hydration period, indicating that the amount of C-S-H gel in pastes increase correspondingly. The weak absorption peak appears around 876cm^{-1} is due to Si-OH bending vibration. The absorption peak around 876cm^{-1} representing C-S-H gel becomes clear while the big band around 975cm^{-1} sharpens, suggesting that the amorphous C–S–H gel tends to crystallize along with the hydration going on.

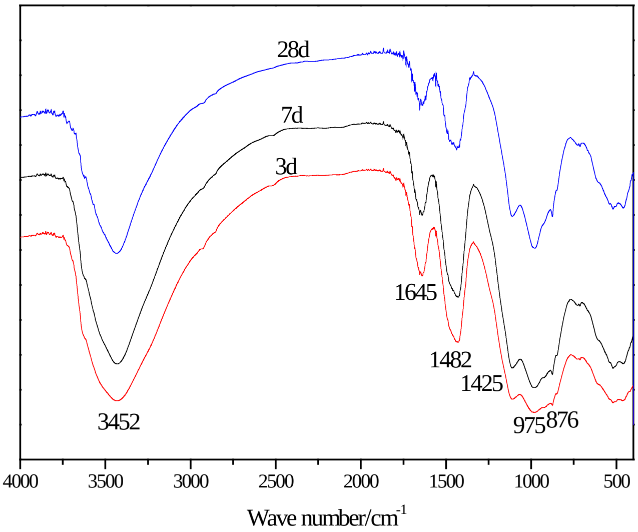


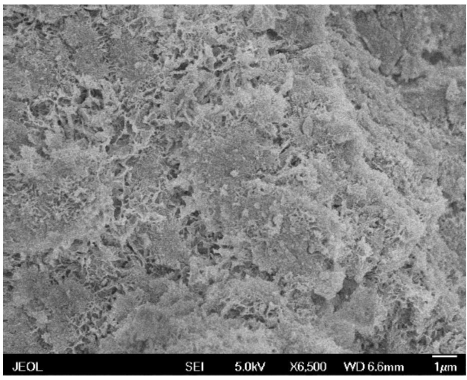
Fig. 6 FTIR spectra of sand stabilization pastes hydrated for 3, 7 and 28 days

3.2.4 SEM analysis

The microstructure of the sand stabilization material pastes hydrated at 3, 7, and 28 days analyzed by SEM is shown in Fig. 7. It is noted that the hydration products such as needle-shaped or virgulate ettringite phase and C-S-H gel can be found in SEM observation, which provides remarkably good correspondence with the XRD analysis. It is evident that slender needle-shaped ettringite phase is formed in the pastes at the age of 3 days, which grows along the pore wall. The hydration products in 3 days have low compactness and large amount of ventage. This can be attributed to the low intensity in early period of hydration process. The compactness of hydration products at the age of 7 days is bigger than that of 3 days. The ventage of pastes is filled by ettringite, which is beneficial to densification of pastes. Needle-shaped ettringite becomes thicker gradually at the age of 28 days with increasing of the hydration period. The ettringite crystals are dispersed through the netty C-S-H gel, resulting in a dense structure and low porosity, which makes strength of pastes higher.

1. (b)

(c) (d)



(e) (f)

Fig. 7 Microstructure of hydrated sand stabilization pastes at different curing ages: (a) (b) 3 days, (c) (d) 7 days, (e) (f) 28days

3.3 Environmental friendly performance of the sand stabilization material

As the investigated sand stabilization material composed of fly ash, calcium silicate slag, BFS and coal-gangue would be used to control desertification, it is necessary to evaluate its environmental impact. Here the leaching toxicity of raw materials (fly ash, calcium silicate slag, BFS and coal-gangue) and sand stabilization material sample at 3d, 7d, and 28d have been tested to investigate the environmental friendly performance of the produced sand stabilization material. The results are shown in Table 3.

The main environmental concern about utilization of sand stabilization material composed of coal refuse is the possibility of certain hazardous constitutes leaching into the groundwater with concentrations determined to be potentially harmful to human health. Standards for drinking water quality in Chinese Standard GB 5749-2006 used to evaluate the leaching toxicity results are listed in Table 3 [23-25]. It can be seen from Table 3 that As ions and Cr ions leaching concentrations of fly ash exceed the limits specified in standards for drinking water quality, while that of other raw materials are below the standards. It is noteworthy that none of the metal leaching concentrations of sand stabilization material at 3d, 7d and 28d exceeds the limits specified in standards, indicating that the sand stabilization material handle the function of adsorbing and curing heavy-metal-ions in wastes. One of the main hydration products of the sand stabilization material is ettringite, which can accommodate foreign ions in field cylinder by chemical replacement. Another main hydration products is C-S-H gel, having high energy of ion exchange, which is beneficial to foreign ions curing. Ettringite has good ability to cure Zn^{2+},this is the reason why the leaching of raw materials has high concentrations of Zn^{2+}while the leaching of sand stabilization material at 3d, 7d and 28d has low concentrations that can't be detected. Ettringite has been found that it can capture heavy metal elements such as Cd^{2+} and Cu^{2+} into its lattice. It has been proved that Cd^{2+} can be dissolved in the crystal lattice of ettringite [26].As ions has been found that they are easier to be dissolved in the crystal lattice of ettringite than Cr ions and Se ions [27]. C-S-H gel has a strong ability to cure Cr^{3+}, even though under carbonization condition, C-S-H gel still handles curing ability for Cd^{2+}, Cr^{3+}, Pb^{2+} and Zn^{2+}[28,29]. These research results indicate that the developed sand stabilization material composed of coal-base solid waste is environmentally acceptable.

Table 3 Leaching toxicity test results of raw materials and sand stabilization material

Element	GB 5749-2006 limits (ppm)	Calcium silicate slag (ppm)	Fly ash (ppm)	BFS (ppm)	Coal-gangue (ppm)	Sand stabilization material at 3d (ppm)	Sand stabilization material at 7d (ppm)	Sand stabilization material at 28d (ppm)
Barium	0.700	0.166	0.068	0.532	0.369	0.036	0.093	0.077
Arsenic	0.010	0.021	0.347	-	-	-	-	-

Cadmium	0.005	-	-	-	-	-	-	-
Chromium	0.050	-	0.121	-	-	-	-	-
Copper	1.000	0.174	0.179	-	-	-	-	-
Nickel	0.020	-	-	-	-	-	-	-
Lead	0.010	-	-	-	-	-	-	-
Antimony	0.005	-	-	-	-	-	-	-
Zinc	1.000	-	0.952	0.025	0.076	-	-	-
Mercury	0.001	-	-	-	-	-	-	-

4. Conclusions

This study used coal refuse (fly ash, calcium silicate slag and coal gangue), BFS, Portland cement and gypsum as raw materials to prepare an ecological sand stabilization material. The mechanical properties, hydration products and environmental friendly performance of this sand stabilization material were investigated. From the studies carried out, the following conclusions can be drawn:

1. An optimal design for the sand stabilization material composed of coal refuse was determined by the hardened paste containing 5.75% fly ash, 5% calcium silicate slag, 10% BFS, 2.5% cement, 1.75% gypsum, 50% coal gangue and 25% sand. The compressive strength of this developed sand stabilization material at 3, 7 and 28 days can reach 6.4, 8.1 and 12.4 MPa, which was significantly higher than that of international requirements (compressive strength of the sand fixation layer is more than 3MPa). The produced sand stabilization material can provide a stable surface on the sand and stabilize

shifting sands in situ marvelously.

2. The main hydration products of the sand stabilization material prepared by coal refuse are ettringite and C-S-H gel. The content of ettingite and C-S-H gel increased with increase of the hydration time. Moreover, the amount of $Ca(OH)_2$ increased at first and decreased because of the consumption of second hydration reaction. As the predominant hydration products, ettringite and C-S-H gel play a positive role in promoting the densification of the paste structure, and also play an important role in the strength development of the sand stabilization material.
3. The leaching results show that As ions and Cr ions leaching concentrations of fly ash exceed the limits specified in standards for drinking water quality, while none of the metal leaching concentrations of sand stabilization material at 3, 7 and 28 days exceeds the limits specified in standards. The results indicate that the produced sand stabilization material handle the function of adsorbing and curing heavy-metal-ions in wastes and is environmentally acceptable.
4. This study provides an effective way for the use of coal refuse to produce sand stabilization materials, which points out a promising direction for the proper utilization of fly ash, calcium silicate slag and coal gangue in large quantities. Possessing important environmental and social significances, the developed sand stabilization material composed of coal refuse can not only alleviate environment pollution of solid wastes, but also improve the ecological

environment of the desert.

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