Section A- Research Paper



FLEXURAL BEHAVIOR AND MICROSTRUCTURAL CHANGES IN GEOPOLYMER STABILIZED AGGREGATES BASES WITH COPPER-SLAG AS FINE AGGREGATES

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Abstract:

Scarcity of natural materials and sustainable solutions have become major concerns of civil-engineers. As a part of it, the pavement engineers started using low-carbon emission binder materials such as geopolymer binders and recycled materials in different layers of flexible and rigid pavements. The present article focused on flexural behavior of geopolymer stabilized aggregate bases by replacing natural fine aggregates with copper-slag at a replacement dosage of 0, 5, 10, 15, 20 and 25% by total weight of dry aggregates and different dosages of geopolymer binder (3, 4 and 5% by weight of total dry aggregates). A set of prismatic specimens were prepared and subjected to both static and repeated four-point bending for the determination of flexural strength and indirect tensile fatigue test was conducted to determine the fatigue life of geopolymer stabilized mixes at different stress ratios (0.6, 0.7 and 0.9). Further, the changes in microstructure with respect to type of geopolymer binder are investigated through scanning electron microscopy (SEM) at constant dosage of 5%. The study revealed an insignificant reduction in flexural strength with a maximum replacement dosage (20%) of natural aggregates with copper-slag. Further the results revealed a significant influence of dosage of geopolymer binder on flexural strength and fatigue behavior of geopolymer stabilized aggregate mixes.

Keywords: geopolymer stabilization, copper-slag, base and subbase layers, flexure, microstructure.

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DOI: - 10.31838/ecb/2023.12.si5.075

Background:

In recent decades, researchers have engaged on conservation of natural resources to promote sustainable solutions across various fields of civil engineering [*Rafi et al. 2010, Wu et al. 2013, Nwakaire et al. 2020, Goli et al. 2022, Masi et al. 2022*].

For the development of sustainable infrastructure, the use of geopolymer binders in place of portland cement as well as recycled materials such as recycled concrete aggregates, copper slag, reclaimed asphalt pavement materials, and steel slag in place of natural aggregates are intended in the research. Copper-slag (CoS) is a non-ferrous residue obtained from copper-smelting process. CoS is mainly composed of fayalite (2FeO.SiO₂) and magnetite (Fe₃O₄) [*Wang et al. 2013*].

Few researchers tried finely ground CoS as supplementary cementitious material (SCM) [Zain et al. 2004; Moura et al. 2007; Shi et al. 2008; Zhang et al. 2022] and reported an insignificant influence on working conditions but showed considerable impact on mechanical properties [Najimi et al. 2011; Edwin et al. 2016; Onuaguluchi. 2012 (PhD thesis)]. Further, the replacement of fine aggregates with fine CoS showed an increase in workability and enhanced mechanical properties in cementitious based materials. The present study is mainly focused on replacing the fine aggregate mixes for rural roads.

It is now possible to stabilise pavement layers like the base and subbase layers in both flexible and rigid pavements thanks to research on geopolymer binders. According to a study on the mechanical characterisation of flyash, slag, and flyash plus slag based aggregate mixes with recycled materials, these materials have acceptable strengths for a minimum dosage of 4% by weight of dry aggregates [Mohammadinia et al. 2016]. The utilization of rice-husk ash (RHA) in geopolymer stabilized aggregate bases resulted in reduction in strength and recommended a maximum of 50% replacement of flyash with RHA for the construction of pavement bases for high traffic roads [Poltue et al. 2019]. Arulrajah et al. 2016 studied the mechanical characteristics of waste materials from construction and demolition stabilised with calcium carbide residue (CCR) and slag based geopolymer and reported an optimum dosage of 5% slag plus CCR. While flyash activated with NaOH solution was advised to achieve a minimum unconfined compressive strength of 4.5MPa, flyash alone stabilised with

reclaimed asphalt pavement material did not impart sufficient strength. [Avirneni et al. 2016]. Studies on the strength development of geopolymer stabilised mixes with recycled materials, such as recycled concrete aggregates, reclaimed asphalt pavement material, and construction and demolition material, have been extensively investigated, but there are only a very limited number of studies on the flexural strength and fatigue behaviour of CoS plus aggregate mixes with various types of geopolymer binders made with flyash and slag. In order to assess the flexural strength and fatigue behaviour of CoS plus aggregate mixes stabilised using different geopolymer binders, this article focused on the flexural strength and fatigue behaviour of these mixes.

Materials and methods:

Two stocks of natural aggregates with a nominal maximum aggregate size (NMAS) of 25mm and 9.5mm and copper-slag with a NMAS of 4.75mm were mixed with different geopolymer binders (composed of flyash and/or ground granulated blast furnace slag (GGBS)) with a liquid-to-powder ratio of 0.4. In the earlier research article, the preparation of copper-slag plus aggregate mixes with different types of geopolymer binders such as Flyash based geopolymer (FG), GGBS based geopolymer (FSG) was presented.

The present article is mainly focused on flexural strength and fatigue resistance of the specimens prepared with these mixes. The flexural strength of the prismatic specimens was determined by using a series of 4-point bending tests conducted as per IS-516 (PART-IV)-2018. A set of 100mm × 100mm × 500mm with a span-to-depth ratio of 4 are prepared and flexural strength was determined. The other details of mix-proportioning were explained in the previous research article [*Reddy and Siddegowda. 2022*]. Further a set of cylindrical specimens prepared with FG and FSG-based geopolymers were subjected to repeated indirect tensile loading at a stress-ratio of 0.6, 0.7 and 0.9.

Finally, a regression analysis was carried out to predict the fatigue life of copper-slag incorporated aggregate mixes stabilized with geopolymer binders.

Results and discussions:

Flexural behavior of geopolymer stabilized CoS and aggregate mixes

A total of 3 series of mixes were prepared with three types of binders (FG, SG and FSG based geopolymers) and a set of 168 prismatic specimens were prepared with these mixes. The specimens were subjected to four-point bending and corresponding flexural strengths were evaluated. In case of FG-based mixes, a maximum flexural strength of 2.71MPa was observed at 5% dosage for mixes without CoS. While a reduction in strength was observed with increase CoS dosage and decrease in binder dosage. A maximum reduction of 41% was observed in FG3 mixes with 25% increase in CoS dosage whereas the reduction in strength was observed to be 24% for FG5 mixes. So, the higher dosage of geopolymer binder resulted in a lower reduction in strength as compared to lower binder dosage. A same trend was observed in case of mixes stabilized with FSG based geopolymer binders. But, in case of mixes stabilized with SG based geopolymer binders, an approximate same reduction of strength was observed (around 40% reduction) at lower and higher dosages. The results showed a maximum flexural strength of 2.91MPa, 2.49MPa and 2.71MPa for the mixes without CoS and with FG, SG and FSG based geopolymer binders (at 5% dosage) respectively. These obtained strengths are comparatively higher than the strengths of geopolymer stabilized RAP (60%) mixes reinforced with geogrid [*Poltue et al. 2019*].

Further, the flexural strengths of the aggregate mixes (without CoS) stabilized with 4% FG-based binder (2.36MPa) and 4% SG-based binder (2.06MPa) are approximately equivalent to 4% cement treated aggregate mixes (2.2MPa) as presented in *Ismail et al. 2014*. Further, the flexural strengths of CoS plus aggregate mixes with SG-based geopolymer binder are approximately same and CoS mixes with FSG binder imparted higher strengths as compared to the average 28-day strength of cement treated aggregate mixes presented in [*Lv et al. 2019*].

The results also showed the flexural strengths of 25% CoS mixes are comparable to the flexural strengths of 25% RAP aggregate mixes stabilized with approximately 6% cement [*Khay et al. 2014*].



Figure 1. Flexural strength of geopolymer stabilized aggregate mixes with and without CoS

Further, a significant influence of binder type and dosage on flexural strength of CoS plus aggregate mixes was conformed from the figure 1. The distribution of flexural strengths for different binder types at different dosages for geopolymer stabilized bases are depicted through a whisker plot with 1st and 3rd quartile strengths (figure 2). From, this study, it can be recommended to replace the natural sand or crushed sand with CoS by 25% and 4% of FG/FSG based geopolymer binder.



Figure 2. Range of flexural strength for aggregate mixes stabilized at different geopolymer binder dosages

Flexural strength (f_{cr}) is analysed using multiple linear regression with binder type, binder dosage, and dosage of CoS as independent factors. In this instance, arbitrary values of 0, 1, and 2 are specified for the SG, FG, and FSG-based geopolymers, respectively.

The corresponding equation with an adjusted R^2 value of 0.92 is given as below

 $\begin{aligned} f_{cr} &= 0.3927 \times (binder \ type) \\ &+ 0.39267 \times (binder \ dosage) \\ &- 0.02724 \times (CoS - dosage) + 0.406 \end{aligned}$

Microstructural changes

The microstructural changes in geopolymer stabilized aggregate mixes (28 days cured samples) with 10% CoS and stabilized at 5% FG, SG and FSG based geopolymer are depicted through the micrographs obtained by scanning electron microscope (SEM).

The micrographs conformed an irregular shape for slag particles while an approximately rounded shape revealed the presence of flyash particles as observed in few past studies [*Zawrah et al. 2016, Wang et al. 2020, Nagajothi and Elavenil. 2021, Rao and Rao. 2015*].



(a) 10% CoS aggregate mixes stabilized with flyash based geopolymer binder

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(b) 10% CoS aggregate mixes stabilized with slag based geopolymer binder



(c) 10% CoS aggregate mixes stabilized with flyash + slag based geopolymer binder
Figure 3. The microstructural changes in CoS-aggregate mixes stabilized with FSG-based geopolymer binder (left side images: 10µm scale and right side images: 1µm scale)
Additionally, the voids in the micrographs had a stronger correlation with the flexural strengths.

The lower voids and higher voids for FSG based geopolymer stabilized mixes can be considered as the better way to describe the change in flexural strengths of mixes with SG and FSG based geopolymer stabilized mixes with 10% CoS as shown in figure 3. The micrographs conformed that "higher the strength lower will be the voids".

Fatigue behavior of geopolymer stabilized CoS and aggregate mixes

In the present study, the fatigue behavior of CoS plus aggregate mixes stabilized with FG and FSG based geopolymer binders is presented.

As the flexural strength of SG-based geopolymer stabilized mixes is low, these mixes were discarded from further study to reduce the experimental matrix. The repeated load on diametrically placed cylindrical samples is applied corresponding to a stress-ratio of 0.6, 0.7 and 0.9.

The corresponding results are presented in the figure 4 and figure 5 for FG and FSG based geopolymer binder stabilized mixes respectively.

Further, the dosages of 10% and 20% are not considered to reduce the experimental matrix of the study.



Figure 4. The relation between fatigue life and stress-ratio for FG-based geopolymer stabilized mixes

From figure 4, the results revealed lower fatigue for higher CoS dosages and higher stress ratios whereas the fatigue life (N_f) ranged from 700 cycles to 76000cycles for FG-mixes.

The fatigue life for FG based geopolymer mixes with 3% binder dosage was found to be in the range of (12000, 76000) while the corresponding range for FG5 mixes was found to be (4900, 800).

It means the increase in binder dosage showed adverse effect on fatigue and caused almost a

reduction in fatigue life by about 15 times at a stress ratio of both 0.6 and 0.9.

Whereas the reduction was found to be about 1.5 times for the mixes stabilized with 4% binder.

So, it is recommended to use a lower dosage of FG based geopolymer (4%) for overcoming the brittleness of material to enhance the life of the pavement.



Figure 5. The relation between fatigue life and stress-ratio for FSG-based geopolymer stabilized mixes

The fatigue life for FSG based geopolymer mixes with 3% binder dosage was found to be in the range of (13000, 77000) while the corresponding range for FG5 mixes was found to be (4200, 900). It means the increase in binder dosage showed a significant negative impact on fatigue and caused almost a reduction in fatigue life by about 18 times and 15times at a stress ratio of 0.6 and 0.9 respectively. Whereas the reduction was found to be about 1.6 times and 3 times for the mixes stabilized with 4% binder at a stress ratio of 0.6 and 0.9 respectively. So, it is recommended to limit the binder dosage of 4% for FG based and FSG based geopolymer for overcoming the brittleness of material to enhance the life of the pavement.

Conclusions:

This article mainly presented the flexural behavior of geopolymer stabilized aggregate mixes with partial replacement of fine aggregates with copperslag ranging from 0 to 25% by weight of total dry aggregates. Further, this study presented the micrographs of geopolymer stabilized aggregate mixes with different types of binders prepared with flyash and/or ground granulated blast furnace slag. The following conclusions were drawn from the

Eur. Chem. Bull. 2023, 12(Special Issue 5), 619-626

investigation of flexural and microstructural behavior.

- The dosage and type of geopolymer binder are significant factors influencing the flexural strength of geopolymer stabilized aggregate mixes copper slag as fine aggregate
- The replacement of fine aggregates with more than 20% of copper-slag leads to significant reduction in flexural strength (approximately 30%)
- The micrographs revealed higher voids for slag based geopolymer mixes which strengthens the reason for the lower flexural strengths
- The results of indirect tensile fatigue test confirmed a permissible dosage of 4% for both flyash based and flyash plus slag based geopolymer stabilized aggregate mixes where copper-slag replaced fine aggregate partially

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