Int. J. Chem. Sci.: 14(S1), 2016, 188-202
ISSN 0972-768X
www.sadgurupublications.com

UTILIZATION OF COPPER SLAG IN BITUMINOUS MIX

B. JAYASHREE*, S. SANTHANU and B. KALAIARASAN

Department of Civil Engineering, Aarupadai Veedu Institute of Technology,
CHENNAI – 603104 (T.N.) INDIA

ABSTRACT

Copper slag is considered as one of the waste materials which will have a promising future in construction industry as a partial substitute of either natural aggregates. For each ton of copper production, about 2.2 tonnes of copper slag is generated. In order to reduce the accumulation of copper slag and also to provide an alternative material to aggregates an approach has been done to investigate the use of copper slag in the partial replacement of bitumen mix.

Key words: Copper slag, Marshall stability test, Indirect-tensile strength, Waste material.

INTRODUCTION

In India, there is great demand of aggregates mainly from civil engineering industry for road and concrete constructions. Instead of using natural aggregates in road and concrete constructions some of the waste industrial by products can be used widely. Many highway agencies, private organizations, and individuals are in the process of completing a wide variety of studies and research projects concerning the feasibility, environmental suitability, availability of waste products and performance of using waste industrial produces in highway construction. These studies are mainly to match society's need for safe and economic disposal of waste products with the highway industry's need for betterment and more cost-effective construction materials.

This study is to explore the potential use of copper slag (CS) as fine aggregate (up to 15%) in the design of bituminous mix like Dense Bituminous Macadam (DBM), which enhance the property of the bituminous mixes.

Copper slag as construction material

The processing of most ores involves a series of some standardized steps. After

---

*Author for correspondence; E-mail: Jaisri2charu@gmail.com

mining, the bulk ore is processed to remove any gangue (excess waste rock and minerals). This process typically consists of pulverizing (grinding) the ore to a relatively fine state, followed by some from of gravity separation of the metal s from the gangue (using a series of devices including cyclone separators, inclined vibratory tables, and flotation tanks). The ore, which is refined are processed thermally to separate metal and nonmetal constituents and then further reduced to free metals. Since most of these metals are unsuitable for use in a pure state, they are subsequently combined with other elements and compounds to form alloys having the desired properties.

In preparation for metal ion reduction some non-oxide minerals are often converted to oxides by heating at air temperatures below their melting point. Sulphide minerals, when present in copper, are converted to oxides. The reduction of metal ion to free metal is normally accomplished by smelting. In this process, a reducing agent, such as coke as impure carbon, along with CO and H2, is combined with the roasted product and melted in a siliceous flux. The metal is then subsequently gravimetrically separated from the composite flux, leaving the residual slag.

CS is produced by three process (Fig. 1):

(i) Roasting process, in which sulphur in the ore is eliminated as sulphur dioxide (SO_2)

(ii) Smelting process, in which the roasted product is then melted in a siliceous flux and the metal is reduced.

(iii) Converting process, where the melt is desulphurized with lime flux, iron ore, or a basic slag and then oxygen is lanced to remove other impurities.

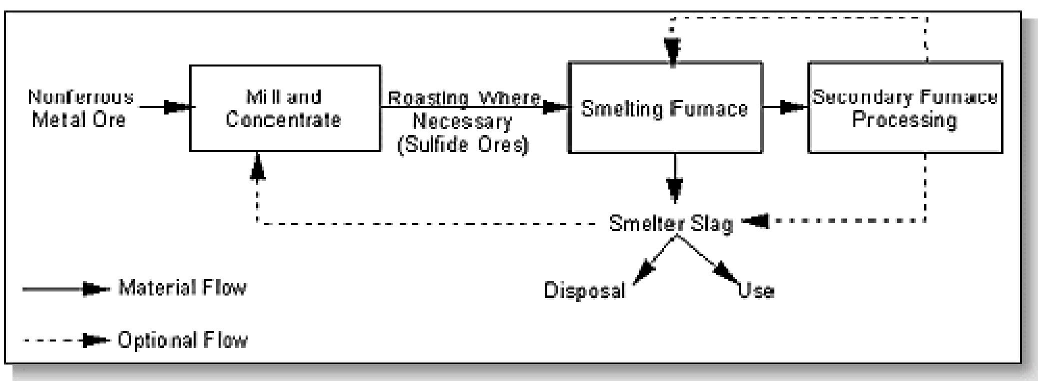

Fig. 1: Production of copper slag

In India, copper slag are produced about 60000 MT per month. The companies which produce Copper slag now wants to focus on road, abrasives and other industries. The slag is highly stable and non-leachable in nature as per scientific estimate, for every tone of copper metal produced, around 1.8-2.2 tonnes of slag is generated. With increasing scarcity of river sand and natural aggregates across the country, construction sector is under tremendous pressure to explore alternative to these basic construction material to meet the growing demand of infrastructure demands. At present, across the world around 33 tons of slag is generated while in India three copper producers Sterlite, Birla Copper and Hindustan Copper Produce around 6-6.5 tones of slag at different sites. The utility of copper slag as alternative material for other industrial in state like Kerala, Maharashtra and Gujarat and other states sand mining in rivers is already been banned owing to its disastrous Impact Ecology. Therefore, slag has a big potential of getting developed as a suitable alternative material to these resources.

Table 1: Properties of aggregates

Properties	Unit	Method of test	Test value
Properties of coarse aggregate			
Specific gravity	-	IS : 2386 (I)	2.728
Flakiness and elongation (combined) Index	%	IS : 2386 (I)	35
Water absorption	%	IS 2386(I) (Part-3)	0.2
Stripping	%	IS : 6241	95
Impact value	%	IS : 2386 (IV)	12.5
Properties of copper slag			
Specific gravity	-	IS : 2386 (I)	3.456
Unit weight	Kg/cum	IS : 2386 (I)	1173.285

Laboratory investigation

DBM are base course layers, in construction. This investigation aims to use CS as construction material in bituminous road construction. In India, no mix design procedure is available till date for closed graded mixes like DBM, which has a tendency to crumble at 160^0C because of more closed texture. Since DBM is used as a base course layer below the wearing course and it seems to be logical that the stability at temperature of 160^0C. Therefore, the testing temperature was adopted at 160^0C.

Marshall method of mix design

The overall procedure for mixture design always begins with acceptance tests performed on the aggregates and bitumen considered for the design.

Aggregates

Stone aggregates, the major components of road structure, bear load due to particle interlocking and sustain wear and tear due to vehicular movement. The acceptability limits may vary depending upon the type of construction (Table 1). Aggregate is a broad category of coarse particulate material used in Construction field. Aggregates are the most mined materials in the world. The aggregate serves as reinforcement to add strength to the overall composite material. CS is a black, glassy and vesicular matter (Unit wt. 1173.285 kg/m3, water absorption 0.2% and having a specific gravity of (3.456).

Marshal stability test

Absolute viscometer& kinematic viscometer

Bitumen

Bitumen (C, 80-87% by wt) is basically a hydrocarbon, less than 10 percent by weight is due to atoms of S, N and O, which are attached to hydrocarbon molecules. Three basic components of bitumen are (i) asphaltene, (ii) maltene, and (iii) carbine. Asphaltene is hard and aromatic. Maltene is a solvent and imparts viscoelasticity to bitumen. It is resin like intermediate molecule of hydrocarbon. Carbine is the fraction, which is insoluble in CCl4. Bitumen as a material has drawn attention to the engineers since a long time because it is (i) water proof, (ii) durable, (iii) resistance to strong acids, and (iv) possesses good cementing properties.

At normal temperature, bitumen is a thermoplastic semi-solid cementing material and at higher temperature, bitumen behaves like a viscous liquid, whereas at a very low temperature bitumen is brittle as glass. Bitumen is believed to behave viscoelastically at the standard operating temperatures of highways. VG30 bitumen has been used as binder (Table 2).

Table 2: Properties of VG 30 bitumen

Properties	Unit	Method of test	Test value
Penetration at 25^0C	0.1 mm	IS 1203:1978	66
Softening point, R & B	^{0}C	IS 1205:1978	46
Ductility at 25^0C	cm	IS 1208:1978	75+
Specific gravity	-	IS 1206:1978	1.038
Viscosity at 60^0C	poise	IS 1206:1978	2950
Viscosity at 135^0C	cst	IS 1206:1978	380

Proportion of aggregates

Aggregate gradation is one of the most important properties in bituminous mixture, which affects almost all the important properties like stability, durability, workability and resistance to moisture damage. Therefore, gradation is a primary consideration of bituminous mix design. The typical aggregate gradation (Fig. 2) is taken for the design of DBM, grade 2 as per the MORTH Specification in order to explore the potential use of Copper Slag in optimum level, which enhances the property of the mixes to get the final grading (Tables 3, 4).

Table 3: Combination of materials DMB grade 2

Size of aggregate	Job Mix (%)				
	Conventional mix	CS 10%	CS 15%	CS 20%	CS 25%
26.5	32	32	28	26	24
13.2	15	15	15	14	15
6.7	15	12	12	14	12
2.36	38	31	30	26	24

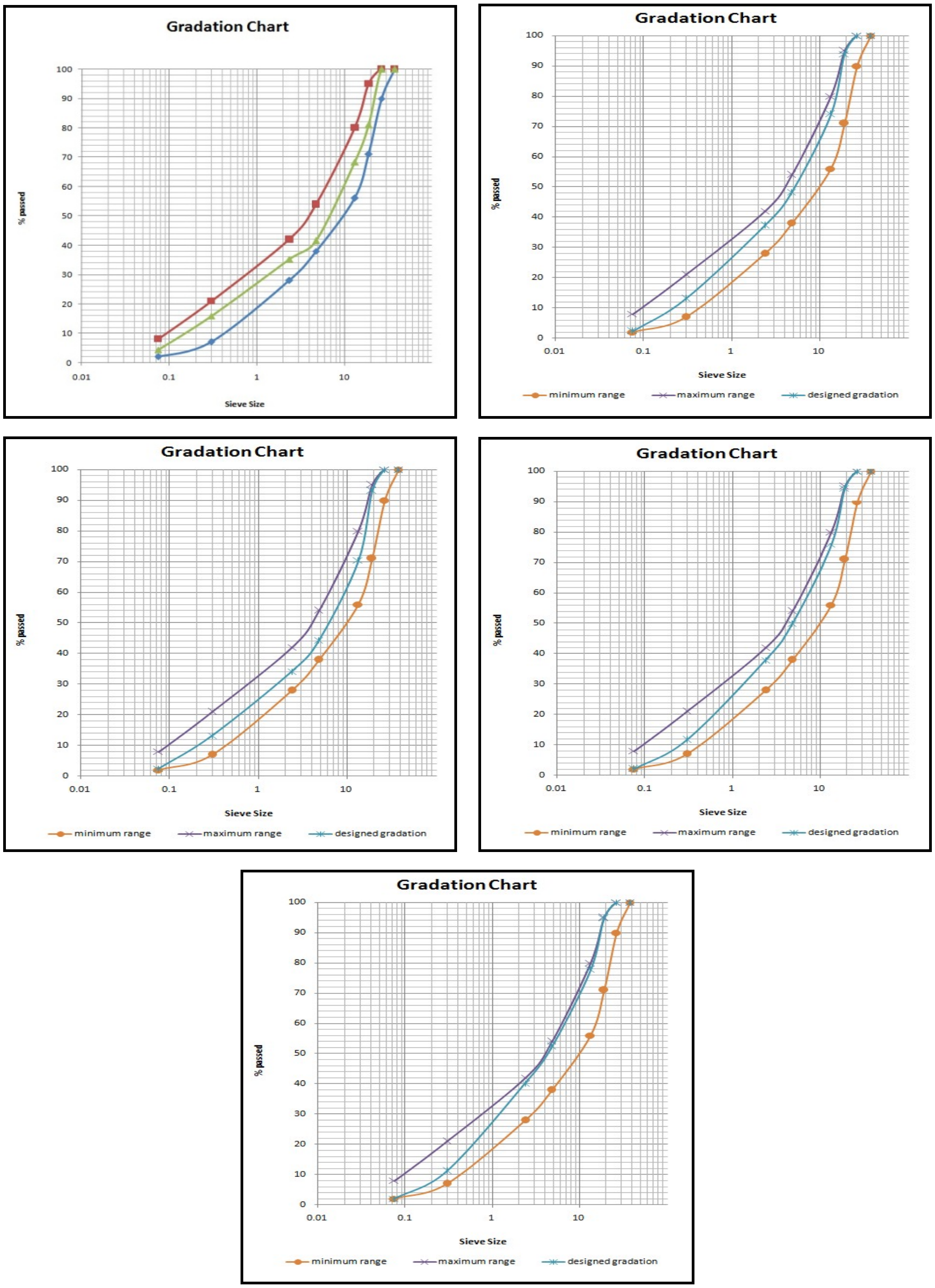

Fig. 2: Gradation charts for conventional mix & CS 10%, 15%, 20%, 25%

Table 4: Weight retained & passed

Sieve size (mm)	Weight retained							
	26.5 mm		13.2 mm		6.7 mm		2.36 mm	
	Wt.Rt.	% Passed	Wt.Rt.	% Passed	Wt.Rt.	% Passed	Wt.Rt.	% Passed
37.5	0	100	0	100	0	100	0	100
26.5	0	100	0	100	0	100	0	100
19.0	429	78.55	0	100	0	100	0	100
13.2	1421	7.50	0	100	0	100	0	100
4.75	150	0.00	2000	0.00	1439	28.05	0	100
2.36	0	0.00	0	0.00	561	0	153	84.70
0.3	0	0.00	0	0.00	0	0	442	40.50
0.075	0	0.00	0	0.00	0	0	329	7.60
Total	2000		2000		2000		924	

Determination of optimum binder content

At each grading, Marshall Samples were prepared by varying the binder content and tested for its volumetric properties. Significance of Volumetric Parameters: Bitumen holds the aggregates in position and the load is taken by the aggregate mass through the contact points. If all the voids are filled by bitumen, load is transmitted by hydrostatic pressure through bitumen, and strength of the mix, therefore, reduces. That is why stability of the mix starts reducing when bitumen content is increased beyond certain value. Also during summer season, bitumen melts and occupies the void space between aggregates and if the void space is not available, it causes bleeding. Thus, some amount of void is necessary in a bituminous mix, even after the Final stage of composition for determination of optimum binder content (OBC), the value of stability and air voids are plotted against the binder contents (Fig. 3-7).

Selection of Optimum Bitumen Content (OBC)

OBC is a delicate balancing act in which there are a number of variables – like voids in mineral aggregate (VMA), air voids (VA), and voids filled with bitumen (VFB).A balance is to be maintained such that all the specification limits recommended in the code of practice are simultaneously satisfied. OBC for various mixes was found as follows (Table 5): Conventional mix, 4.70; 10% CS, 4.78; 15% CS, 4.78; and 20% CS, 4.89%.

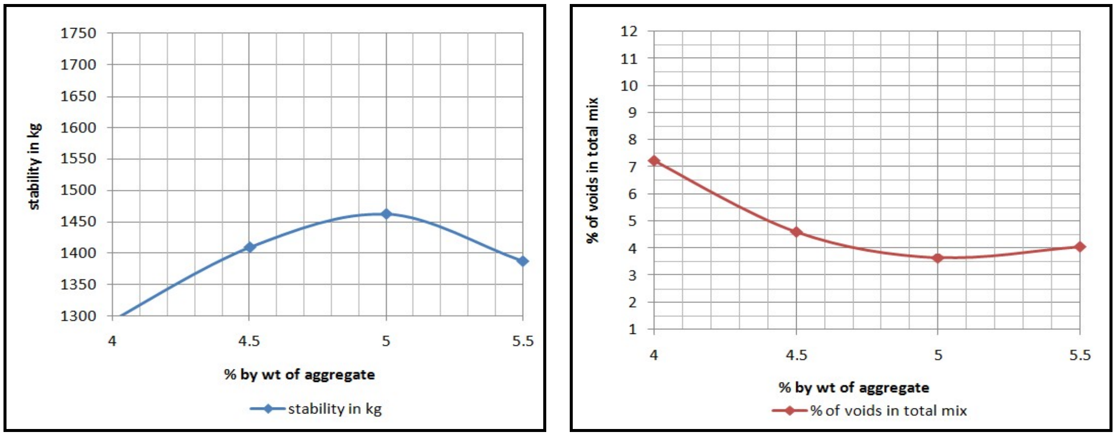

Fig. 3: Parameters for conventional mix

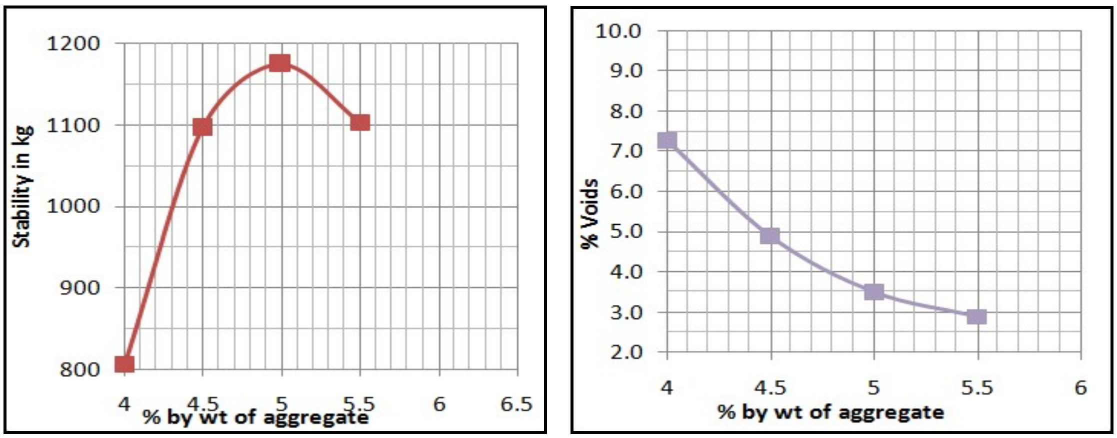

Fig. 4: Parameters for CS 10%

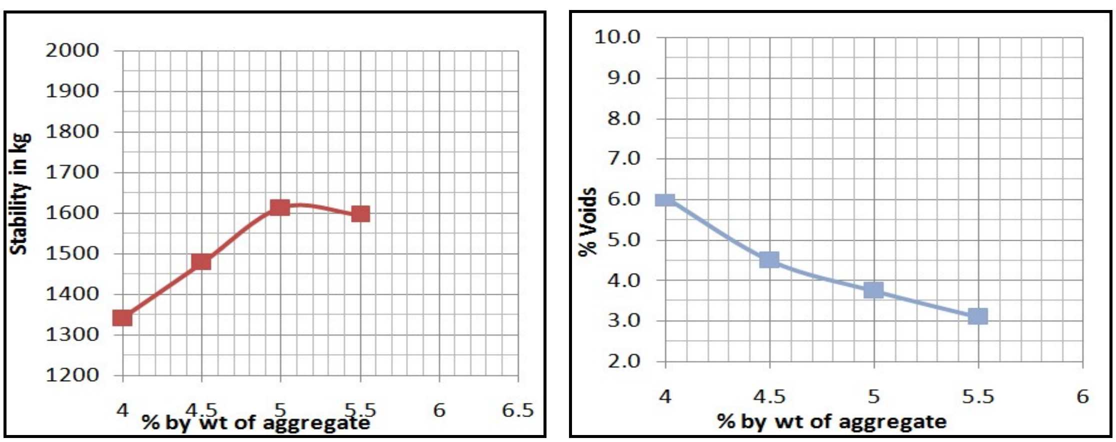

Fig. 5: Parameters for CS 15%

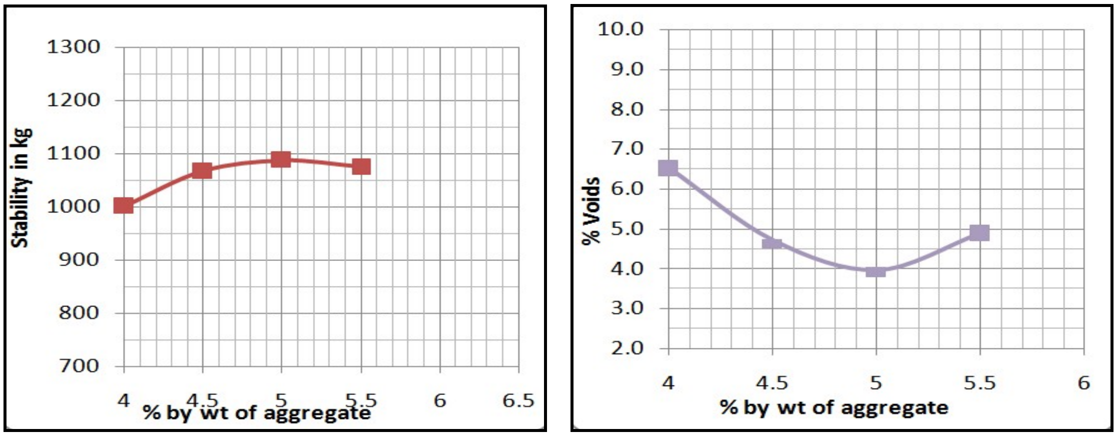

Fig. 6: Parameters for CS 20%

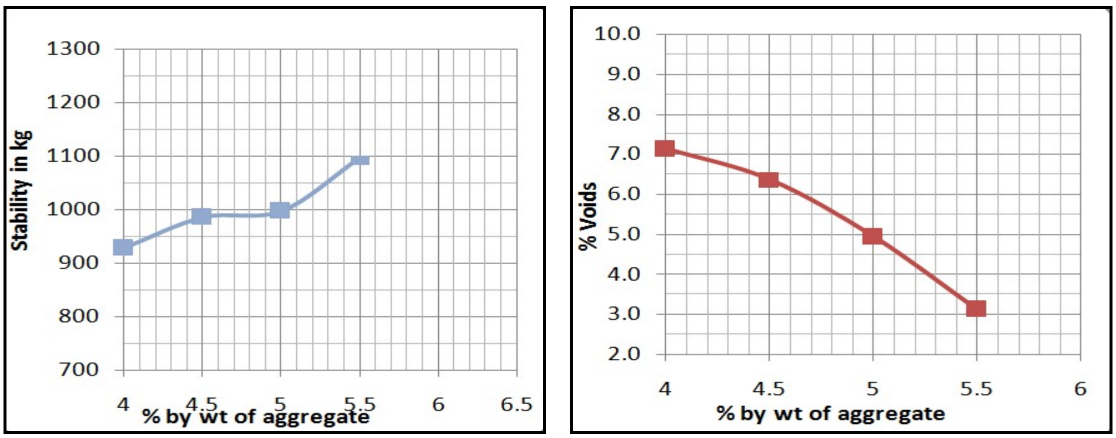

Fig. 7: Parameters for CS 25%

Table 5: Properties of DBM grade II mix using CS OBC

Property	Required criteria	Conventional mix	10% CS	15% CS	20% CS	25% CS
Binder % by wt of mix	4.5	4.49	4.56	4.56	4.67	5.02
Air voids, %	4.0	4.22	4.119	4.070	4.131	4.135
VMA, %	12	14.75	14.935	15.020	15.446	16.381
VFB, %	65-75	71.39	72.454	72.871	73.231	74.652
Stability, kg	900	1430	1139.8	1553.09	1082.53	1003.13
Flow, mm	2-4	3.25	2.885	3.035	3.024	2.985

Thermo gravity analysis

Thermo gravimetric analysis (TGA) is a method of thermal analysis in which changes in physical and chemical properties of materials are measured as a function of increasing temperature (with constant heating rate), or as a function of time (with constant temperature and/or constant mass loss). TGA is commonly used to determine selected characteristics of materials that exhibit either mass loss or gain due to decomposition, oxidation, or loss of volatiles. TGA (Figs. 8, 9) can provide information about chemical phenomena (Eg : oxidation or reduction.

Fig. 8: TGA Analyzer

Fig. 9: Hot furnace (TGA)

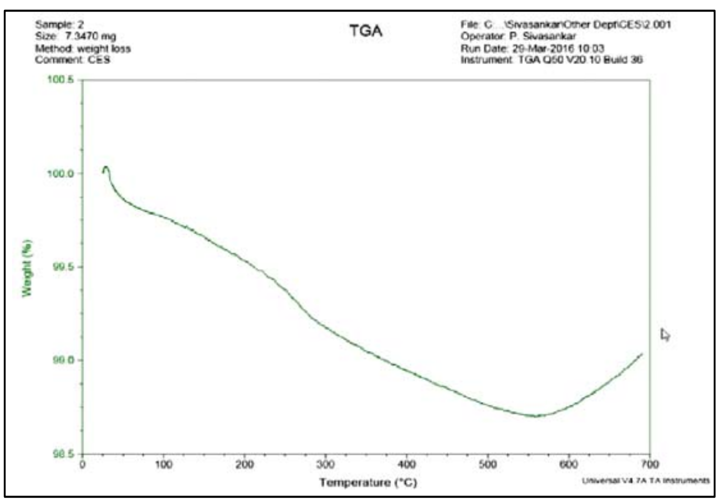

Fig. 10: TGA Graph

Tests for the sample copper slag (Fig. 10) sent by Anna University, results show that there are no changes in weight % from temperatures (150 degree to 200 degree). The temperature of bitumen heating for road construction is about 160^{o}C. Thereby it does not exhibit either mass loss or gain around 160-200^{o}C temperatures.

SEM - (Scanning electron microscope analysis)

SEM provides detailed high resolution images of the sample by rastering a focused electron beam across the surface and detecting secondary or back-scattered electron signal. An Energy Dispersive X-Ray Analyzer (EDX or EDA) is also used to provide elemental identification and quantitative compositional information. SEM provides images with magnifications up to ~X50,000 allowing sub micron-scale features to be seen i.e. well beyond the range of optical microscopes.

Element	Wt%	At%
OK	22.43	41.81
FK	03.99	06.26
MgK	01.16	01.42
AlK	02.52	02.79
SiK	18.26	19.38
KK	00.79	00.61
CaK	02.74	02.04
FeK	48.11	25.69
Matrix	Correction	ZAF

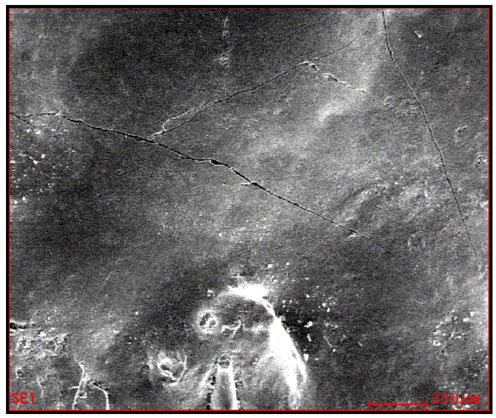

Fig. 11: Sample 1 (Normal copper slag)

Element	Wt%	At%
CK	75.87	86.02
OK	07.89	06.72
NaK	00.57	00.34
MgK	00.49	00.27
AlK	02.22	01.12
SiK	06.69	03.24
SK	03.38	01.43
KK	00.43	00.15
CaK	01.17	00.40
FeK	01.30	00.32
Matrix	Correction	ZAF

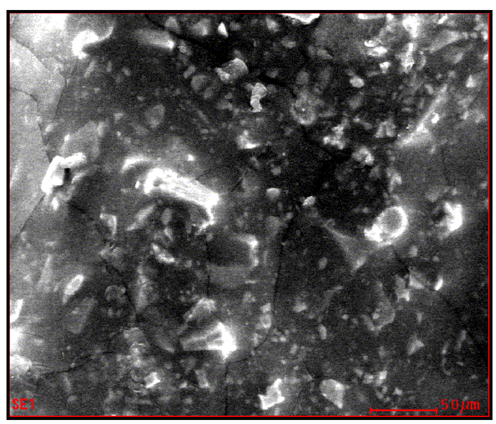

Fig. 12: Sample 2 (Bitumen mixed Copper slag)

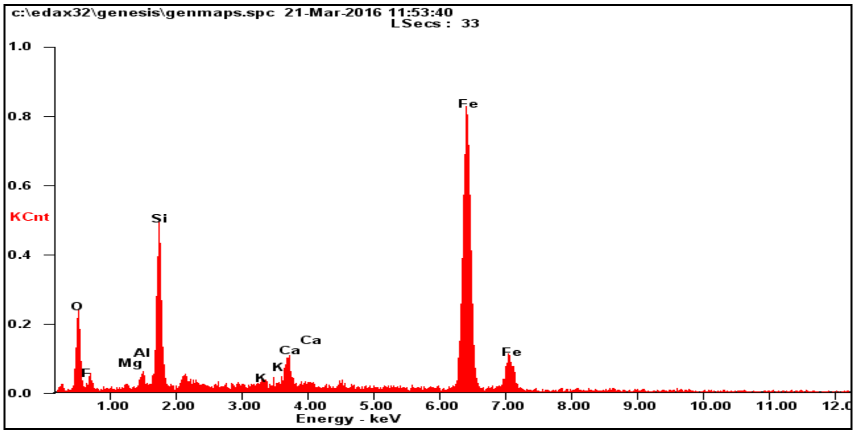

Fig. 13: Normal copper slag-elemental analysis graph

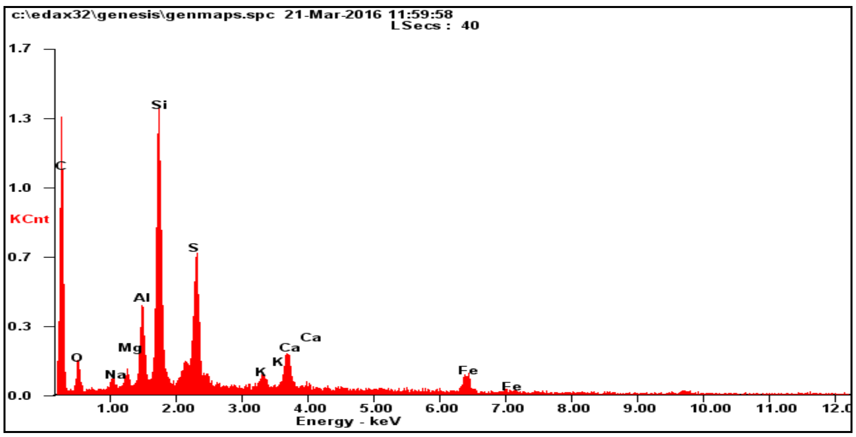

Fig. 14: Bitumen mixed copper slag-elemental analysis graph

EDS Analysis, IIT MADRAS

Tests for the Sample 1 & 2 (Fig. 11, 12) sent by SAIF, IIT Madras ; the results show that there is no change in chemical composition as in Normal Copper slag and bitumen mixed Copper slag. The elemental analysis graph indicates that the elements which are present in both normal and Bitumen mixed copper slag are well within the permissible limits and hence these samples are non-hazardous in nature and can be used in Highway road construction.

Computation of moisture sensitivity

For Moisture sensitivity test as per AASHTO T 283, samples were tested for dry and wet strength conditions at OBC. The wet set was first placed in water bath maintained at 60ºC for 24 hr and then placed in an environmental chamber at 25ºC for 2 hr. The load was applied at the rate of 50 mm/min by loading a Marshall specimen with compressive load acting parallel to and along the vertical diametric loading plane. The moisture sensitivity is determined as a ratio of the average tensile strengths of the wet and dry tensile strength of the specimens. The Indirect Tensile Strength (ITS) is calculated from the equation given below:

$$S_t = 2\ p/\pi\ d\ t$$

where, P = load (kg), d = dia of specimen (cm), t = thickness of specimen (cm).

The moisture sensitivity tests on Bituminous mix provides information to evaluate the effects of moisture and stipping potential of Bituminous mixes (Table 6).TSR values above (90%) of all bituminous mix improves the moisture susceptibility criteria

Table 6: Parameters measured for TSR test

Mould No.	Binder content by wt. of mix (%)	Wt in air (g)	Wt in water (g)	SSD, (g)	Bulk vol. (cc)	Bulk density (g/cc)	Dimension T (mm)	Dimension Dia (mm)	Gmm	%Va
1	4.56	1249.1	748.5	1254.3	505.8	2.470	64.20	102.10	2.597	4.91
2		1267.2	761.3	1269.6	508.3	2.493	63.20	102.10	2.597	4.01
3		1244.2	747.3	1246.2	498.9	2.494	63.20	102.10	2.597	3.98
4		1224.8	742.5	1227.2	484.7	2.527	62.10	102.10	2.597	2.71
5		1256.8	760.0	1259.3	499.3	2.517	63.30	102.10	2.597	3.08

Conditioned						Dry	
Va, cc	**SSD, g after partial vacuum**	**Vaw, cc**	**Sr, %**	**Ultimate load, N**	**Tensil strength, Kpa, S1**	**Ultimate load, N**	**Tensil strength, Kpa, S2**
24.9						934	90.7583
20.4	1283.00	15.80	77.5	986	97.3272		
19.8	1257.00	12.80	64.5	1070	105.619		
13.1						1236	124.166
15.4						985	97.0749

Conditioned sample average tensile strength, S1 =	101.47
Dry sample average tensile strength, S1 =	104
TSR =	**0.976**

ACKNOWLEDGEMENT

We would like to show our gratitude to the Director, Tamilnadu Highway Research Station, Guindy for granting permission to publish this paper. We express our sincere thanks to Aarupadai Veedu Institute of Technology, for providing the needed Facilities and creating a comfortable atmosphere required for this project.We convey our soulful thanks to SAIF, IIT Madras for provision of expertise and technical support in assessing of samples. We express our sincere thanks to Anna University, Chennai for their technical support in assessing of samples. We are grateful to Sterlite Industries, Tuticorin for their logistical and financial support and for providing necessary guidance concerning projects implementation.

CONCLUSION

Addition of copper slag of (15%) in aggregate of DBM grade II mix improves good interlocking and eventually improved the volumetric properties as well as the mechanical properties of the mix. Because of the improved property by the incorporation of copper slag, it can be used in bituminous mixes as a partial replacement in aggregates, which normally used in the conventional bituminous mixes. A field study may be undertaken at different climatic/traffic conditions on national/state highways.

REFERENCES

1. J. J. Emery, Slag Utilization in Pavement Construction, Extending Aggregate Resources, ASTM STP, 774, 95 (1982).
2. R. H. Miller and R. J. Collins, Waste Materials as Potential Replacements for Highway Aggregates, Report 166 (NCHRP, TRB, Washington DC) (1976).
3. S. Bose, M. C. Harit, C. Kamaraj and M. Singh, Development of Mix Design Procedures for Bituminous Macadam Mixes for Roads and Highways, Highway Research Bulletin, Indian Roads Congress, **65**, Dec 2001, Special Research Presentation at Annual Session of IRC, Kochi, Jan 2002.
4. P. B. Queneau, L. D. May and D. E. Cregar, Application of Slag Technology to Recycling of Solid Wastes, Incineration Conference, Knoxwille, TN, May (1991).

Accepted : 04.05.2016