

MICROSTRUCTURE STUDY OF GRASS ASH FOR REPLACEMENT OF CEMENT

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ABSTRACT

Cement is a versatile and relatively high cost material worldwide which is also the most important ingredient of concrete. Cement production is not only highly energy-intensive, next to steel and aluminium, but also consumes significant amount of natural resources. Concern about environmental protection from a global viewpoint has led the researchers to make utilize industrial or agricultural by products as supplementary cementitious material. Many researches are currently being conducted concerning the use of such products in construction and have proved that partial replacement of cement with mineral admixtures improves the performance of cement based materials in fresh and hardened state. In this research, development of a new building material due to its easy availability in abundance for use in the rural areas of the developing countries is experimentally investigated and the material is "Grass ash". Grass ash (GA), can be produced from grass using appropriate combustion technique and can be used as a supplementary cementitious material. This paper discuss about the production techniques and the quality of ash produced by corresponding technique, to consider the possibility of using grass ash as a pozzolana material. Three combustion techniques and one grinding method was used to investigate physical and chemical aspects of grass ash produced. Combustion technique, grass ash particle size, silica crystallization phase and chemical content of the produced GA were studied using X-ray diffraction (XRD), Energy-dispersive X-ray spectroscopy (EDX) and scanning electron microscopy (SEM). From this investigation, it was found that combustion period, cooling duration, and grinding process and duration are important parameters in obtaining GA of standard fineness and quality. Observations and analysis shows that the highest amounts of amorphous silica occur in open heap burning at 500 °C for 2 hours. According to ASTM requirements, GA can be classified as class C pozzolana. Therefore, grass ash can be favourable to be used as pozzolanic cement additive.

Keywords: Grass ash (GA), micro structure, SEM, XRD, EDX, pozzolana

I. INTRODUCTION

In the recent years, growing consciousness about global environment and increasing energy security has led to increasing demand for renewable energy resources and to diversify current methods of energy production. The use of various waste products in civil engineering construction has gained considerable attention in view of the shortage and high costs of suitable conventional aggregates, the increasing costs of waste disposal and environmental constraints. Due to current boom in construction industry, cement demand has escalated which is the main constituent in concrete. Also, the cement industry is one of the primary sources which release large amounts of major consumer of natural resources like aggregate and has high power and energy demand for its

operation. The production of cement emits huge amount of CO₂ into the atmosphere that contributes to the greenhouse effect and the global warming of the planet. Use of supplementary cementing materials has become an integral part of high strength and high performance concrete mix design. These can be natural materials, by-products or industrial wastes, or the one requiring less energy and time to produce. Some of the commonly used supplementary cementing materials are fly ash, Silica Fume, Ground Granulated Blast Furnace Slag and Rice Husk Ash etc. Selection of the suitable material is important for the overall economy and safety. Initially use of locally available material was in general practice. But now-a-days due to heavy construction it is required to transport superior material from other place too. Use of waste materials coming from the industry and agricultural works is environmentally friendly and economical. Best way for the management of the solid waste is to reuse it in different application without changing the form of the waste. In civil engineering construction there is potential to use such waste in bulk. This calls for the use of sustainable binders so as to reduce the quantity of cement used and one of such promising material can be grass ash. On an average, burning of grass produces 8–10% of ash by the weight of grass burnt and its composition can be highly variable depending on type of grass and method of burning. The most prevailing method for disposal of the ash is land filling and rest being either used as soil supplements. The characteristics of the ash depend upon biomass characteristics, combustion technology and the location where ash is collected. The use of pozzolanas as alternatives for the commonly used Portland cement have been introduced in the last few decades either for cost reduction, performance, durability or environmental reasons. Grass can be burnt into ash that fulfils the physical characteristics and chemical composition of mineral admixtures. Pozzolanic activity of grass ash depends on (i) silica content, (ii) silica crystallization phase, and (iii) size and surface area of ash particles. In addition, ash must contain only a small amount of carbon. Suitable incinerator/furnace as well as grinding method is required for burning and grinding grass in order to obtain good quality ash. In this research, three methods of grass combustion were used based on combustion temperatures and burning periods. The produced ash was ground to ensure that it meets the requirements of BS 3892 standard. As grass ash primarily consists of fine particulate which can easily get air borne by winds, it is a potential hazard as it may cause respiratory health problems to the dwellers near the dump site or can cause groundwater contamination by leaching toxic elements in the water. As the disposal cost of the ashes is rising and volume of ashes is increasing, a sustainable ash management which integrate the ash within the natural cycles needs to be employed. So, utilization of such by products and agricultural waste ashes solves a twofold problem of their disposal as well providing a viable alternative for cement substitutes in concrete. Hence, incorporating the usage of grass ash as replacement for cement in blended cement is beneficial for the environmental point of view as well as producing low cost construction entity thus leading to a sustainable relationship. The aim of this study was to study the microstructure of grass ash burnt at three different temperatures and durations.

II. PREPARATION OF GRASS ASH

Grass ash is a potential material, which is amenable for value addition. To get a good quality of grass ash, the technologies of ash production vary from open-heap burning to specially designed incinerators like muffle furnace. Open heap burning of grass produces large quantity of ash, but is banned in many countries due to pollution problems. Uncontrolled burning results in a structure of highly crystalline form that is of low

reactivity. Further, un-controlled, or open field burning results in high carbon content, which will adversely affect the performance of ash. The incinerating conditions (temperature and duration of incineration) significantly alter the final quality of the Grass ash. The grass used in this research was obtained from school ground in Ferozepur. This grass belongs to *Cynodon dactylon* species. The natural color of leaves was green. The grass was in a damp and fresh condition. Firstly, the grass was spread on the concrete floor and directly exposed to sunshine for 1 to 2 weeks. Then, only dried grass was collected and burnt within 3 to 5 days. Since grass is self-burning, no extra fuel was required except at the igniting stage of burning. Three different temperatures and durations were selected for this research; open heap burning for 3 hours, 500⁰C for 2 hours and 1000⁰ C for 4 hours, and named as method A, B and C respectively and in all three methods the ash was cooled in natural environment. The burning of grass yields approximately 9% of ash which can easily be pulverized to powdered form. The grinding of grass ash was carried out using the Los Angeles machine. The pozzolanic reactivity of material is highly depended on the silica form. It is found that high Loss of ignition (LOI) is caused by high moisture content and ash becomes darker in that case. Colour of ash observed in methods A, B, and C was found to be progressively lighter which was consistent with the decreasing value of LOI.

Grass must be burnt at the correct temperature and duration to achieve the requisite pozzolanic activity and significant impact on the fineness of GA particles produced. The color of the ash obtained at different temperatures is also different, this can be due to the different combustion of the particles of grass at different temperatures. Color changes are associated with the completeness of combustion process as well as structural transformations of silica in the ash. Ash of black color obtained from open heap burning is an indication of complete oxidation of the carbon, which is also an indication of availability of large portion of amorphous silica in the ash (Fig1). At high temperature (500⁰C) strong interaction between potassium and silica ion cause the formation of potassium polysilicate combined with carbon resulting in grey color ash (Fig2). At higher temperature (1000⁰C) with prolonged burning result in ash with light pink color representing silica of crystalline form (Fig3). The above three color distinctly stand for amorphous, transition and crystalline states of ash formation. About 5.0 kg of ash was placed into the Los Angeles machine for each grinding. Researchers [1,2,3] agree that finer pozzolanic ash is better. Fineness of ash is important because it influences the rate of reaction and the rate of gain of strength. According to the standard [4], particles retained on 45-micron sieve should not be more than 12.0%. Therefore, to get the required fineness, proper grinding of burnt ash is very important.

III. MICROSTRUCTURE

Microstructure is defined as the structure of a prepared surface of a material as revealed by a microscope above 25× magnification. Micro structural analysis generates high resolution images and precisely measures very small features and objects allowing sub-micron-scale features to be seen. More sophisticated microstructure examination involves higher powered instruments like optical microscopy, Scanning Electron Microscopy (SEM), Transmission Electron Microscopy (TEM) and X-Ray Diffraction (XRD).

3.1 Scanning Electron Microscopy (SEM)

Scanning electron microscopy also known as SEM analysis is used very effectively in microanalysis and failure analysis of solid inorganic materials. It uses a focused beam of high-energy electrons to generate a variety of signals at the surface of solid specimens. In most SEM microscopy applications, data is collected over a selected

area of the surface of the sample and a two-dimensional image is generated that displays spatial variations in properties including chemical characterization, texture and orientation of materials. Both the surface and internal structure of grass ash particles were analyzed using the SEM equipped with back scattered and secondary electron detectors. It provided detailed imaging information about the morphology and surface texture of individual particles of ash.

Fig 4,5,6 shows the micrograph of the grass ash prepared at the different temperatures. It is clear from the figure that the structure of the ash samples prepared at different temperatures is different because of burning temperatures and durations. The morphology of a grass ash particle is controlled by combustion temperature, burning time and cooling rate. Hollow cenospheres and irregularly shaped unburned carbon particles tended to be in the upper end of the size distribution. Agglomerated particles and irregularly shaped amorphous particles are present due to inter-particle contact or rapid cooling. The phenomenon of breaking down cellular structure and thus resulting reduction of fine pores is the main factor in increasing strength and durability. But after grinding it for 30 minutes, the cellular structure disappeared and became much smaller particles. The large surface area of grass ash was caused by the coarse micro-structural shape like cubes of cellular beehives. The cellular shape creates water absorption problem in a damp surrounding. This problem could be overcome by breaking down cellular structure and thus reducing the ability to absorb water. That is why grinding of burnt husk for 60 min or more is recommended.

3.2 X-ray Diffraction (XRD) Analysis

XRD analyses were performed for samples to identify differences in the formation of amorphous or crystalline silica for different combustion times, temperatures and cooling regime. A qualitative assessment of the crystallinity of the samples can be obtained from the intensity of the narrow reflections as compared to the broad band around 22° (2θ). Fig 7,8,9 shows the XRD pattern of ash samples. The intense broad peak observed for the grass ash produced at 500°C for 2 hours indicates the amorphous nature of silica. Similar results were obtained for the ash obtained from open heap burning showing hardly any crystalline reflections. For the ash obtained at 1000°C for 4 hours show reasonably sharp and intense reflections start to show up on top of the broad amorphous background, evidencing that at these temperatures crystalline cristobalite starts to form. The presence of amorphous silica makes it fit as cement replacing material due to pozzolanic activity.

This is in line with the researchers who agree that silica does not become crystalline if combustion temperature is below 700°C . At 1000°C temperature, silica begins to change into crystalline phase and no more amorphous structure is available. At this stage, formation of crystalline cristobalite starts. If temperature further increases, tridymite is produced. However, GA produced at the muffle furnace did not show major crystallization form and contained mostly amorphous silica.

3.3 Energy-dispersive X-ray spectroscopy (EDX)

The chemical properties of grass ash have been analyzed using EDX technique. The chemical composition of ash is found to vary from one sample to another due to the burning temperatures, methods and durations. Silica content is maximum mineral in ash obtained from the open heap burning but the amount of carbon dioxide is also more as compared to the ash obtained from the muffle furnace. This can be because of uncontrolled temperature and duration in open burning. The chemical properties of the grass ash obtained from different temperatures are discussed in Table 1.

Table 1 shows the chemical analysis results of GA obtained using combustion methods A, B, and C. It can be observed that the content of SiO₂ of GA produced in this study was between 47% and 59%. Alkaline substances like KO₂ were main foreign particles in GA having content between 1% and 7%, it depends on types and amount of fertilizers used during growing period of the grass. Loss on ignition (LOI) progressively decreased for combustion methods A, B, and C. LOI of grass ash obtained using combustion method C was the lowest. Ash with low carbon content increases pozzolanic activity and water requirement in concrete is usually less for ash with low LOI. It can be observed that method B produced the best quality ash among the other methods followed. Thus, the quality of ash produced by burning of grass in the muffle furnace was good enough to use in concrete as a supplementary cementitious material.

IV. FIGURES AND TABLES



Figure 1 Ash obtained from Open Burning (A)



Figure 2 Ash obtained from burning at 500°C (B).



Figure 3 Ash obtained from burning at 1000°C (C).

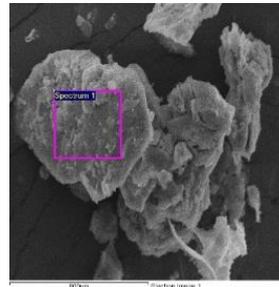


Figure 4 SEM micrograph of grass ash prepared at open burning.

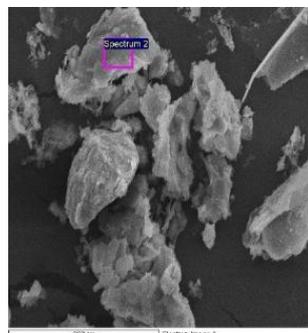


Figure 5 SEM micrograph of grass ash prepared at 500°C.

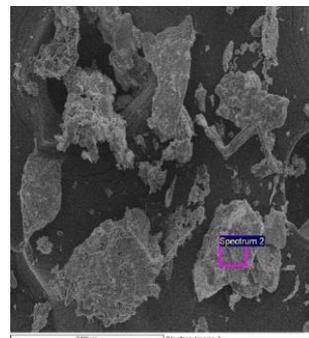


Figure 6 SEM micrograph of grass ash prepared at 1000°C.

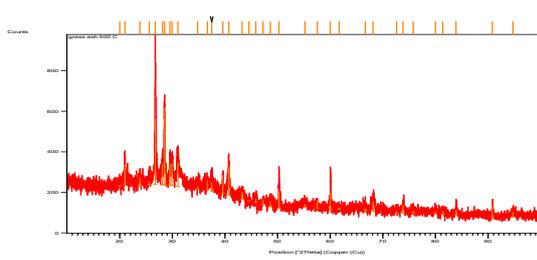


Figure 7 XRD pattern of ash sample collected at 500⁰C.

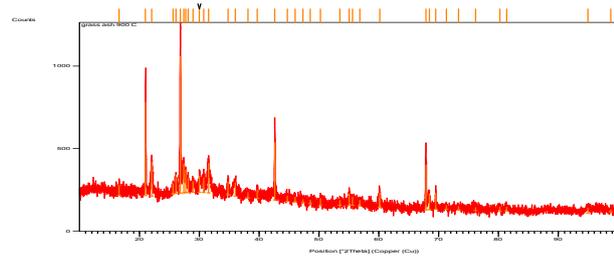


Figure 8 XRD pattern of ash sample collected at 1000⁰C.

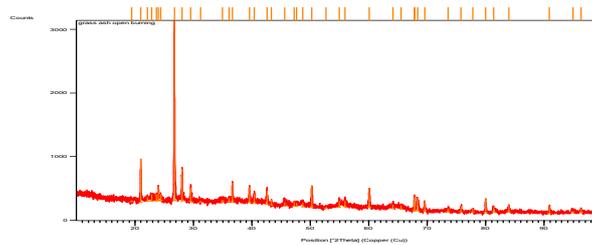


Figure 9 XRD pattern of ash sample collected in Open Heap Burning.

Table 1 Chemical property of Grass Ash at Different Temperatures

Chemical Constituents	Open Burning (A)	Burning at 500 ⁰ C (B)	Burning at 1000 ⁰ C (C)
Silica Dioxide (SiO ₂)	59.29	38.38	47.29
Alumina Oxide (Al ₂ O ₃)	4.71	16.07	12.48
Ferric Oxide (FeO)	1.10	1.91	2.49
Calcium Oxide (CaO)	0.95	4.13	5.63
Magnesium Oxide (MgO)	1.33	1.42	4.72
Potassium Oxide (KO ₂)	1.45	4.48	7.50
Sodium Oxide (Na ₂ O)	-	4.89	2.47
Carbon Dioxide (CO ₂)	30.81	28.72	16.74
Phosphorous Pentoxide (P ₂ O ₅)	0.36	-	0.68

V. CONCLUSION

This investigation aimed to achieve best ash from grass so that it can be effectively used as a replacement for cement in the construction industry using a simple and cost-effective method. From the SEM images, it is inferred that the particle size of ash when sintered in muffle furnace has got reduced as compared to open heap burning. The chemical composition results are evident for the increase in silica content of the sintered GA at room temperature when compared with GA at muffle furnace. Also, XRD data showed that the ash obtained from 500⁰C contains amorphous silica making it fit as cement replacing material due to its high pozzolanic activity. It is hence proposed that ash sintered at 500⁰C for 2 hours can be used as a supplementary cementitious material to improve the performance and strength of cement. Thus, use of grass ash will help to transform it from

an environmental concern to a useful resource for the production of a highly effective alternative cementing material.

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