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Experimental investigation of basic oxygen furnace slag used as aggregate in asphalt mixture

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Abstract

Chinese researchers have commenced a great deal of researches on the development of application fields of basic oxygen steel making furnace slag (BOF slag) for many years. Lots of new applications and properties have been found, but few of them in asphalt mixture of road construction engineering. This paper discussed the feasibility of BOF steel slag used as aggregate in asphalt pavement by two points of view including BOF steel slag's physical and micro-properties as well as steel slag asphalt materials and pavement performances. For the former part, this paper mainly concerned the mechanochemistry and physical changes of the steel slag and studied it by performing XRD, SEM, TG and mercury porosimeter analysis and testing method. In the second part, this paper intended to use BOF steel slag as raw material, and design steel slag SMA mixture. By using traditional rutting test, soak wheel track and modified Lottman test, the high temperature stability and water resistance ability were tested. Single axes compression test and indirect tensile test were performed to evaluate the low temperature crack resistance performance and fatigue characteristic. Simultaneously, by observing steel slag SMA pavement which was paved successfully. A follow-up study to evaluate the performance of the experimental pavement confirmed that the experimental pavement was comparable with conventional asphalt pavement, even superior to the later in some aspects. All of above test results and analysis had only one main purpose that this paper validated the opinion that using BOF slag in asphalt concrete is feasible. So this paper suggested that treated and tested steel slag should be used in a more extensive range, especially in asphalt mixture paving projects in such an abundant steel slag resource region. © 2006 Elsevier B.V. All rights reserved.

Keywords: Basic oxygen furnace slag; Micro-structure analysis; Asphalt mixture; Performance; Experimental pavement

1. Introduction

With rapid development of steel industry in China, the amount of the steel slag sharply increases. It is well-known that steel slag is a kind of byproduct during steel making procedure, and the majority of them engrosses a mass of ground and gradually become an environmental problem. Hence availably handling steel slag is more of importance. On the other hand, in the most industrial countries, there is a great demand for aggregate mainly in civil engineering industry, especially in the field of road construction. The last statement holds us responsible to save natural resources by using industrial byproducts and to increase their

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utilization rate wherever their technical properties are suitable concerning the relevant application fields.

The recent convey shows that annual output of steel slag in China is close to 17 million tons and the utilization rate is rather lower. Due to the former research during the last 40 years, today nearly 80% of the produced steel slag is used in the given fields of application, and the remaining 20% of them are still dumped. But more than 1024 million tons natural aggregate resources such as basalt, gravel, limestone and other processed rocks are produced and requested by civil engineering industry. The demand of so many natural aggregate resources either brings about environmental problem, or makes cost of projects increase sharply because of lack of such aggregates. It is known that the steel making industry traditionally produced byproducts which have been used successfully in many fields of application, for example, used steel slag as cement clinker aggregate, phosphatic fertilizer

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in agriculture, metallurgical use, and in waste water treatment. Generally speaking, however, the utilization rate or fields of application of steel slag are rather lower and limited. Contrary to the Chinese steel slag dumping rate is more than 20% [1], so most of them are needed to be used in different fields of application.

BOF steel slag is a final waste material in the basic oxygen furnace steel making process. In basic furnace iron making, limestone (as fluxes) is added to react with the gangue minerals (iron ore and coke) to form iron slag. Slag is separated from the molten iron owing to the different specific gravity of molten iron and slag. In a basic oxygen furnace, the molten iron is converted into steel with oxygen, which also requires some flux materials to react with gangues. The slag formed in the BOF, after being solidified, is called BOF slag [2].

This paper intended to report the characteristics and properties of BOF slag as road construction aggregate, besides the asphalt pavement performance, then evaluate the feasibility of BOF slag used as aggregate in asphalt mixture and pavement construction field of application.

2. Experimental

2.1. X-ray diffraction

X-ray diffraction (XRD) was utilized to give a mineralogy composition of the materials. XRD analysis was performed on BOF slag and measurements were made with a Cu target rotating anode generator with power of 40 kV and 50 mA, in the 10–70° 2θ range at scan speed 20°/min.

2.2. Chemical composition analysis

By using ICP (inductively coupled plasma), the chemical composition of BOF slag can be analyzed quantitatively. Test instrument is model Atom Scan 2000.

2.3. SEM

By the SEM photos, the evident changes of the apparent shape, the porosity size and crystal structure can be observed. Test instrument is model JSM-5610LV produced by JEOL Company.

2.4. Mercury porosimeter

Mercury porosimeter is a technique used to measure the porosity of a substance, in which mercury is forced into the pores of the samples at levels of steadily increasing pressure. The distribution of pore size as well as the total porosity, bulk and apparent density and specific pore volume can be obtained by relationship between the pressure necessary for penetration (the pore dimension) and the volume of penetrated mercury (pore volume) [3]. Based on the work required to immerse a surface in mercury as presented by Rootare and Prenzlow (1967), a formula is written as:

$$\sum \Delta A = -\frac{\sum P \Delta V}{\gamma \cos \theta}$$

where A is the area of solid; V the volume of mercury in pores; γ the Hg surface tension (480 dynes/cm); θ the contact angle between mercury and solid (140°); test instrument is model Poremaster33-6 produced by Quantachrome Company.

2.5. TG analysis

Thermo-gravimetric (TG) analysis was performed to characterize the thermal properties and measure the heat stability and physical process during quality change of BOF slag [3]. Test instrument is produced by Germany NETZSCH Company.

2.6. Mixture performance test

Using small wheel rutting and modified soak wheel rutting test to evaluate the asphalt mixture's high temperature stability and water sensibility.

Single axes compression test has been confirmed that using critical compressive strain energy density at low temperature $(0 \,^{\circ}C)$ to evaluate the asphalt mixture low temperature cracking resistance performance is available. The relevant equation is shown as following:

$$\frac{\mathrm{d}W}{\mathrm{d}V} = \int_0^{\varepsilon_0} \sigma_{ij} \,\mathrm{d}\varepsilon_{ij}$$

where dW/dV is the critical compressive strain energy density function; σ_{ij} the stress fraction; ε_{ij} the strain fraction; and ε_0 is the strain at maximum compressive stress.

The repeated flexural test is conducted to evaluate the fatigue properties of asphalt mixture and, hence, to estimate pavement life for fracture. Repeated haversine loads are applied at the three point of specimen. The load rate is 10 Hz and the size of sample is 5 cm × 5 cm × 24 cm while test temperature is 15 °C. In this paper, test adopts stress control mode and used four kinds of stress level including, $0.3\sigma_f$, $0.4\sigma_f$, $0.5\sigma_f$, and $0.6\sigma_f$ (σ_f means flexural stress in extreme failure). In this mode, the general equation for the plot of log of applied stress versus log of cycles to failure is:

$$N_{\rm f} = K \left(\frac{1}{\sigma}\right)^n$$

where $N_{\rm f}$ is the number of cycles to failure and K, n are regression constants.

3. Material performance

3.1. Chemical and mineral composition

Fig. 1 shows the XRD spectrum for the analysis. By XRD, it can be observed that peaks are so complicated and some of them are overlapped. The main compositions of BOF slag are calcium silicate phases (C_3S , C_2S), and RO phases. These are few amounts of olivine, rhodonite and alite.



Fig. 1. X-Ray diffraction spectrum of BOF slag.

Table 1 Chemical composition of BOF slag

Compound	Content (%)		
SiO ₂	13.71	-	
CaO	45.41		
MgO	6.25		
Al ₂ O ₃	3.80		
FeO	21.85		
Fe ₂ O ₃	3.24		
MnO	3.27		
P ₂ O ₅	1.42		
Na ₂ O	0.10		
K ₂ O	0.06		

The key factors for slag quality exists by many oxides are showed in Table 1 by ICP analysis, such as CaO, MgO, SiO₂, and Al₂O₃. But the key factor to slag quality is the content of free CaO (f-CaO) and free MgO (f-MgO). In steel making procedure, using different resources leads to the change of content of f-CaO and f-MgO. The content of f-CaO and f-MgO is the most important component for the utilization of steel slag for civil engineering purposes with regard to their volume stability. So the volume stability is a key criterion for using steel slag as a construction material.

Fig. 2 shows the IR spectrum, indicating that there are few peaks which almost exist at low frequency area in the spectrum. The spectrum also shows that the stretching vibration of [OH]



Fig. 2. IR spectrum of BOF slag.

Table 2	
Technical properties of BOF slag and natural aggregates	

Characteristics	BOF slag	Basalt	Limestone
Nominal maximum sieve size (mm)	20	20	20
Bulk density (g/cm ³)	3.29	2.90	2.75
Pile density (g/cm^3)	1.92	1.71	1.46
Shape, flat and longest (%)	<10	<10	<15
L.A. abrasion (%)	13.1	14.9	22.0
Crushing value (%)	12.0	12.9	15.1
Polishing stone value (%)	57	49	44
Water absorption (%)	1.18	0.70	1.05
Binder adhesion (%)	>95	>85	>90

appears at 3693 cm⁻¹ which is not clear, and the stretching vibration of H₂O appears at 3422 cm⁻¹ while it's bending vibration at 1797 cm⁻¹ because of existing water absorption. Absorptions of higher intensity are found between 875 cm⁻¹ and 1033 cm⁻¹ due to the Si–O valence vibrations of SiO₄, and other absorption of higher intensity appear at 521 cm⁻¹ due to the Si–O valence vibrations of SiO₄.

3.2. Technical properties

As it is known, only those high quality aggregate can be used in stone matrix asphalt (SMA) mixtures. Therefore, the technical properties of processed aggregates which are used for these constructions are of fundamental importance. The most important properties test results are shown in Table 2.

According to Table 2 BOF slag can be processed and used as aggregate of high quality which comparable with those natural aggregates. The high bulk density qualifies steel slag used as construction materials. The loose extend and pile density of BOF slag is 1.92 tonnes/m³, rather higher than the natural aggregates'. The two indexes of density guarantee good crushing resistance and abrasion resistance performances of BOF slag by L.A. Abrasion and Crushing tests. These are two very important index of road construction aggregate because of rolling and contacting when paving asphalt road. The later index of density above indicates the cube shape of slag particle and lower flat and longest value, also guarantees internal friction angle. This is more important for aggregate used in SMA because of stone on stone contact requirement [4]. All other technical properties listed in Table 2 are comparable or even better than those of natural aggregates. In particular the high level of strength described by the impact and crushing value and additionally the rough surface texture are predominant. In a view of durability and water resistance, a binder adhesion >95% qualify the slag as aggregates for high traffic road layers under high temperature especially for asphalt surface layers. Successfully using in hydraulic engineering and backfilled ground base materials purposes, this confirm those good technical properties of BOF slag and guarantees to be used in asphalt layer against to rut, deformation and polishing.

3.3. SEM

The shape of materials, surface texture even particle distribution of BOF slag can be observed and analyzed by scanning



Fig. 3. SEM micrograph of BOF slag



Fig. 4. SEM micrograph of basalt.

electron microscopy micrograph and compared with those natural aggregates such as basalt and limestone [5]. Figs. 3–5 shows the BOF slag, basalt, and limestone's SEM micrographs.

According to these micrographs, the slag shows different in texture and morphology from natural aggregates, especially in porosity characteristic. This difference makes slag surface texture rougher than other natural aggregates, and of course is a factor that will affect their adhesion ability with asphalt binder. Further research on porosity characteristic would be discussed.

10kv ×3,000 5µm

Fig. 5. SEM micrograph of limestone.

Table 3	
Mercury porosimeter analysis results	

Aggregate type	Total porosity (%)	Proportion of pore size with various diameters (%)			
		>0.001 µm	>0.01 µm	>0.1 µm	>1 µm
BOF slag	5.76	100	99.9	48.0	20.5
Basalt	0.24	100	100	100	58.3
Limestone	4.26	100	100	99.5	84.7

3.4. Porosity

Test was performed on a Quantachrome Instrument Poremaster while the maximum pressure is 200 MPa. In a view of comparing porosity characteristic, BOF slag, basalt and limestone were tested by this means. The pressure varied from 0.2 MPa to 191 MPa. Test results are showed in Table 3. About 5 g of sample were used for each experiment.

During the test, mercury was forced to enter into the pores by applying controlled increasing pressure. Test results clearly indicate the total porosity of all aggregates is listed in approximate order of value, and the total porosity of BOF slag is greater than other aggregates'. The difference of proportion of pore size that is the mean pore diameter is larger than 0.1 µm is notable. The SEM micrographs also confirm this difference, and on the BOF slag micrographs, the pores that diameters are below 1 μ m can be distinguished clearly, but there are few pores on the other micrographs at the same resolution factor. Analyzed on the test data, it is easy for mercury to be penetrated into pore at the pore diameter size ranging from 0.01 µm to several micrometers, namely the greatest contribution to total pore volume comes from the larger pores. This nearly agrees with the former research [4]. It can be deduced that the good asphalt binder adhesion is relevant to these pores (rough microstructure texture) because of good asphalt binder adhesion of BOF slag. But whether these great contributions pores have good relation with the asphalt binder adhesion or absorption/adsorption, even which pores' diameter size level is the key factor to influence the characteristic of the adhesion or absorption/adsorption between aggregates and binders remain unknown and still need to be researched in the future.

3.5. TG analysis

Fig. 6 shows the curves of TG analysis. There are two distinct mass changes, -9.83% and -3.67% at which temperature are 736 °C and 90.8 °C, respectively. The former reflected the rapid reaction rate and instability of ground fine slag, however, the later correlated with the thermal reaction of dehydration. These two mass losses confirm that the fine slag aggregate instability after slag was ground. It also shows that BOF slag crystalline phases appear defect and distortion of lattice after ground because a number of crystalline phases, the main minerals of BOF slag, have been turned into the non-crystalline phases. Namely, the ground process makes reaction easier and increases the instability of fine slag. But the characteristic and relation between the ground fine slag aggregate with asphalt binder is



Fig. 6. TG curve of BOF slag.

an interesting subject and needs to be studied in the follow-up study.

4. Performances of BOF steel slag asphalt mixture

4.1. Design and preparation

Commonly, aggregates which are used in design and preparation of asphalt mixtures are required keeping homogeneous and original state in characteristic. It means same type aggregate is permitted to be used in a continuous gradation of asphalt mixtures. But for the industrial byproducts, because of instability of slag it is impossible to use whole gradation of BOF slag as to use natural aggregate. According to test on coarse (>4.75 mm) and fine (>4.75 mm) slag aggregate, this paper found that the characteristic of fine slag aggregate with asphalt binder is rather poorer than those natural fine aggregates'. Excessive asphalt binder absorption causes the thinner asphalt film and lower cohesive affinity for fine slag aggregate, what's more, the angularity of fine slag aggregate is inferior to the course's, and this may have effect on the durability of asphalt mixtures. Fig. 7 shows the gradation curve.

Based on the above, we have decided to use in this paper coarse BOF slag and common natural fine aggregate together to design and prepare the steel slag stone matrix asphalt mixtures (S_{mix}). There are two main purposes to decide applying SMA mixture, one is due to BOF slag aggregate's characteristics and properties, and the other one is to keep agreement with this paper's major idea that save natural aggregate resource and handling waste slag due to nearly 80% of coarse slag used in mixture as aggregate.



Fig. 7. Gradation curve.



Fig. 8. Rate of expansibility vs. time.

4.2. Activity test

Due to the instability and expansibility of steel slag, activity test is necessary to design S_{mix} . The procedure of activity test is to put S_{mix} samples into the water at 60 °C for 24 h, 48 h, 72 h, 96 h, and 120 h. By using the drainage contain, the volume of samples which were dipped in water for different hours could be measured and compared with the dry samples.

The result of the activity test of the S_{mix} is shown in Fig. 8. Rate of expansibility of steel slag mixture samples increased slowly and more close to 1%, the smoother the curve shows. Based on the result of tests and regression equation of curve, it is confirmed that the rate of expansibility of S_{mix} is less than 1%. This is an important index that guarantees the S_{mix} to be used in road construction.

4.3. High temperature stability

There are many factors to influence the high temperature stability of asphalt mixture, but always can be induced to two points: inside and outside conditions. The former reflects the characteristics and properties of materials themselves, and the later always includes the climate and traffic load.

Among those factors which caused permanent deformation, the properties of aggregates that have effects on the high temperature stability so much are those key factors. Commonly, crushing, cube shape, angularity, consistency and roughness texture of aggregates extremely influence the high temperature stability of asphalt mixtures. Based on the above test on BOF slag technical properties, S_{mix} appeared rather high temperature stability and rut deformation resistance ability that is logical and superior to the others.

Fig. 9 shows the dynamic stability at different temperature, which used steel slag, basalt, and limestone respectively as aggregate. Test results indicate that the high temperature stability of S_{mix} is best excellent of all mixtures' at any other temperature. Because of good granular shape (nearly cube), adhesion bond with asphalt binder, abrasion resistance, PSV, and impact resistance, steel slag mixture can withstand more traffic load and higher temperature than other mixtures do. So S_{mix} shows



Fig. 9. Dynamic stability vs. temperature.

rather excellent permanent deformation resistance performance [6,7].

4.4. Water sensibility

Water damage is one of familiar early damage forms which causes the quality and decreases service life of asphalt pavement.

Recent researchers [8,9] show more interesting in water resistance performance of resistance performance of asphalt mixtures. This paper utilized the soak wheel track depth in some way and it is feasible to use the wet rut depth evaluate the water test to asphalt mixtures. More and more investigations indicate that water influence the rut evaluate the effects of water on steel slag asphalt mixtures (S_{mix}) sample's permanent deformation under different temperatures and loads, 60 °C, 70 °C, and 0.7 MPa, 0.9 MPa, respectively. In order to close to and simulate the asphalt pavement practical condition, S_{mix} samples were pre-treated by two different procedures, soak and saturation with freezing and thawing cycle. The wet rut depth results are given in Figs. 10 and 11.

There are three kinds of fibres, polyester, polyacrylonitrile, and lignocelluloses fibres were used in S_{mix} in soak wheel track test. The curves and columns reflected different wet rut depth of S_{mix} with the ambient temperature and load changing. On



Fig. 10. Rut depth without freezing and thawing.



Fig. 11. Rut depth with freezing and thawing.

the curves, it can be concluded that nearly among all of results, under the same temperature and load, the wet rut depth without freezing and thawing is great than that with freezing and thawing cycle. This seems different to the former research (Theresa M. Williams, 1998) [8] that considered that the results showed no difference before and after the rigorous pre-conditioned such as soak, freezing and thawing cycle in the higher temperatures and loads cause of the internal connective pores water in asphalt mixtures were blocked by the kinkled and stripping fine mixture particles under freezing and thawing cycle [9]. But, the authors thought that the main factors of load and water resistance are binder adhesion bond and aggregate internal friction resistance, it can be concluded that undergoing the rigorous pre-conditioned procedure, binder adhesion bond deceased, then stripping and cracking appeared, and as a result ability to withstand deformation in water decreased in some way. Comparing the Fig. 10 with Fig. 11, the decrease is not distinct due to internal friction resistance S_{mix} does still work primarily, and this may be great difference from other mixtures under soak wheel track test. The authors noticed that singly changing the test condition, temperature or load, the wet rut depth changed in some tendency, which meant the wet rut depth changing range while keeping same load (0.7 MPa or 0.9 MPa) and changing temperature from 60 °C to 70 °C is great than that while keeping same temperature ($60 \degree C$ or 70 °C) and changing load from 0.7 MPa to 0.9 MPa. This paper concluded that as the indications of soak wheel track test, it was hard for S_{mix} to be destroyed easily by water under the higher temperature instead of the heavier load.

4.5. Low temperature cracking resistance

By running single axes compression test at 0 °C, critical compressive strain energy density value can be achieved and used to evaluate the low temperature cracking resistance performance [10,12]. Table 4 indicates the test results while used three aggregates, steel slag, basalt and limestone.

Fig. 12 shows the relation about compression stress versus strain. By regression analysis, the compression stress versus strain function can be built. Based on the equation, the critical compressive strain energy density value can be calculated. Table 4 show the critical compressive strain energy density value

 Table 4

 Single axes compression calculated result

Aggregate type	ε_0 (%)	dW/dV (kJ/m ³)		
Steel slag	13.5	89		
Basalt	11.0	74		
Limestone	7.5	58		

different with aggregate changed and the value of S_{mix} is the highest of all, which shows that S_{mix} appears rather excellent low temperature crack resistance performance because S_{mix} needs more energy than other mixtures when it reaches material failure situation.

Lots of investigations [10–12] considered that one of the most factors to influence the low temperature cracking resistance performance is asphalt binder characteristics, such as temperature sensibility, stiffness, penetration and ductility, etc. of asphalt, but this paper thought that the aggregate properties are influencing factors directly either, especially the adhesion bond and internal friction angle. The authors have performed same test on those used good performance asphalt binder (PG76-22) and poor properties aggregates which against to two points above. The critical compressive strain energy density at 0 °C is still rather low. That meant the low temperature cracking resistance performance upgraded not in evidence without taking consideration of asphalt binder and aggregates properties.

4.6. Fatigue life

There are two kinds of load controlling modes for asphalt mixtures fatigue test: one is stress controlling mode and the other is strain controlling mode. This paper applied the stress controlling mode on S_{mix} because the former appeared more practical to asphalt pavement [13].

Table 5 shows the results of three point flexural test for three kinds of asphalt mixtures (used three types aggregate and same gradation) and their fatigue equations while Fig. 12 shows the curves by using the stress ratio (σ/σ_f) versus cycles to failure. From Fig. 13, *K* and *n* value vary in little scope, but fatigue life of S_{mix} is superior while using different aggregate and same gradation. Nearly 1.2×10^5 cycles load to failure make S_{mix} appear good fatigue resistance performance.



Fig. 12. Compression stress vs. strain.

Table 5
Flexural test results

Aggregate type	$\sigma_{\rm f}$ (MPa)	Fatigue equation
Steel slag	1.64	$\log N_{\rm f} = 4.34 - 3.50 \log \sigma$
Basalt	1.52	$\log N_{\rm f} = 3.78 - 3.28 \log \sigma$
Limestone	1.18	$\log N_{\rm f} = 3.42 - 3.20 \log \sigma$



Fig. 13. Repeated cycles at different stress ratio.

4.7. Test road approach

The performances of S_{mix} indicate that it can fulfill the requirement of asphalt pavement in expressway construction well. So test road was paved on the old expressway asphalt surface as skid resistance and abrasion resistance layer. This test road has two directions and four lanes with 2 km long and 24 m wide. The design vehicle speed is more than 110 km per hour. For the sake of testing performance of S_{mix} accurately and strictly, the location of test road was decided under more considerations. For instance, the annual rainfall is near 2000 mm and the average highest temperature during summer day is about $40 \,^{\circ}\text{C}$ while below $-10 \,^{\circ}\text{C}$ during winter. The traffic volume is more than 3×10^7 ESALs. So in such a location, whatever environment or traffic condition, to asphalt pavement, is rather atrocious. Under this condition, near the 2 years service, the steel slag test road appears excellent performance, such as roughness, BPN coefficient of surface, without coming into being the rutting, cracking, and stripping which render the asphalt pavement early damage. Table 6 presents the test results clearly and indi-

Table 6	
Performance of	test road

Test item	Service time				
	6 months	12 months	18 months	24 months	
Bulk density of core (g/cm ³)	2.511	2.520	2.524	2.525	
Abrasion and friction coefficient (BPN)	62	60	56	55	
Surface texture depth (mm)	1.2	0.9	0.8	0.8	

cates that in this 2 years, how the pavement performance of the test road changes.

5. Conclusions

By testing and analyzing, BOF steel slag can be used as asphalt mixture aggregate in expressway construction, even some of the performances of S_{mix} transcends common mixture which uses natural resource. Whatever pavement performance or driving quality of S_{mix} pavement can meet requirement of expressway construction. The S_{mix} test road is successfully paved and normally used that demonstrated that point more forcefully. So this could be a new approach to treat and utilize the biggest solid waste, steel slag, in terms of amount of steel slag will decrease, the threat to environment will disappear, and the natural resource will be protected and saved if this way can be validated and applied availably in other areas.

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