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A Study of Microstructure of Steel Slags Used for Pollutants Adsorption and Removal in Waste Water

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Abstract: "Waste treatments waste" – the reuse of waste could bring a number of environmental and economical benefits such as reducing waste and treatment cost in many industrial sectors. This study made use of steel slag as treatment material for polluted waste. High performance of fill teration may come with the parameters of volumetric mass density of steel slag is 3.1 ~ 3.6 g/cm^3, standard screen is 0.175 mm, weight density powder is about 0.174g/cm^3, surface is about 0.32 m^2/g, average size is 5.3 nm. Chemical components of steel slag analyzed by XRF (X-ray fluorescence spectrometer), which shows CaO is the main component of steel slag, 48.2% and 42.5% were steel slag and steel slag powder, respectively. The following major components are Fe_2O_3 and MgO content. Metals content determined by TCLP (Toxicity Characteristic Leaching Procedure) that shows Pb, Hg, Cd, Cr, As, Ni, Cu, Mn, Zn content which is less than National Standard II (GB 3838-2002); structure of steel slag analyzed by SEM (Scanning Electron Spectroscopy) and XRD (X-Ray diffraction) which shows include magnetite, tri-calcium silicate (Ca_3S), di-calcium silicate (Ca_2S), and iron in tin (FeO). Arsenic absorption and removal by steel slag are simple and possibly experimental and that can apply in large-scale so any effective from that point efficient.

Keywords: Waste treatment waste, steel slag, toxic, wastewater, absorption.

1. Introduction

Nowadays, there're many methods that adsorp and remove arsenic in wastewater such as: physics, chemistry and biology. A number of methods are accumulation, absorption, ion exchange, separation membrane and biology, etc. [1-3]. Absorption method is perfect, simple and the efficient and confident. When treatment arsenic contamination, choosing materials which adsorb arsenic not only check in the ability and effect arsenic adsorption and removal but also cost of material and scale of application. Especially, the method can be used in rural and emergency areas. For this reason, we choose steel slag as arsenic absorption and removal material in this study.

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Steel slag – metallurgical industrial waste, it is one of pollutants. In 2012, steel slag's volume estimation is about 18.10^9 tons, thus, study of arsenic absorption and removal by steel slag is positive heading to project environmental "waste treatments waste" according. Moreover, the price of steel slag is cheap; collecting is easy; therefore, it is a great value. Especially, this act is appropriate for rural areas and mountainous region where water resources were contaminated high arsencosis. Recently, experiment on absorption materials for treatment wastewater by steel slag has provided a good result. Because, steel slag with an indispensable characteristics such as mineral component, porosity and surface, it is considered as a potential material that absorps pollutants [4-9]. Molten steel slag is mixed different metal oxides; thus, it is resistant to alkali and heat stability [10]. Furthermore, steel slag is resulted from tin ore and metallurgy technology, thus, it is different in mineral component and structure, therefore, it should be analyzed, particularly before experiment, to have enough understanding its parameters bring into practice [11-14].

In this study, we present initial results of steel slag's structure that absorb and remove pollutants in waste water. Density of volume mass is 3.1~3.6 g/cm^3, sieve standard is 0.175 mm, density of volume weight is about 1.74 g/cm^3 in comparison which surface is 0.32 m^2/g, a medium-sized is 5.3 nm. With this parameter, steel slag is potential material for good performance in pollution adsorption and removal.

2. Materials and methods

Steel slag size is 2~5 mm, it was obtained from Baogang Development Joint-Stock Company. Chemical component is analyzed by XRFS (X-ray Flourescence Spectrometer)[15]. Metals content determination in steel slag as following process: grinded steel slag, sieved through 0.60mm, dissolved by HNO_3-HF-$HClO_4$ then analyzed ICP-MS (inductively coupled plasma mass spectrometry) by plasma spectrum batch [16-18]. At the same time, metals were filtered by TCPL process: take 3 steel slag samples (0.15mm, 0.25mm, 0.60mm), put 10g in polyethlence pot, add 200mL TCLP (acetic acid pH=2.88), shake within 18h in room temperature, filter through 0.45μm and add HNO_3 solution up to pH < 2. Metal content them was determined by ICP-MS [19, 20].

2.1. Materials micro-structure analyzed by Scanning Electron Spectroscopy

Steel slag was made into thin sheets by the preparation process of the flakes [21]: The slag was cut into slightly larger pieces with a rectangular slag is 20mm × 15mm × 10mm by a slicer; then grind it with 150 grit to 20mm × 15mm × 10mm and wash it with clean water; grind it with glue first, then grind with 185μm and 370μm corundum, and finally use alumina slurry to ground on a glass plate, to remove all scratches, and washed with water; the sheets are then polished with a polishing machine. SEM testing used QUANTA 200 of FEI company in USA, resolution 4.5nm (cathode voltage wire), enlarge 15X~250000X, cathode strafed electron, voltage increasing on a scale of0-30kV, lens system with threes electronic lens [16].

Based on the above research and combination with the problems of arsenic in the original steel slag, the original steel slag was modified through high-temperature activation, strong alkali treatment, and organic and inorganic loads, etc., on the basis of multiple batch tests, the actual acquisition was used. The high-arsenic water was subjected to a dynamic test on the percolation column to compare the absorption process and absorbed performance of arsenic. The microscopic sites and structures of the arsenic absorbed by the modified steel slag were preliminary identified by scanning electron microscopy (SEM) [22-24]. Combination with the calculation of the maximum arsenic absorption amount, the steel slag modification conditions was selected suitable for high-arsenic water absorption.

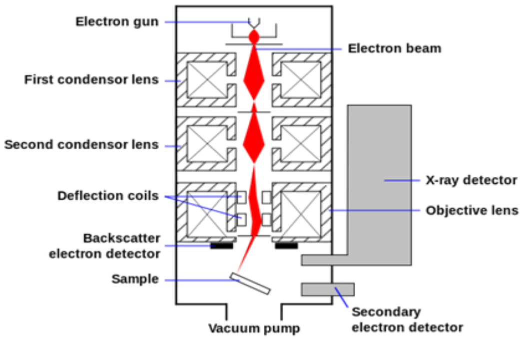

Figure 1. Diagrammatic SEM [25].

2.2. Micro-structure of material powder analyzed by X-Ray diffraction

Sample grinded by follow processing rule: totals damage rate is not more than 5%, shrink is less than 3%, sample analyzed by following equation [26-29]:

$Q=kd^2$

In this equation: Q is decrease content achieve (kg)

k is decrease coefficient, in that steel slag is 0.2

d is the largest diameter (mm)

XRD is Y500, lend voltage is 40 kV, electric current is 20 mA, scan speed 0.06°/second, scan range $2^{\theta}=10^{o}\sim70^{o}$. Samples was inspected anhydrous ethanol in wet porcelain tray until the surface is 400 m^2/kg, dry in 60°C, then check steel slag powder.

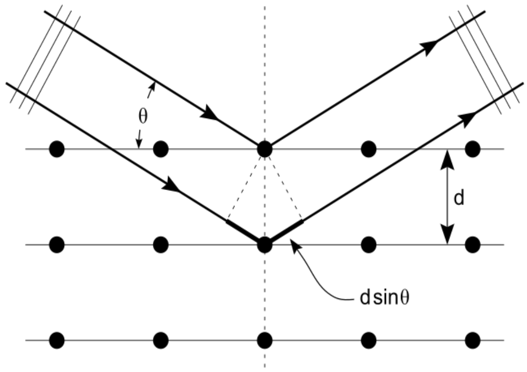

Figure 2. X-Ray diffraction(XRD) based on Bragg's Law [30].

3. Results

3.1. Major chemical structure of steel slag

Steel slag with a lot of calcium, silica, the main contents are CaO, SiO_2, Fe_2O_3, $A1_2O_3$, MgO, P_2O_5, MnO, CaS, FeS, etc. The main component content is shown in table1.

Table 1. Main chemical component of steel slag in experiment (w/%)

Steel slag source	CaO	SiO_2	Fe_2O_3	$A1_2O_3$	MgO	P_2O_5	MnO	FeO	f-CaO
Grains steel slag	48.2	15.1	22.9	3.24	14.12	0.78	1.25	11.3	4.12
Powder of steel slag	42.3	9.13	8.96	6.72	4.12	6.14	0.69	4.12	8.12

Table 1 shows that the main component of steel slag is CaO with nearly of 50%, the following are Fe_2O_3 and MgO, they are important mineral oxides which exist in steel slag for absorption and removal of pollutants. Table 1 shows also the difference in component of steel slag grains and powder. Previous researches has proved that the same smelting furnace but different at melting period, the chemistry components were much different. This is the reason which must analyze steel slag structure to before that was being used in practice.

3.2. Analyzing micro-structure of steel slag

Figure 3 shows surface steel slag by SEM as: 1) Black precinct has CaSi and mineral beads with the main component of siliciferous (SiO_2), calcium oxide (CaO) and a few silicate tri-calcium (Ca_3Si). There is a few small beads with the main component of un-crystalline and iron oxide (Fe_2O_3); 2) Gray black precinct has mixture of iron-calcium with the main component ofFe_2O_3 and CaO, and the main mineral of iron acid salt ($2CaO.Fe_2O_3$),a soluble matter; 3) Gray white precinct has hydrated lime with iron salt (MgO.2FeO),a soluble matter, and black mineral is magnesium oxide; 4) Silver white beads is iron metal with high iron content. The above characteristics of structure steel slag are suitable for absorption of pollutants in waste water.

Figure 3 shows also abrasive level on surface of steel slag, in here there are many different grasp which bring to increase surface area and absorption ability of pollutants in water.

Similarly, steel slag analyzed step by step by XRD that shows its main contents include magnetite, tri-calcium silicate (Ca_3S), di-calcium silicate (Ca_2S), iron in tin (FeO) (figure 4). It contains 19% of tin with outside of glass.

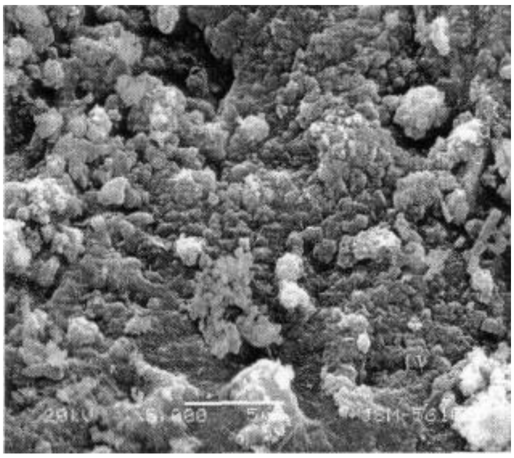

Figure 3. Steel slag on SEM.

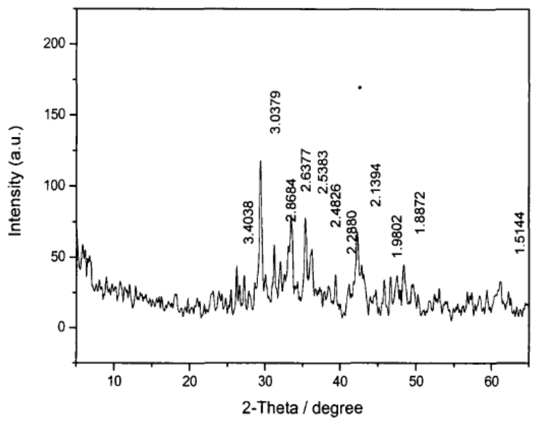

Figure 4. Steel slag analyzed by XRD.

3.3. Metals content in steel slag and in separate filter

In technical of point of view, steel slag has the advantage of physic, chemistry, and mechanical features those make steel slag absorb pollutants and stable chemical properties. However, in chemical point of view, steel slag has a small content metals that are Pb, Hg, Cd, Cr, As, Ni, Cu, Mn, Zn etc., with variety in content. The treatment pollution may create heavy metals by steel slag in the filtered waters that causes secondary pollution. Therefore, steel slag need to be double-checked the content and effects in the separated filter before use for treatment of wastewater.

Table 2 shows the metals content in separate filter including Pb, Hg, Cd, Cr, As, Ni, Cu, Mn, and Zn. They are lower than Standard II water (GB 3838-2002). Basically, this occurs most commonly as second pollution, but not affect heavily on ecological system and human life.

Table 2. The results of heavy metals in separate filter

Heavy metals	Cu	Zn	As	Hg	Cd	Cr	Pb	Fe	Mn	Co
Sample 1	0.012	0.035	0.004	<0.001	0.001	0.003	0.008	0.087	0.001	0.001
Sample 2	0.017	0.044	0.005	<0.001	0.001	0.006	0.009	0.121	0.003	0.001
Sample 3	0.019	0.051	0.008	<0.001	0.001	0.007	0.013	0.159	0.004	0.002

Note: Sample 1, Sample 2, Sample 3 are 0.60 mm, 0.25 mm, 0.15 mm, respectively.

Table 2 shows also that with size of steel slag decreased, metal contents increased in separate filter. In this study, the more size of steel slag is smaller; the strength of absorption pollutants is raising metals content is created, but more content in separate filter. In addition, as it can be seen from table 2-2, with the decrease in the size of steel slag, its heavy metal leaching increased. Previous studies shown that the finer in particle size of the steel slag, the stronger absorption capacity. The current research shows that the finer steel slag particles, the higher the heavy metal dissolution rate.

Table 3. Standard limit of metals in water on the terrestrial surface (mg/L) [31]

Metals	Classification				
	Type I	Type II	Type III	Type IV	Type V
Cu	≤ 0.01	≤ 1.0	≤ 1.0	≤ 1.0	≤ 1.0
Zn	≤ 0.05	≤ 1.0	≤ 1.0	≤ 2.0	≤ 2.0
As	≤ 0.05	≤ 0.05	≤ 0.05	≤ 0.1	≤ 0.1
Hg	≤ 0.00005	≤ 0.00005	≤ 0.0001	≤ 0.001	≤ 0.001
Cd	≤ 0.001	≤ 0.005	≤ 0.005	≤ 0.005	≤ 0.01
Cd^{6+}	≤ 0.01	≤ 0.05	≤ 0.05	≤ 0.05	≤ 0.1
Pb	≤ 0.01	≤ 0.05	≤ 0.05	≤ 0.05	≤ 0.1
Fe solution	Below 0.3	≤ 0.5	≤ 0.5	≤ 0.5	≤ 1.0
Mn totals	Below 0.1	≤ 0.1	≤ 0.1	≤ 0.5	≤ 1.0

4. Discussion

Steel slag removed and adsorbs phosphorus that has two main effects. Firstly, their special structure is possibly created an efficient phosphorus adsorption. Secondly, high CaO content in smelting furnace might create $Ca(OH)_2$ and metal ion very easy in solution, then create to precipitate with phosphorus. The leftover phosphorus caused to eutrophication in waters, steel slag can adsorb phosphorus quickly, not create secondary pollution, simple in operation. The result of steel slag carried out in related experiment shows the increase in leftover steel slag, mixed time and initial concentration of potential hydrogen which influenced efficiency phosphorus absorption. The result such as: steel slag is 0.5g/L, wastewater concentration is105mg/L, pH value is about 7.50~7.60. After an hour, removal rate achieved just over 99% [13,32]. Other research suggested that steel slag adsorb phosphorus by precipitate and exchange of gamete reaction. According to Langmuir isothermal equation, in theory, the amount of absorption is $4.27{*}10^4 mg.kg^{-1}$, the approximate of phosphorus removal is 95% [32]. In wetland, add steel slag that can dissolves Ca^{2+}, Fe^{3+}, Al^{3+} which hydrated and oxidize many compounds then phosphorus is removed lowly, which facilitate precipitate and phosphorus adsorption [33,34]. Steel slag may change structure wetland, improve other land and enhance possibility of endosome Ca, Fe, Al in wetland. Especially, aluminums oxide in steel slag increases possibility phosphorus so enhance phosphorus removal effect in wetland. Therefore, steel slag is good materials that removed phosphorus. However, steel slag should be used only one because its alkaline is not suitable for grow from plants. It may be used on soil surface and middle adsorption layer in wetland [35].

Ions in wastewater that is harmful to environmental and human life. If it is not treated properly so environment will be polluted with increasing of heavy metal. Nowadays, the main methods for treatment of heavy metals are precipitation, biology adsorption, electrolysis membrane, contrary endosmosis, separate electric-absorb evaporation-condensational, ion exchange, electric-condensational, etc. Steel slag includes SiO_2, Fe_2O_3, Al_2O_3, P_2O_5, SiO_2, Fe_2O_3, Al_2O_3, P_2O_5 and other, they combine with large density of steel slag bead that makes condensational speed quickly, solid-liquid separate process shortly, alkalinity and absorption are properly. Heavy metal ions have perceptibility and absorbability, thus, study on treatment of heavy metals may be conducted by precipitation and adsorption methods [36-38]. Some researches suggest to treat nickel by steel slag in wastewater with pH $\geq$ 3, concentration of $Ni^{2+} \leq$ 300 mg/L, weightless ratio of nickel/steel slag is 1/15, contact time 40 minutes, and the effect of treatment is 99% [39, 40]. Other researches treat chromium by steel slag in wastewater which removed Cr^{3+}strongly, concentration of $Cr^{3+} \leq$ 400 mg/L, weightless ratio of chromium /steel slag is 1/35, and the effect of treatment is over 99% [41-46]. The process of heavy metals removal by steel slag was combined steel slag solution with alkaline, heavy metal ions created by hydroxide precipitate, the size and diameter of steel slag were smaller than surface so adsorption certain metal ion [47, 48]. There are not any the publication sonarsenic absorption, but the effect of a few researches is fairly good.

5. Conclusions

In experiment, steel slag includes CaO, SiO_2, Fe_2O_3, $A1_2O_3$, MgO, P_2O_5, MnO, CaS, FeS, etc., in its content of separatedCaO, MgO and iron oxide is possibly increased the effectiveness of adsorption pollutants and accumulation in water. Steel slag analyzed by XRD shows its main contents are magnetite, tri-calcium silicate (Ca_3S), di-calcium silicate (Ca_2S), and iron in tin (FeO). Content of iron and magnetite are 19%.

Steel slag scanned by SEM shows much silver white bead iron; its characteristic structuresarebeneficial to absorption in water. Furthermore, roughness of surface, small gaps which increased areas and effectiveness of absorption. In experiment, heavy metal content in water lowers than Standard II for the earth's surface. The more small steel slag size, the more separate filter was. This is a basis to choose arsenic absorption and removal materials by steel slag.

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Nghiên cứu kết cấu vi mô của xỉ thép trước khi đưa vào hấp thụ và loại bỏ chất ô nhiễm trong nước

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Tóm tắt: Hướng tiếp cận "sử dụng chất thải để xử lý chất thải” có thể tái sử dụng chất thải nhằm mang lại lợi ích về môi trường thông qua khả năng giảm thiểu chất thải vào môi trường và đồng thời mang lại lợi ích về kinh tế cho các ngành xử lý chất thải bằng cách hạn chế phần lớn chi phí xử lý. Nghiên cứu này ứng dụng xỉ thép để làm nguyên vật liệu xử lý ô nhiễm môi trường. Mật độ thể tích của xỉ thép là 3.1~3.6 g/cm^3, thông qua tiêu chuẩn sàng là 0.175 mm, mật độ thể trọng của bột xỉ khoảng 1.74 g/cm^3, so với bề mặt khoảng 0.32 m^2/g, kích thước trung bình là 5.3 nm, đạt các tiêu chuẩn đó sẽ cho hiệu suất lọc tốt. Kết quả phân tích thành phần hóa tính cơ bản của xỉ thép bằng quang phổ XRF cho thấy thành phần chủ yếu của xỉ thép trong thực nghiệm là CaO, hàm lượng 48.2% trong xỉ thép và 42.3 % trong bột xỉ thép, tiếp theo là hàm lượng Fe_2O_3 và MgO; xác định hàm lượng kim loại nặng trong xỉ thép bằng phương pháp TCLP (Toxicity Characteristic Leaching Procedure) cho thấy trong xỉ thép hàm lượng Pb, Hg, Cd, Cr, As, Ni, Cu, Mn, Zn đều rất ít, thấp hơn nhiều so với tiêu chuẩn II trong nước (GB 3838-2002); phân tích kết cấu vi mô của xỉ bằn quét điện tử (SEM) cho biết kết cấu hình dạng thật của xỉ thép và phân tích nhiễu xạ (XRD) cho biết thành phần xỉ thép magnetit, tricalcium silicat (Ca_3S), dicalcium silicat (Ca_2S), sắt trong thiếc (FeO). Lựa chọn phương pháp xỉ thép hấp phụ chất ô nhiễm có thể là phương pháp đơn giản, hiệu quả để đưa vào thực nghiệm và áp dụng trên qui mô rộng cho hiệu quả cao.

Từ khóa: Thải trị thải, xỉ thép, chất độc, nước thải, hấp phụ.