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14817 their development and applications





UNITED NATIONS INDUSTRIAL DEVELOPMENT ORGANIZATION

RICE-HUSK ASH CEMENTS: THEIR DEVELOPMENT AND APPLICATIONS

CORRIGENDUM

Page I

For the existing text substitute:

FOREWORD

Cement is a basic commodity essential to the infrastructural, industrial and social development of all countries. It is also a product for which many developing countries have to rely largely on imports. Even in developing countries that produce standard Portland cement domestically, its high cost and its importance as an intermediate product have an adverse impact on the economics of user industries.

In the past 20 years or so, and particularly in the 1970s, significant advances have been made in the use of rice-husk ash (RHA) in the production of cement. Its potential economic advantages for developing countries that have large quantities of rice husks readily available, virtually free of cost, are enormous.

Since 1978, the Economic and Social Commission for Asia and the Pacific (ESCAP) in Bangkok and an ESCAP institution, the Regional Centre for Technology Transfer (RCTT) in Bangalore, with the co-operation of Governments, particularly in the South and South-East Asian regions, have undertaken to promote and co-ordinate the technical development of cements based on RHA.

ESCAP and RCTT, in collaboration with UNIDO, arranged a series of workshops that led, *inter alia*, to the promotion of further research and development, the setting up of demonstration pilot plants, the assessment of marketing characteristics and the establishment of an information transfer network.

Given all the advances that have taken place in this field, UNIDO decided to commission a recognized world expert on RHA cement to draw together a comprehensive state-of-the-art assessment of all known production and utilization factors associated with the product's development over recent years. The present publication is intended as a guide to the complex variety of technical and economic choices facing an individual country, district or organization when considering a strategy for the production and utilization of RHA cement. UNIDO is prepared to provide technical assistance to countries wishing to develop this branch of their building materials industry. As a further support to developing countries UNIDO, ESCAP and RCTT are considering the possibility of preparing simple instruction manuals on the practical aspects of establishing and operating RHA cement plants.

Finally, grateful acknowledgement is made to the Australian Government, which provided the funds for preparing this document, and to ESCAP and RCTT for their efforts in supporting the project.

Abd-El Rahman Khane Executive Director



UNITED NATIONS INDUSTRIAL DEVELOPMENT ORGANIZATION Vienna

RICE-HUSK ASH CEMENTS:

their development and applications

Prepared in co-operation with the Government of Australia

FOREWORD

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Abd-El Rhaman Khane Executive Director

PREFACE

In its efforts to promote and accelerate developing country industrialization, UNIDO pays particular attention to the utilization of locally available raw materials. In this context, agricultural wastes such as rice husks play an increasingly important role due to their widespread availability and their potential for sustaining decentralized industrialization integrated with overall rural development.

Lack of information about available technologies is, however, still hampering progress. The need to disseminate recent know-how and technology in the field of rice-husk ash (RHA) utilization has been stressed at many meetings, most recently at the Workshop on Selected Building Materials held at Sydney from 19 to 30 April 1982 as a joint effort of the Australian Government and UNIDO.

It is in response to these recommendations that UNIDO is publishing the current monograph prepared by Dr David Cook, Associate Professor of Civil Engineering, University of New South Wales, Australia. This has been made possible with the help of a generous contribution from the Government of Australia to the United Nations Industrial Development Fund. Dr Cook and UNIDO also wish to acknowledge the co-operation and assistance provided for the project by officials of the ESCAP Regional Centre for Technology Transfer (RCTT) in Bangalore, India, the ESCAP/UNIDO Division of Industry, Human Settlements and Technology in Bangkok, Thailand, and the Cement Research Institute of India (CRI).

The monograph is a state-of-the-art assessment of cements based on RHA. It covers the basic research and development through to small-scale village production including quality control and specification. Its intention is to provide information that will permit evaluation of RHA cements in context of the range of natural and artificial pozzolans currently being used to manufacture cementitious materials.

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EXPLANATORY NOTES

References to dollars (\$) are to United States dollars.

The use of the hyphen between dates (e.g. 1960-64) indicates the full period involved, including the beginning and end years.

The following forms have been used in tables:

Three dots (. . .) indicate that data are not available or are not separately reported.

A blank indicates that the item is not applicable.

The following abbreviations are used:

CSH calcium silicate hydrate

RH relative humidity

RHA rice-husk ash

RHAM rice-husk ash mortar

Organizations

- ASTM American Society for Testing and Materials
- CBRI Central Building Research Institute, India

CRI Cement Research Institute of India

ISO International Organization for Standardization

KVIC Khadi and Village Industries Commission

PCSIR Pakistan Council of Scientific and Industrial Research

SIRIM Standards and Industrial Research Institute of Malaysia

I. BACKGROUND

Introduction

In 1978, the Food and Agricultural Organization of the United Nations (FAO), published a paper entitled 'Rice Husk Conversion to Energy' (1) the foreword to which stated that the focal point of the study was 'the pressing need to utilize rice husk as an energy source, particularly in developing countries.' The agencies involved with the preparation of this document are of the view that, where appropriate, the energy value of the husk should also be utilized in the development of cements based on the rice-husk residue or ash after combustion.

Indeed, it would be ideal to have a vertically integrated system of husk utilization for energy where the ash residue was used to produce such a valued material as cement. However, the pyroprocessing of husks to produce an ash with optimum properties for cement production is not entirely compatible with general practices of energy extraction. For cement production, the main component of the ash, silica, must remain in an amorphous form and the conditions for optimum combustion are controlled by both temperature and time. In contrast, where the husks are used as a source of energy, the combustion temperature is generally quite variable — as is the time of exposure of the husk to the combustion temperature. Only the Mehta-Pitt process (2) combines efficient extraction of heat from the husks with the production of an ash containing amorphous silica but at this stage the process is not considered appropriate for developing countries.

As a result, the development of RHA as pozzolanic material has not been directly linked with parallel developments in heat recovery and utilization. Instead, pyroprocessing has essentially concentrated on producing ash with maximum reactivity. However, it is pertinent to note that much of the current research with cements based on RHA is directed toward better coupling of the heat extraction and ash reactivity aspects of husk disposal, utilizing the heat for production of process steam.

This report is a state-of-the-art assessment of cements based on RHA covering basic research and development through to small scale village production including quality control and specification. It is intended that the information provided will permit evaluation of RHA cements in the context of the complete range of natural and artificial pozzolans currently being used in the manufacture of cementitious materials.

Potential and advantages

The estimate for 1982 production of paddy on a global basis was 406.6 million tonnes (3) which represents about 81 million tonnes of husk or 16 million tonnes of ash if an ash content of 20 per cent is assumed. The availability of the ash, however, varies from country to country depending on husk utilization practices but probably does not exceed 30 per cent of the total. Hence after subtracting rice production in developed countries, the available ash for cement production is approximately 4.5 million tonnes.

Utilization of this ash to supplement cement production in developing countries is particularly attractive for a number of reasons:

(a) The capital cost per tonne of production capacity is substantially lower than for Portland cement production. In addition, Cook (4) has estimated that the cost per tonne to produce lime-RHA cement is about one-third that of Portland cement.

(b) As husks are not readily transportable, cement production will take place in rural areas, which are the areas most deprived of cement.

(c) The capacity of the plants ranges from 2 to 5 tonnes per day, a size compatible with village level industry, which in turn has the socio-economic advantages connected with industrial decentralization.

(d) In many situations, utilization of the ash for cement will assist in reducing pollution, since after combustion the ash may have been simply dumped.

(e) It is recognized that cement must be counted among the basic commodities on which development programmes rely, with an importance comparable to water, energy and fertilizers. Consequently, any efforts that assist in creating national or even regional self-sufficiency in cement production are always given high priority in development planning.

The technical advances

The FAO report mentioned above cites two 1924 German patents as the forerunners in describing the use of RHA in concrete. Later work in the United States of America by McDaniel (5) in 1946, Hough (6) in 1953 and Hough and Barr (7) in 1956 describe the manufacture and behaviour of blocks made from mixtures of Portland cement, ash and rice husks. Hough and Barr also investigated incorporation of lime in the mix and adobe bricks made from ash, soil and emulsified bitumen. The ash was obtained from furnaces where husks had been used as fuel or burnt simply as a means of disposal. The strengths of the blocks ranged from 0.5 to about 4 MPa and subsequent experience indicated they were quite suitable for house construction. Other properties such as resistance to erosion and thermal conductivity were also investigated. This latter property was also the subject of studies carried out in Pakistan by Ahsanullah and others (8, 9, 10) between 1950 and 1960. Blocks were made from Portland cement and RHA with cement:ash ratios varying from 5:1 to 1:20. As with the previous work, the ash was obtained from furnaces using rice husks as fuel.

Up to 1972, research reported in the literature showed continued attempts to utilize ash derived from relatively uncontrolled combustion. While block making was still the main objective, the ash was also used as a source of silica in autoclaved lime-silica bricks (11) and in production of refractory bricks (12). In 1972 Mehta, in the United States, published the first of several papers dealing with rice husk utilization (13, 14, 15).

Mehta's work is significant because it was the first careful study of the pyroprocessing parameters and their influence on ash reactivity. A fluidized bed furnace was designed (2) as a system utilizing rice husks to produce energy while also producing amorphous, and hence reactive, ash. A pilot plant was constructed in Sacramento, California and a commercial plant in Stuttgart, Arkansas. Mehta also patented applications for the ash as a reinforcing filler for rubber and as a highly reactive pozzolan when mixed with lime or substituted for Portland cement (16). The latter is sold at a premium as an acid resisting cement.

The concept of a cement based on RHA provided the impetus for renewed research, particularly in South-East Asia. In 1978, the ESCAP Regional Centre for Technology Transfer (RCTT) was given the responsibility for promoting and co-ordinating technical development of cements based on RHA. Accordingly ESCAP-RCTT in association with UNIDO and the Pakistan Council of Scientific and Industrial Research (PCSIR) organized the first workshop on RHA cements in Peshawar, Pakistan in January 1979. Some 15 papers were presented outlining the current state of the art (17). It was apparent that unreported work had been in progress in India, Pakistan and Malaysia for some years. Details of three methods for producing cement appropriate to developing countries were presented but only two had progressed to the pilot plant stage. The first involved controlled burning of the husks in incinerators and then intergrinding the ash with lime. The second simply ball-milled the ash after the husks had been used as a fuel source.

To assist further regional development subsequent to Peshawar, groups were established to: (a) Promote further research and development at laboratory and pilot plant level on rice husks and other agricultural residues;

- (b) Develop pilot plants as demonstration units;
- (c) Assess the marketing characteristics of cements based on RHA;
- (d) Develop an information transfer network.

A further UNIDO-ESCAP-RCTT meeting (18), held in Alor Setar in Kedah, Malaysia in October 1979, was prompted by the significant international interest in the proceedings of the Peshawar meeting and requests for assistance from most rice-growing developing countries. The main outcome of the meeting was the adoption of the Indian standard for masonry cement, IS 4098, for masonry cements based on RHA. It was considered that the main use for RHA cements would be for mortar and plaster applications; the availability of the standard would assist the marketing of RHA masonry (RHAM) cement.

A third ESCAP-RCTT meeting was held in New Delhi in November, 1981 (19). On reviewing the 14 technical papers presented, it was apparent that no significant technical development had occurred in the intervening period. It was also evident that the path for future work lay not so much in the area of basic technology but more with plant design and operation. Small plants with capacities up to 4 t/d were in operation in India, Nepal and Malaysia. Marketing problems indentified included the cost of transporting the husks and the impact of government controls on cement. The meeting concluded by indicating that the available technology on RHA cements was sufficiently proven and that documentation should proceed so that this information became more widely available.

II. THE RAW MATERIAL - RICE HUSKS

Structure, composition and properties

The rice husk is the woody sheath surrounding the kernel or grain and consists of two interlocking halves. Unlike most other cereals, the rice grain must be physically removed from the husk after harvesting. This is done by hand threshing or during milling. Hence the husk is a by-product of the process of obtaining grain.

A typical composition of the husk is shown in table 1. It can be seen that the proportions of crude fibre, lignin and ash are high and as a result the nutritional value of the husks (for animal feed) is low.

Houston (20) reports ash content ranging from 13.2 to 29 per cent but 20 per cent is generally considered a realistic average. As can be seen in table 2, this ash content is considerably higher than that of other agricultural residues, and is a reason for the need to develop a use for it. In the typical ash analysis shown in table 3, the predominant constituent is silica, which varies from 87 to 97 per cent. The next major constituent is alkali (sodium and potassium) while the presence and amount of constituents such as phosphorus, iron and magnesium depend primarily on fertilizing practice. According to Lanning, Ponnaiya and Crumpton (21), the silica enters the rice plant through its roots in a soluble form, probably as a silicate or monosilicic acid and then moves to the outer surfaces of the plant where it becomes concentrated by evaporation and polymerization to form a cellulose-silica membrane.

It should be noted that silica has been shown to be an essential soil constituent in the normal growth of rice. Low availability of silica increases the susceptibility of rice to diseases and insects. Field tests indicate that application of suitable silicon compounds to the soil greatly diminishes the appearance of blast and brown spot and increases the yield. Thus the usefulness of the traditional practice of spreading RHA on the paddy fields in Bangladesh and other developing countries is verified by scientific evidence.

The concentration of silica in the outer surface of the husks results in a rather abrasive material which is accentuated when the husks are crushed or broken. The silica content also accounts for the hardness of the husks which is similar on the Mohs scale to opaline silica $(5\frac{1}{2} \text{ to } 6\frac{1}{2})$.

The unit mass of the husks ranges from 96 to 160 kg/m³ while the bulk density is approximately 740 kg/m³. The extremely low unit mass acts against transporting the husks any distance; hence utilization should ideally be undertaken as close to the rice mill as possible.

The fuel value of husks is quoted by Houston to range from 13.8 to 15 MJ/kg. By way of comparison, oven dry timber averages 18.8 MJ/kg while typical values for coal and fuel oil are 29.7 and 39.8 MJ/kg respectively. Hence in fuel value terms, one tonne of rice husks is equivalent to 0.48 tonnes of coal or 0.36 tonnes of fuel oil.

Due to its cup-like form when removed from the grain, bulk husk has relatively good insulating properties. Depending on the degree of consolidation, its thermal conductivity ranges from 0.036 to 0.086 W-m⁻¹·K⁻¹. This compares with 0.041 W-m⁻¹·K⁻¹ for shredded asbestos, 0.030 for mineral wool and 0.028 for granulated cork.

TABLE 1

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TYPICAL COMPOSITION OF RICE HUSKS

Component	Weight (%)
Crude fibre	40.8
Cellulose	39.0
Lignin	34.0
Nitrogen-free extract	31.7
Ash	21.1
Pozzolans	19.5
Water	6.8
Crude protein	4.5
Crude fat	1.7

TABLE 2

ASH AND SILICA CONTENT OF PLANTS

Plant	Part of plant	Ash (%)	Silica (%)
Rice husk		22.1	93.0
Wheat	Leaf sheath	10.5	90.5
Sorghum	Leaf sheath epidermis	12.5	88.7
Rice straw		14.6	82.0
Breadfruit tree	Stem	8.6	81.8
Bagasse		14.7	73.0
Согл	Leaf blade	12.1	64.3
Bamboo	Nodes (inner portion)	1.5	57.4
Sunflower	Leaf and stem	11.5	25.3
Lantana	Leaf and stem	11.2	23.3

TABLE 3

Constituent	Weight (%)
SiO ₂	93.1
K ₂ O	2.3
MgO	0.5
Al_2O_3	0.4
CaO	0.4
Fe_2O_3	0.2
Na ₂ O	0.1

TYPICAL CHEMICAL ANALYSIS OF RICE-HUSK ASH*

* Ignition loss, 2.77%; density 2290 kg/m³.

Husk content of paddy

As Houston (20) notes, husk is generally considered to comprise one-fifth of the weight of the paddy, although the actual amount can vary. Differences result in part due to the particular rice strain but are also influenced by season, temperature, growing methods and location. Milling conditions also contribute and poorly cleaned paddy can result in husk percentages as high as 35 per cent through the inclusion of chaff, bran, broken rice, etc. Houston quotes values ranging from 16.3 to 26 per cent but indicates that 20 per cent is an acceptable average.

Husk availability and utilization

Rice production on a global basis from 1978 to 1982 is shown in table 4 (3). As discussed in chapter I, the approximate availability of husk in 1982 is therefore 81 million tonnes.

In reviewing the manner in which this renewable resource is handled, it is clear that probably as much as 50 per cent is not properly utilized and about one-third is discarded with no attempt at utilization. This situation, however, differs substantially between developed and developing countries and from region to region in individual countries. In developed countries the mills are generally much larger than in developing countries. Beagle (1) indicates that in California in 1978, there were eight mills to handle an annual crop of 2 million tonnes. By way of contrast, to handle an annual crop of approximately 1.2 million tonnes, in 1975–76 in west Malaysia there were 1,824 mills, of which 1,741 had a capacity of less than 2 t/h (22). Peak husk production in California at any particular mill was of the order of 450 t/d whereas for most of the mills in west Malaysia, it was of the order of 4 t/d. Beagle cites a similar situation in Bangladesh where 80 per cent of the annual crop is milled in the villages by hand threshing. Similar comparisons exist in most developing countries. Clearly the availability of husk and its utilization or disposal present different problems according to the size of the mill.

TABLE 4

(million tonnes)									
	1978	1979	1980	1981	1982				
Australia	0.5	0.7	0.6	0.7	0.9				
Bangladesh	19.6	18.8	20.8	20.1	20.4				
Brazil	7.3	7.6	9.8	8.6	9.7				
Burma	10.5	10.5	13.3	14.2	14.0				
China	138.2	146.7	142.3	146.2	148.0				
India	80.7	63.6	79.8	81.7	74.0				
Indonesia	25.8	25.9	29.8	32.8	32.8				
Japan	16.3	14.9	12.2	12.8	13.3				
Nigeria	0.5	0.9	1.1	1.2	1.2				
Pakistan	4.9	4.7	4.7	4.9	4.8				
Philippines	7.2	7.5	7.6	7.7	8.4				
Republic of Korea	8.4	7.9	4.9	7.0	7.6				
Thailand	17.5	15.8	17.4	17.8	17.0				
Unites States of America	6.0	6.0	6.6	8.4	7.1				
USSR	2.1	2.4	2.6	2.4	2.5				
Countries not listed									
above	40.4	41.7	43.8	45.7	44.9				
World Total	385.9	375.6	397.2	412.2	406.6				

WORLD RICE PRODUCTION

* Provisional.

† Forecast.

Geographical distribution is also a factor since in a small region there may be significant husk production but transport cost would need to be considered in the economics of any planned utilization. For example, in Alor Setar, there were sufficient mills in a 15 km radius to produce more than 40,000 tonnes of husk per annum. A husk dump associated with one mill in this area is shown in fig. 1.

A further difficulty associated with the utilization of an agricultural residue is its seasonal availability, a problem that can also be aggravated by the mill size. At a large mill, any utilization of say up to 450 t/d will require either larger capital investment or storage area than for a small mill. The cash return from such investment must be balanced at least with the returns from utilization, although this can be discounted to a certain extent if the husks are used for fuel in the mill or if the cost for disposal is high.

Changing pollution control requirements in many (mainly developed) countries mean that disposal by heap burning in the paddy fields is no longer acceptable and strict emission laws control disposal in furnaces. For example in Australia, it costs about \$5 per tonne to dispose of husks and this amount (approximately half a million dollars per annum) can obviously be turned into a saving if a suitable method of utilization can be found.

Previous reviews of techniques available for husk utilization have indicated potential applications ranging from filter aids for pressing non-citrus fruits to abrasives (1, 20). However, it is apparent that in developed countries, the predominant use of husk is as a seed supplement (whole, ground, mixed with other supplements and pelletized or chemically

altered), for agricultural use. While husks are used for a similar purpose in developing countries, they are primarily disposed of as fuel, either in the rice mill itself, for parboiling rice, for brickmaking or for domestic cooking.

For building, attempts have been made to utilize rice husks mixed with Portland cement to make blocks. In general, strength requirements can be met but the cup-like form of the husks requires an excessive amount of cement; hence the blocks are uneconomic. Similarly when husks are used to make building board, the amount of adhesive used, whether it be Portland cement or thermosetting plastic, is generally beyond that required for other materials such as wood fibre, sisal or coir. Also the short length of the husk mitigates against mechanical interlocking contributing to strength and wet processing to make boards.

Few data are available on husk utilization but it can probably be concluded that more than one-third of the husk on a global basis is disposed of by burning. In energy terms, this is equivalent to throwing away more than \$500 million each year.



Figure 1. Rice-husk dump near Alor Setar, Malaysia

III. THE INTERMEDIATE PRODUCT - RICE-HUSK ASH

Factors influencing the silica form

In the conversion of rice husks to ash, the combustion process removes the organic matter and leaves a silica rich residue. However, such thermal treatment of the silica in the husk results in structural transformations that influence both the pozzolanic activity of the ash and its grindability.

When rice husks are first heated, weight loss occurs up to 100° C due to evaporation of adsorbed water. At about 350°C, the volatiles ignite, causing further weight loss and the husks commence to burn. From 400°C to 500°C, the residual carbon oxidizes and, as shown in fig. 2 (23), the majority of the weight loss occurs in this period. The silica in the ash is still in an amorphous form. Above 600°C, some conversion to quartz occurs. As the temperature is increased conversion to crystalline forms of silica progresses with the formation of firstly crystobalite and then, at higher temperatures, tridymite. Prolonged heating at temperatures beyond 800°C produces essentially crystalline silica. However, the ash will not sinter until the temperature exceeds 1700°C.

Chopra, Ahluwalia and Laxmi (24) have examined the morphology of RHA obtained from different thermal conditions using scanning and transmission electron microscopy. For husks burnt at 600°C, the ash consisted of a discrete arrangement of mineral particles that were not clearly defined. At higher temperatures (1,000°C), the silica grains were more regularly organized. In addition, interlocked hexagonal silica crystals were evident in stacks scattered over the husk skeleton. The arrangement of the silica crystals appeared to be in the form of rib-like structures.

The relative proportions of the forms of silica in the ash depend not only on the temperature of combusion but also the duration. Mehta (13) states that totally amorphous silica can be produced by maintaining the combustion temperature below 500° C under oxidizing conditions for prolonged periods or up to 680° C provided the hold time is less than one minute. Yeoh (25) *et al* on the other hand, showed that if the duration time was less than one hour, a combustion temperature of 900° C could be used with the ash still remaining amorphous. At 1,000°C, a duration time of greater than 5 minutes was sufficient to produce crystalline silica forms.

The forms of crystalline silica in the ash are most commonly evaluated using X-ray diffraction techniques. Chopra, Ahluwalia and Laxmi reported that for incinerator temperatures up to 700°C, the diffractograms indicated that the silica form was amorphous. A diffractogram for ash obtained by pyroprocessing for two hours at 600°C is shown in fig. 3. At higher temperatures the formation and sharpening of peaks progresses with the formation of firstly quartz, then cristobalite and tridymite. Kapur (26) has studied the influence of temperature on husks incinerated for 12 hours and his results are shown in fig. 4. Chopra, Ahluwalia and Laxmi studied the influence of time of incineration for husks burnt at 700°C. As would be expected, the diffractograms indicated that the silica crystals grew with time of incineration.

Apart from influencing the degree of crystallinity of the silica in the ash, the timetemperature relationship also influences the specific surface area of the ash, a parameter which is obviously closely related to the chemical reactivity of the ash. In addition, Ankra (27) has shown that the combustion environment affects specific surface area, so that time, temperature and environment must be considered in the pyroprocessing of rice husks to produce ash of maximum reactivity.

In his study, Ankra considered the effect of grinding the husks before burning, burning under oxidizing conditions, chemical treatment of the husks before burning and burning under different time-temperature conditions. Ankra proposed that the objective of pyro-







Figure 3. X-ray data on heat-treated RHA at different temperatures



Figure 4. X-ray data on rice husk burnt at 500°C for 2 hours ($Cu_{K\alpha}$ radiation)

processing was to remove the cellulose and other combustibles while preserving the pore structure of the silica-rich skeleton. An example of such a skeletal structure is shown in fig. 5. Ankra showed that the specific surface area was controlled mainly by the micropore volume and effects which reduced the micropore volume decreased the specific surface area. Both temperature and time at that temperature caused the pores to collapse and the particles to coalesce.

However, the specific surface area does not decrease uniformly with time or temperature due to further particle coalescence which results in the creation of new micropores. Hence, as is shown in fig. 6, clearly defined peaks can be observed in the relationship between specific surface area and temperature. The relationship between specific surface area and time shows a similar form. Vigorous combustion also leads to collapse of the pore structure and pyroprocessing in an oxidizing atmosphere resulted in a reduced specific surface area. On the other hand pyroprocessing in a carbon dioxide atmosphere produced a lower heat of reaction and the pore structure was less damaged. Hence the specific surface area was greater.

In fact, any method of processing the husks that permitted lower temperatures and retention times resulted in higher specific surfaces. Accordingly, breaking down to cellulose with zinc chloride prior to pyroprocessing in a carbon dioxide environment resulted in very high specific surface areas as did grinding the husks prior to combustion. However, grinding the husks and pyroprocessing in an oxidizing atmosphere resulted in a decrease in specific surface area since it produced vigorous combustion and a reduction in the micropore volume.

If pyroprocessing occurs between 450° C and 550° C, carbon will remain in the ash and the ash will be black. Ankra has shown that the carbon in the ash is amorphous and combined with other elements.

As the temperature of pyroprocessing becomes higher the ash becomes progressively whiter. However, ash recovered from the interior of large masses of burnt husks where air access is restricted, such as in heap burning, is a lilac pink colour. X-ray defraction analysis indicates that this latter ash is completely crystalline (see fig.7). There is some evidence to suggest that rapid pyroprocessing at high temperatures, i.e. 900°C for one minute, also produces black ash. This is probably due to the lack of time available for the carbon to oxidize, particularly in the interior of the husk. As shown in fig. 8, such rapid pyroprocessing results in the formation of quartz, the low temperature crystalline form of silica, but the peaks associated with tridymite and cristobalite are not present. It has been found that reheating the ash to remove the carbon takes a relatively long period of time and higher temperatures, with the consequent effect that silica is further converted to crystalline forms.

Ash properties

Beagle (1) defines a char as 'the residue remaining from a burning or pyrolytic conversion of rice husk, with carbon content of over 1.5%.' As such, chars can range from a black ash with a carbon content in excess of 40 per cent to a grey ash with a carbon content around two per cent.

Little information could be found in the literature on the physical properties of the ash, but it is apparent that the properties are influenced by the condition of pyroprocessing. The unit mass of the ash ranges from 200 to 400 kg/m³ while values from 2,000 to 2,300 kg/m³ have been quoted for bulk density. Qurashi *et al* (10) determined the thermal conductivity of ash derived from burning husks as a boiler fuel as 0.062 W-m³-K³.



Figure 5. Skeletal structure of RHA (x 736 magnification)



Source: Ankra (27).

Figure 6. Influence of temperature and combustion time in oxidising environment on the specific surface area

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Figure 7. X-ray data for ash containing crystalline silica ($Cu_{K\alpha}$ radiation)



Figure 8. X-ray data for rice husk burnt at $900^{\circ}C$ for I minute (Cu_{Ka} radiation)

The chemical reactivity of the ash, particularly in combination with lime, is related to the form of silica in the ash and the carbon content. Mehta proposed the silica activity index as a measure of the 'degree of amorphousness of silica' in the ash. The index is determined by calculating the percentage of available silica that dissolves in an excess of boiling 0.5N sodium hydroxide in a three-minute extraction in a stainless steel beaker on a minus 325 mesh sample. The higher the amount dissolved the more amorphous the silica. For example, quartz and crystobalite have silica activity indexes of 0.05 and 4 per cent respectively, while RHA containing silica in the amorphous state can have an index as high as 74 per cent. Results reported by Shah (28), Yeoh et al (25) and Chopra, Alhuwalia and Laxmi (24) indicated that the amount of silica dissolved decreased as the temperature of incineration of the ash increased, as would be expected. Shah further proposed that the silica activity index was somewhat tedious and that a simpler method was to titrate a solution containing one gram of the ash in 25 mL of distilled water against methylene blue. The amount of methylene blue required to produce a colour change could also be used as an index of the state of the silica in the ash. Shah's results are shown in table 5. The results of Yeoh *et al* shown in table 6 also indicate the influence of time of combustion as well as temperature on the silica activity index.

The method proposed by Mehta is similar to that appearing in ASTM C593-66T. But while it is useful in the form described above, it does not relate directly to the strength potential of the ash (pozzolan). As such it has been replaced by the lime-pozzolan strength-development test, or lime-reactivity test as it is more simply known. Strength development can be accelerated by storing the specimen above water at between 52 and 56°C. Chopra, Alhuwalia and Laxmi (24) found that the lime reactivity decreased from 16 MPa to 2 MPa as the temperature of incineration increased. ASTM C593-66T specifies a minimum of 4.1 MPa at 7 days, while the relevant Indian Standard, IS 1727/1344, specifies a minimum value of 5.0 MPa at 10 days.

Pyroprocessing conditions	Silica activity index (%)	Volume of methylene blue (mL)		
500°C for 1 h	70.4	15.0		
600°C for 1 h	70.4	14.5		
700°C for 1 h	70.4	13.0		
800°C for 1 h	66. 7	9.5		
900°C for 1 h	55.1	6.0		
1000°C for 1 h	54.7	5.0		
Ash from PCSIR incinerator*	69.3	13.0		
Ash from scaled up PCSIR incinerator†	80.0			

TABLE 5

RELATION BETWEEN ASH REACTIVITY AND PYROPROCESSING CONDITIONS

Source: R.A. Shah (28).

* See fig. 15 for details of the incinerator.

+ See fig. 16 for details of the incinerator.

Pozzolanic reactivity can also be evaluated using the method specified in the ISO R836 Pozzolanicity Test for Pozzolanic Cements. Chopra, Alhuwalia and Laxmi (24) reported that for a given fineness, the ash gradually lost its pozzolanicity as the temperature of incineration was increased and that ash derived from husk used as fuel exhibited no pozzolanic activity.

Ashes that contain amorphous silica usually contain unoxidized carbon. The amount ranges from 5 to 15 per cent, depending on the conditions of combustion, but where the pyroprocessing conditions can be controlled, the amount is limited to about 10 per cent. Research has shown that the ash reactivity is reduced if the carbon content exceeds 15 per cent (see chapter 6 for further details). As shown in table 7, the presence of carbon can mask the influence of pyroprocessing parameters on ash reactivity. Such results nevertheless do indicate the optimum conditions of incineration.

TABLE 6

SILICA ACTIVITY INDEX OF ASH PREPARED UNDER DIFFERENT PYROPROCESSING CONDITIONS (Percentage)

	Incineration temperature (°C)							
Duration of incineration (min)	300	400	500	600	700	800	900	1000
5	52	49	45	54	54	45	44	36
30		49	64	66	66	56	31	18*
60	_	57	74	65	63	68	29	17*

Source: Yeoh et al (25).

* Samples showing crystalline X-ray diffraction patterns.

TABLE 7

RELATIONSHIP BETWEEN PYROPROCESSING CONDITIONS, CARBON CONTENT AND ASH REACTIVITY

Pyroprocessing Temperature (°C)	conditions Time (h)	Strength (MPa)	Carbon content (wt. %)
600	1	6.1	25.9
600	2	6.2	21.4
600	4	7.3	9.0
600	12	7.1	5,3

Ash utilization

The potential applications for utilizing RHA are probably more extensive than for rice husks themselves. They are shown in tabular form in table 8. In practice, in developing countries, ash utilization is extremely limited, with less than five per cent being used for monetary gain. As far as can be ascertained, there is no commercial use in the building industry.

Category	an a	Tech	Commercial Status†			
	Form**	Proved	Possible	Conceptual	Proved	Possible
Absorbent						
Marine spills	LC			+		
Oil absorbent (Greasweep)	LC	+			xo	
Sweeping and floor cleaning (Greasweep)	LC					
Building material component						
Calcium heterolsilicate brick (Silicior)	LC	-				0
Cement (impingement process)	LC		+			0
Concrete (acid-resistant)	HC		· ‡ ·			0
Concrete blocks	LC	water-				0
Lightweight concrete	LC	+				0
Carbon source						
Activated carbon	HC	+				0
Adsorption media (water purification)	HC		-+-			
Coagulant aid (water purification)	HC		+			
Decolorising charcoal	HC	-				0
Fine filter media (water purification)	HC		-			0
Carrier						
Catalyst	LC					
Fertiliser						
Anti-caking agent	LC	+			xo	
Coating for prills	LC		4-			0
Filter media						
Liquid, gas or oil	LC	+				0
Solids removal	LC	**				0
Filling material						
Grit toothpaste	LC	-			0	
Rubber compounding (as carbon silica)	HC HC		+			x

Table 8 (continued)

Category		Techi	nical Status*	 Construct Reference and a second s Second second secon second second sec	Commercial Status†	
	Form**	Proved	Possible	Conceptual	Proved	Possible
Rubber compounding (as silica) Silica aerogel insecticides	LC LC		ŧ	÷		X
Hydrophonic media Ash media	LC					0
Insulation uses Bridge undercoat (steel beams etc.) Hot tops (steel plant) Ingot insulation (steel plant)	LC LC HC	+ + +			o xo	O
Pigment Carbon paper, ink extender Paint Porcelain enamel suspension agent Varnish, shoe polish	HC LC HC			+ + + +		
Refractories Ceramics Heat-insulation brick from ash Insulating brick (medium temperature) Porous media	LC LC LC LC	+ + + +			0 0 0	0
Silica source Dehydrating agent Desiccant and deodoriser Finely divided silica Fumed silica	LC LC		+	+ + +		0
Glass manufacture Landscaping effects (rocks) Silica coating (welding electrode) Silicon carbide nitride Silicon tetrachloride	LC LC LC LC LC	+	+ + +	.+		0 0
Sintered glass material Sodium silicate	LC		- 	+		0

Table 8 (continued)

Category	Technical Status*				Commercial Status†	
	Form**	Proved	Possible	Conceptual	Proved	Possible
Thickening agent	LC			·+		
Soap manufacturing Soap ingredient from ash	LC	÷			0	
Soil use Mulch media for surface control Silica regeneration (in silica-deficient soils)	HC LC	+	+		хо	o

Source: E.C. Beagle (1). * Technical status: + = technically feasible. † Commercial status: o = small commercial use. ** Form of husk product: LC = low-carbon ash. HC = high-carbon ash.

IV. RICE-HUSK ASH CEMENT PRODUCTION

Rationale

It has been shown that RHA contains silica whose reactivity is controlled by the conditions of pyroprocessing the husk. When combined with lime, the silica forms calcium silicate hydrates which are the compounds in Portland cement primarily responsible for strength. Materials containing reactive silica are known as pozzolans and are commonly used in cement production and the manufacture of concrete. They occur as natural soils, e.g. trass or are artificially produced, e.g. fly ash. For example, it is currently estimated that there are seven million tonnes of Portland pozzolan cements produced in India per annum (out of a total of 19 million tonnes). Their use is widely documented, the basic chemistry adequately researched and accepted standards for control and specification are available.

In contrast, RHA is available as a waste material in many developing countries where Portland cement is an expensive commodity. Due to the scattered availability of the ash, however, the development of cements based on RHA has never been seen as a competitor to conventional cement production. Rather, RHA cements have been considered as binder materials produced in the rural sector and primarily marketed and sold in that sector. Other benefits include the contribution to regional self-sufficiency, the utilization of a waste material and a reduction in pollution.

Methods

The Central Building Research Institute of India (CBRI) has proposed two processes for producing lime/RHA cements (29). The first consists of burning sun-dried hand-made balls of husk and clay. After combustion the material is ground with hydrated lime in a ball mill to produce a cementitious material. No control of the combustion is attempted. The process no doubt converts the clay to a reactive form but the contribution of the RHA is somewhat variable. Lime reactivity tests carried out in accordance with Indian Standard IS 1727-1967 (which is essentially equivalent to ASTM C593) give values ranging from 6.3 to 10.4 MPa. The minimum permissible strength is 5 MPa.

In the second process lime sludge, a waste from the sugar and paper industries, is used instead of the clay. The cakes or balls again are hand-made and fired in the bench type kiln shown in fig. 9. It is claimed that the temperature of combustion is sufficient to calcine the lime sludge. As this temperature is approximately 850°C, it is likely that the RHA will be converted to a crystalline, and hence low reactivity, form. Strength results, however, were quite reasonable, a mortar consisting of one part cement to three parts sand had a compression strength after 28 days ranging from 3.9 to 7.8 MPa when tested in accordance with IS 712-1973. Datta and Dass (30) reported the results of field tests near Roorkee but neither process has been developed to the pilot plant stage.

The use of RHA derived from processes where the husk had been used as a source of energy, dates from 1946 with research reported by McDaniel, and in 1953–56, reported by Hough and others. In this early work there is no reference to the ash being ground, a development patented by Kapur in 1974 (31). As will be appreciated, the combustion conditions when the husks are used as a fuel vary considerably; hence the reactivity of the ash will also be variable. This variability can be compensated by the length of grinding which, as is discussed in chapter 7, has an influence on the economics of the process.

The method of utilizing boiler ash as a silica source was developed by Kapur and marketed as Ashmoh: Ashmoh is a lime-ash cement; Ashment is a Portland cement-ash blend. At least two small plants are in production, one at Alor Setar, the other at Kurukshetra in India. Three other plants, at Salem, Mysore and Banda in India are also said to be in production.



Source: Dass and Mohan (29). Figure 9. Burning of rice husk/lime sludge cakes


Figure 10. Heap burning at the Kurukshetra Ashmoh plant



Figure 11. X-ray data on heap-burnt ash ($Cu_{K\alpha}$ radiation)



Figure 12. Rice-husk ash incinerator



Figure 13. Method of charging incinerator with rice husk

Apparently due to the lack of availability of boiler ash, the plants at Kurukshetra and Alor Setar have been using heap-burnt ash. The arrangement for heap burning at the Kurukshetra plant is shown in fig. 10. Apart from the pollution problems, heap-burnt ash is also variable with respect to the silica structure produced. The external layers of husk in the heap burn vigorously but blanket the interior mass. Combustion in the interior occurs in a predominantly carbon dioxide atmosphere and high temperatures result due to the fuel value of the husks. If the temperature is monitored and the ash mass does not exceed 650°C, it is likely that the ash will be amorphous. Temperature control can be achieved by having a small husk volume in the heap so that it can be quenched if the temperature rises too quickly. However, as can be seen from fig. 10, the husk masses are fairly substantial and as a result the ash produced is crystalline, as shown by the X-ray diffraction pattern in fig. 11.

Temperatures as high as 1,200°C have been recorded in the interior of large masses of burning rice husks. While such a method is expeditious from a production point of view, it results in an ash of low reactivity requiring an excessive grinding time to produce a suitable pozzolan. The incinerator shown in fig. 12 represented an attempt to obtain a large amount of ash in as short a time as possible. The husks were pushed through openings in the domed roof (fig. 13) and continually added as combustion at the bottom reduced the volume. Eventually the incinerator was packed full of very high temperature ash and left for some weeks to cool. The ash exhibited a lilac pink colour and X-ray diffraction analysis, (fig. 14) indicated it was completely crystalline. From a cement point of view the plant owner might just as well have taken sand from the nearby river and ground it in his ball mills.

The last group of methods are based on controlled pyroprocessing. According to a 1979 report by Mehta (14), "For large-scale industrialized burning operations, Pitt has designed a furnace which looks like an inverted cone into which rice husk is sucked due to negative pressures maintained by an exhaust fan. From the furnace the hot gases containing ash are taken to a boiler, and finally to a multicone separator which removes the ash from the gases. Thus the heat produced by combustion of husk is usefully recovered in the form of steam." A flow diagram of the process is shown in fig. 15.

As would be expected the capital investment varies according to the size of the plant and the type of energy recovery system desired. Mehta (14) states that based on 1974 prices, the cost of a one ton/hour husk burning plant, excluding the energy recovery system, was about \$135,000. The total installed cost for such a plant consisting of a husk storage shed, transport conveyors, furnace and grinding mill was estimated at about \$235,000. In 1976, a 7.5 t/h rice-burning unit was put into operation adjacent to a large rice mill in Stuttgart, Arkansas. Although the total installed cost was \$1.5 million, it was claimed that the plant could pay off the invested capital in two years.

The capacity and cost of the Mehta-Pitt fluidized-bed furnace system have been considered inappropriate for the requirements of producing a rural binder. As a result, development has focused on producing a low-cost incinerator while still permitting control over pyroprocessing.

At the Peshawar conference in 1979, the PCSIR proposed that an incinerator made from a 44-gallon oil drum be used for pilot plant studies (32). Details are shown in fig. 16. Control of the combustion environment was to be achieved by varying the air flow through the central tube. Scaling up was proposed for a 4-t/d unit at Deepalpur in Punjab Province. However, it was found that the production capacity of the drum incinerator was inadequate and that corrosion severely reduced the profitability of the process. For example, it was found that 22 drum incinerators were needed to produce half a tonne per day of ash and that the drums, which were relatively expensive, would require replacing in a two-year period. As a result, a brick incinerator (28) of the type shown in fig. 17 was constructed. It was proposed that the incinerator shown in fig. 18 could be constructed to increase the capacity of the incinerator or that banks of the type shown in fig. 17 could be used.

The Deepalpur plant was built next to a rice mill and operated by the mill owners. After a period of less-than-successful development, the owners contracted to sell the husk to a local



Figure 14. X-ray data of ash obtained from incinerator shown in figure 11 ($Cu_{K\alpha}$ radiation)







brick manufacturer and production of RHA cements ceased. It was noted that all problems were essentially related to quality control of every aspect of producing a lime-ash cement. The ash was of variable quality, the grinding times were insufficient and the lime was of poor quality and improperly calcined.

The Cement Research Institute of India (CRI) has developed a brick incinerator that is similar in many respects to that shown in fig. 17. A prototype and a bank of four kilns are shown in figs. 19 and 20 respectively. It will be noted that the CRI incinerator has more openings than the PCSIR design. The purpose of these openings is to allow better air flow through the rice husk mass; the combustion process can also be controlled by shutting off these holes. The bottom discharge door is also set higher off the ground in the CRI design (fig. 21), allowing the ash to be quickly removed. The inside of the incinerator is covered with 16 gauge fine-wire mesh (fig. 22), a common feature of PCSIR designs. A pyrometer to control the incinerator process is considered to be standard equipment for the CRI incinerator (fig. 23).

The CRI has plants in commercial operation at Nilokheri, Madras, Gumtur and Nagpur. The plant at Nilokheri also operates as a demonstration and training centre. It is claimed that at least six additional plants are in the process of either construction or commissioning.

The Standards and Industrial Research Institute of Malaysia (SIRIM) has conducted field studies using a modified Yamamoto paddy drier of the type shown in fig. 24, to produce an amorphous ash (36). Although the tests were reasonably successful, no pilot plant investigations were carried out and it is understood that recent studies have been centred round the feasibility of a Mehta-Pitt system. It should be noted that one of the main reasons for the research programme is the disposal of the husks rather than utilization of the ash to manufacture an alternative cement.

Chopra (33) reported that the Regional Research Laboratory at Jorhat, India, had developed a fluidized bed kiln for producing reactive ash from rice husk. He indicated that trial runs had been made; few other details were available. The Rice Growers Co-operative in Griffith, Australia, also developed a fluidized bed furnace or combustor, as it is called (34). The furnace burns about two tonnes of husk per hour and produces essentially amorphous silica. The heat generated during combustion is used to dry citrus pulp. The cost of the furnace is certainly inappropriate for developing countries and it is likely that the fluidized bed concept is in general too expensive for small-scale RHA cement production.

Shrestha (35) has indicated that small-scale production of lime-ash cements is being undertaken in Nepal. The ash is obtained by burning the husks in locally-designed incinerators, using ash from husks used as fuel for parboiling rice or from other fuel sources. In the Kathmandu valley particularly, the husks are sold as fuel. Their cost and the cost of transportation reduces the economic viability of methods based on controlled pyroprocessing.

Grinding

Common to all methods is the necessity to grind the husk to a particle size compatible with the required ash reactivity. In the plants developed to date, this has been commonly done in conventional steel ball mills, one to two metres in diameter and between one-and-a-half and two metres long. The mills are powered by 10–30 hp motors.

A small mill with a 10 hp (7.46 kW) motor used at the Ashmoh plant in Alor Setar, is shown in fig. 25. The grinding media are either steel balls or rods, the latter being generally accepted as more efficient. The mill charge usually consists of three-quarters grinding media; for a 1.5 m diameter by 1.5 m long mill, this can weigh as much as 1.5 tonnes. Due to the weight and replacement cost of the steel, attempts are being made to utilize ceramic balls but no data are available on their cost effectiveness.

In India, edge-runner mills have been used; however, the sophistication and cost of other











Source: PCSIR (28). Figure 18. Brick double-incinerator developed by PCSIR



Figure 19. CRI prototype incinerator



Figure 20. CRI-designed incinerator at the Nilokheri plant



Figure 21. Method of discharge from the CRI incinerator



Figure 22. Interior of the CRI incinerator showing fine-wire mesh



Figure 23. Pyrometer used to monitor the combustion process in the CRI incinerator



RICE HUSK BURNER



Figure 24. Yamamoto paddy drier adapted by SIRIM to produce amorphous ash

mills, such as vibratory grinders, probably preclude their use.

Grinding time relates directly to the nature of the silica in the ash. To produce pozzolan of the same Blaine fineness, grinding can take as long as seven hours for highly crystalline ash and as little as one hour for ash rich in amorphous silica. Ankra (27) has pointed out that for ash burnt under controlled conditions, the specific surface area should be relatively insensitive to comminution since specific surface area is controlled by the micropore volume. Hence particle size would not have a significant effect on specific surface area until it is similar to the average micropore diameter. This general trend can be seen in the results shown in fig. 26, relating grinding time to specific surface area measured by the BET method.

Since the specific surface area is predominantly controlled by the micropores, factors influencing specific surface area can best be studied using the BET method. It will be apparent, however, that the BET method cannot be used as a measure of quality control for RHA ash fineness given the likely conditions of manufacture of RHA cement. As a result, the Blaine air permeability method is used even though it is recognized that it grossly underestimates the specific surface area. Also, the factors that influence the Blaine specific surface area (and vice versa).

Cook and Suwanvitaya (37) have investigated the influence of combinations of time of pregrinding amorphous ash and intergrinding with lime on the compressive strength. They showed that pregrinding the ash prior to intergrinding produced higher 7-day strengths but thereafter the effect of pregrinding was negligible. Intergrinding for periods in excess of 4 hours only marginally increased compressive strength and was not warranted from an energy consumption point of view. The best balance between grinding time and strength was an intergrinding time of two hours with no pregrinding.

Intergrinding the ash with Portland cement also increases the strength of the blended cement. This is due in part to the further comminution of the Portland cement. However, the increased fineness of the Portland cement will increase the water demand for a constant workability of the blended cement. As a consequence, the strength of the mortar or concrete will be reduced due to the higher water:cement ratio. Therefore it is preferable to pregrind the ash prior to blending with Portland cement, and to intergrind only for a period required for thorough mixing.

It will be apparent that the necessity for grinding has a significant influence on plant capacity since it reduces production to a batch process. As a result, if long periods of grinding are required either the production output will be limited or larger ball-milling capacity will be required, the latter obviously resulting in higher capital costs for the plant. In addition, in developing countries, electricity is comparatively expensive and frequently intermittent in supply. Hence, given all other factors, it is desirable to produce an ash that requires as short a grinding time as possible.



Figure 25. Ball milling equipment used in the Alor Setar Ashmoh pilot plant



Figure 26. Influence of grinding time on the BET surface area

V. THE ROLE OF LIME

Introduction

Rice-husk ash can be produced to conform to most specifications for pozzolans (see, for example, ASTM C595-1975 Standard Specification for Blended Hydraulic Cements). As such it can be used as a partial replacement for Portland cement, as will be discussed in chapter VI. However, its main use, particularly in the rural sector, is as a lime-pozzolan cement. Therefore availability of lime is an integral part of the manufacturing process, whose economics can be considerably improved by incorporating lime-burning as a basic step in producing cements based on RHA.

Production

Lime is produced in a two-stage process: in the first, limestone is calcined in a kiln; in the second stage the calcium oxide is slaked, i.e.

Stage 1 limestone — quicklime (calcium oxide)

Stage 2 quicklime + water -----> slaked or building lime.

Limestone can occur as a sedimentary or metamorphosed rock and hence can range from a soft material such as chalk to a hard crystalline marble; it can also be obtained from other sources such as deposits of shells. Boynton (38) has classified limestone into two types, viz. high calcium and dolomitic. High calcium limestone consists of more than 90 per cent calcium carbonate; dolomitic limestone contains between 40 and 45 per cent magnesium carbonate. While limestone of these compositions may be the most suitable for lime production, other types are also used. For example, the older alluvial plains of Pakistan and India contain calcareous concretions known as kankar. Kankar ranges in size from small grains to large nodules, usually contains from 40 to 80 per cent calcium carbonate, and is commonly burnt for lime production.

World production figures indicate that lime is produced in many developing countries though the small quantities involved suggest that it is primarily produced in small pot and vertical kilns. (see table 9.)

The configuration of the vertical kiln depends on the fuel used. Large kilns (about 50 t/d) are fired by oil or gas and are generally continuous in operation. For smaller capacity kilns, coke, charcoal or firewood can be used as fuel. In many respects firewood is preferable for lime calcination: the length of burning and the low temperature of the flame permit more even burning of the limestone.

Small lime kilns with capacities ranging from 1 to 10 t/d can be either batch or continuous. In the latter (and also for mixed feed kilns), the limestone and fuel are fed continuously into the top of the kiln and quicklime is removed from the bottom. In developing countries today, however, batch kilns are by far the most common type of small kiln used. They are relatively cheap to build and easy to control. They also fit well with the pattern of intermittent production and demand commonly associated with rural industries. Many types have been developed. A batch kiln built in Papua New Guinea (40) is shown in fig. 27. On the other hand, the fuel efficiency of batch kilns is low and the production rate can be slow because of the cycle of operations.

Continuous kilns are similar in construction to batch kilns, except that special facilities are required to feed in the raw materials at the top. For this reason they are commonly built into banks or the sides of hills. The efficiency of continuous kilns is greater than for batch kilns and calcination tends to be more uniform. Details of a continuous kiln developed by the Khadi and Village Industries Commission (KVIC) in India are shown in figs. 28 and 29.

Country	1975	1976	1977
Brazil	3 860	4 740	4 960
Chile	660	660	680
Columbia	1 100	1 100	1 430
Egypt	90	90	100
India*	375	375	390
Italy	1 952	2 122	2 126
Kenya	33*	33	33*
Paraguay	31	35	58
Rep. of Korea	110*	120*	99
Saudi Arabia	17	17	22
United Republic of Tanzania	2	2*	2*
United States of America	19 133	20 228	19 947
USSR*	25 000	25 000	26 000
Zambia	130*	159	165*

LIME PRODUCTION IN SELECTED COUNTRIES (Thousands of tonnes)

Source: Minerals Yearbook, 1977 (39).

* Estimated.

The kiln shown in fig. 29 forms part of the plant for the manufacture of lime-RHA cement at the Nilokheri site. It is interesting to note that the lime kiln operator was not confident of being able to operate this 'modern' development; as a result, a traditional batch-type inverted beehive-style kiln was constructed next to the continuous kiln (see fig. 30).

Depending on the quality of the limestone, it is normally assumed that about two tonnes of raw material are necessary for each tonne of hydrated lime. The amount of fuel used per tonne of hydrated lime varies according to the efficiency of the calcination process. For a simple batch kiln, figures by Ellis (41) and Sauni and Sakula (42) suggest that one tonne of firewood (or 0.4 tonnes of coke) is necessary to produce one tonne of lime. Sakula has suggested that the KVIC kiln, operating in a batch mode, could only require 0.5 tonnes of firewood. In the continuous mode, KVIC claim that the fuel consumption is 0.15 tonnes of coke.

The second stage in the production of building lime is the slaking (or hydration operation). The simplest method, known as platform slaking, usually consists of mixing quicklime with sufficient water to complete the hydration process; any excess water is driven off by the heat evolved during the hydration reaction.

In the manufacture of lime-pozzolan cements, the quicklime can be slightly underslaked to improve the reactivity of the cement.

For use as a plaster, the hydrated lime must be sieved to remove any unhydrated particles or other coarse material. It is then ground to ensure that there are no coarse particles remaining. Lime putty is formed by hydrating quicklime with an excess of water in a tank or pit.

For high calcium limestones, the temperature of calcination is around 900°C. However, for dolomitic limestones, it is much less. Magnesium carbonate normally dissociates around 480°C, but in mixtures with calcium carbonate (as in dolomitic limestones) the magnesium carbonate component dissociates at a higher temperature, averaging around 700°C. Hence,



Source: Hosking (40). Figure 27. Batch lime kilns developed in Papua New Guinea



Figure 28. Continuous kiln developed by KVIC, India



Figure 29. KVIC lime kiln at Nilokheri, India



Figure 30. Village lime kiln at Nilokheri, India

if the dolomitic limestone is calcined at 700°C, the calcium carbonate will be soft-burnt and if it is calcined at 900°C, the magnesium carbonate will be overburnt. In the latter case, the magnesium oxide hydrates very slowly and therefore at a rate different from the quicklime. Problems have occurred, in fact, with lime plasters containing unhydrated magnesium oxide, which begins to hydrate when water is added to prepare the plaster. The expansion accompanying hydration can result in 'pop outs' and cracking. However, in the normal operation of slaking and sieving, any unhydrated material should be retained on the sieve and should not be present in the hydrated lime.

Specifications for lime cover both physical and chemical characteristics. Since it is imperative that the lime be fully hydrated before it is used as a plaster or mortar, most specifications provide limits on particle size and soundness. Soundness can be measured using a Le Chatelier mould or an autoclave, but the purpose of the test is to determine the presence of coarse and unhydrated particles (generally magnesium oxide). The pat test, which consists of simply making a pat from the lime, is not generally favoured since there is no specific measurement for the acceptance and rejection of the lime.

Chemical requirements of the lime include limits on the minimum amount of available lime (70 per cent, calculated as calcium hydroxide) and the maximum amount of magnesium oxide (4.5 per cent).

For manufacturing lime-pozzolan cements, the lime should in general conform to the requirements for building lime. However, if it is underslaked it may not comply with the soundness limits. Also, if the lime is to be interground with the pozzolan the particle size requirements are not so critical.

Hydrated lime gains strength by combination with carbon dioxide in the air to form calcium carbonate. Hence the reactivity of the lime, i.e. its combination with the pozzolan, will decrease if it is poorly stored. It is therefore imperative that the hydrated lime be as fresh as possible or that it be stored in airtight bags prior to combination with the pozzolan.

VI. PROPERTIES OF RICE-HUSK ASH CEMENTS AND THEIR UTILIZATION

The lime-ash reaction

The pozzolanic action of RHA is based on its reactivity with lime, the lime being present as a primary constituent of the mix or as a reaction product formed during the hydration of Portland cement. Since the ash consists essentially of silica, the pozzolanic reaction is that between lime and silica, to form calcium silicate hydrates.

As discussed previously, the reaction is influenced by the nature of the silica, the fineness of the ash (and hence cement) and the presence of other materials such as carbon. The influences of the first two factors on compressive strength is shown in table 10, where the designations LA and BA refer to ash burnt under controlled conditions and ash derived from husk burnt as a boiler fuel, respectively. As would be expected, compressive strength increases as the fineness increases and decreases as the ash becomes more crystalline.

The influence of carbon content on the strength of lime-ash and Portland cement-ash mortars is shown in figs. 31 and 32 respectively. It can be seen that up to a weight proportion of 20 per cent, the influence is negligible.

TABLE 10

Surface area		Compress	Lime	
Sample designation	of ash (cm²/g)	7 day (MPa)	28 day (MPa)	reactivity (MPa)
LA — 1†	15 000	8.0	8.7	8.0
LA — 2†	20 000	13.2	15.7	13.7
BA — 1**	14 500	2.0	8.9	14.0
BA — 2**	20 000	2.0	12.7	15.6

Performance characteristics of lime-ash mortar

Source: Chopra et al (24).

* Tested in accordance with IS 4098-1967.

† Burnt under controlled conditions.

****** Burnt as boiler fuel.

However, as the proportion of carbon is increased to 40 per cent, the effect is more pronounced for lime-ash mortars than for Portland cement-ash mortars.

Carbon content also influences the setting time. For lime-ash mixes, an increase in the carbon content increases both the initial and final sets, while for Portland cement-ash mixes, the reverse is true. (See table 11.)

Strength versus time results for both lime and Portland cement mixes containing ash do not show any significant increase in strength beyond 28 days. The reason for this behaviour is not clear but it is probably related to the reactivity of the ash and the early completion of the lime-silica reaction. Typical strength-versus-time results for both lime and Portland cement mixes are shown in figs. 33 and 34 respectively.

Cook and Suwanvitaya (43) have examined the progress of the lime silica reaction in terms of the reduction in free lime using the modified Franke method (the time variation method).

Cement mix	Cement mix Ash carbon		Setting times		
Portland:lime:ash	content (%)	Initial (min)	Final (min)		
0:20:80	14	135	420		
0:20:80	42	265	675		
0:40:60	14	165	330		
0:40:60	42	270	690		
60: 0:40	14	70	110		
60: 0:40	42	35	50		
80 : 0 : 20	14	120	190		
80 : 0 : 20	42	50	170		
100: 0: 0		40	80		

INFLUENCE OF CARBON CONTENT ON THE SETTING TIMES OF LIME AND PORTLAND CEMENT ASH MIXES

Source: Cook and Suwanvitaya (37).

In table 12, the available lime (CaO) as a percentage of the input that can be extracted, has been determined at 3 hours, 12 hours, and 3, 7, 28 and 90 days after mixing. For the low-lime/low-carbon content mixes (1:4 and 1:1.5) and the low-lime/high-carbon content mix (1:4), it can be seen that nearly all the lime has reacted with the RHA before final set has occurred. Furthermore, although there is a general reduction in the available free lime in the 3 to 7-day period, particularly in the high carbon 1:1.5 mix, the greatest reduction occurs in the first 3-hour period for all mixes. As would be expected for the high-lime mixes, significant amounts of free lime remain even after 90 days. Also, in the period from 7 to 90 days, the percentage of available free lime remains essentially constant for all mixes. These facts would explain the rapid early strength development of mixes containing RHA and the lack of strength development beyond 28 days. In practical terms this kind of behaviour is beneficial because it means that prolonged moist curing normally required with pozzolanic cements, is not necessary.

In general terms, the calcium silicate hydrates (CSH) formed in reactions between RHA and lime are of the CSH(I) type (37). There is some evidence to suggest that CSH(II) forms may also be produced under some circumstances, but they do not appear to be very stable and reversion to CSH(I) occurs. As a consequence, the amount of free lime increases during this period, as is evidenced by the results shown in table 12.

For lime-ash mixes, Cook and Suwanvitaya (43) have indicated that the morphology depends on the lime:ash ratio. For high lime mixes (lime:ash = 1:0.25), the structure consisted of poorly defined crystalline forms; the well-recognizable hexagonal plates of calcium hydroxide are not observed. For lime:ash ratios of 1:1.5 and 1:0.67, crystalline forms were observed, but the crystals were only about 2 μ m across. Attempts to measure the CaO:SiO2 ratio, using energy dispersive X-ray analysis were not successful. In the low-lime mix (lime:ash = 1:4), no crystalline forms were observed.



Source: Cook et al (37).

Figure 31. Influence of carbon content on compressive strength of lime-RHA mortar



Source: Cook et al (37). Figure 32. Influence of carbon content on compressive strength of Portland cement-RHA mortar

Symbol	Lime:RHA	Water/(Lime+RHA)
	$ \begin{array}{rrrrrrrrrrrrrrrrrrrrrrrrrrrrrrrrrrrr$	0.89 0.86 0.80 0.77



Source: Cook and Suwanvitaya (43).

Figure 33. Compressive strength development of lime/RHA mortar

Symbol	Mix Proportion Cement:RHA	Water/(Cement+RHA)
	1:0	0.49
0	1:0.25	0.61
Δ	1:0.67	0.62
	1:1.5	0.74
\diamond	1:4	0.82





Figure 34. Strength development of Portland cement/RHA mixes

EXTRACTED LIME AS A PERCENTAGE OF LIME INPUT

Carbon content (by weight)	Lime:ash ratio		Т	ime afte (%	er mixin ⁄₀)	g	
(%)	(%)	3 h	12 h	3 d	7 d	28 d	90 d
14	1:4	5.9	7.4	7.8	5.6	6.7	7.2
14	1:1.5	6.0	6.5	10.2	5.2	4.6	4.6
14	1:0.67	38.3	52.5	32.1	30.9	33.3	30.9
14	1:0.25	58.3	57.9	60.2	62.1	62.5	52.3
42	1:4	4.6	5.4	5.0	5.2	5.4	5.0
42	1:1.5	34.3	37.0	40.8	27.8	20.1	19.8
42	1:0.67	58.7	56.8	53.1	51.9	48.2	48.2
42	1:0.25	76.4	73.1	70.4	71.3	63.0	61.6

Source: Cook and Suwanvitaya (43).

Cement characteristics

CBRI methods

The properties of the RHA-clay pozzolan are shown in table 13 and those of the lime sludgeash cement in table 14. Performance characteristics of the lime-ash cement are given in tables 15a and 15b. As previously noted, production of these cements has not reached the pilot plant stage; hence no results are available regarding the characteristics of the binders produced under manufacturing conditions.

Methods using boiler or heap-fired ash

Extensive tests on ash obtained from heap burning and boiler ash has been reported by SIRIM (36). Under-ground ash and ash ground to pass the No.350 sieve did not meet the requirements of ASTM C593 (fly ash and raw or calcined natural pozzolans for use with lime). Tests on Portland cement-ash blends indicated that for ash passing the No.350 sieve, the reactivity of the cement was relatively poor unless the ash and cement were ball-milled together. The results shown in table 16 illustrate the better performance of ash obtained from controlled burning in the paddy drier (see chapter IV).

Setting time and soundness results for boiler ash and Portland cement interground for 3, 4 and 5 hours are shown in table 17.

Some information is available regarding the properties and behaviour of Ashmoh and Ashment cements. Chopra (33) has reported that an Ashmoh cement from a pilot plant in Shimoga (southern India) gave compressive strengths of 9, 12.5 and 15 MPa at 3, 7 and 28 days respectively. Further, Raja Muhammed (44) indicated that the Ashmoh cement manufactured in Alor Setar, complied with ASTM C91 requirements as a masonry cement. The results are shown in table 18.

Properties of the Portland cement-boiler ash cements, known as Ashments, are shown in table 19. The proportion of ash in the cements is not precisely known, but it is probably at least 50 per cent.

TABLE 13

PROPERTIES OF A TYPICAL RICE HUSK-CLAY POZZOLAN

Loss on ignition (%)	1.5
Specific gravity (g/cm ₃)	2.34
Specific surface (Blaine), (cm ² /g)	6 200 to 9 500
Lime reactivity test (IS:1727-1967), (MPa)	6.4 to 10.4
Lime pozzolan mortar test (IS:4098-1967), (MPa)	4.3 to 7.0
Setting time (IS:1727–1967) for 1:2 (by wt.) lime pozzolan mix, (h)	
(a) Initial	2-5
(b) Final	22.0
Portland pozzolan mortar test (IS:1727-1967)	98% of the control (compressive strength) at 28 days
(20 per cent cement replaced by pozzolan)	

Source: Dass and Mohan (29).

PHYSICAL CHARACTERISTICS OF CBRI LIME-ASH CEMENT

Characteristic	Result	ISS method
Fineness Residue retained on 150µm IS sieve, (%)	1.0	6932 (Part IV):1973
Residue retained on $74\mu m$ IS sieve, (%)	21.7	6932 (Part IV):1973
Air permeability apparatus Sp. surface area, (cm ² /g)	9840	4031:1968
Bulk density, (kg/m ²) Setting time (by Vicat apparatus) Initial (min) Einal (min)	764 75 500	4031:1968
Soundness Expansion in Le Chatelier's moulds (mm)	2.5	6932 (Part IX):1973
Compressive strength, (MPa) 3 days 7 days 28 days	2.0 2.7 4 9	6932 (Part VII):1973
Heat of hydration, (cal/g) 7 days 28 days	48 65	269:1966

Source: Datta and Dass (30).

TABLE 15a

PERFORMANCE CHARACTERISTICS OF CBRI LIME-ASH CEMENT Relative compressive strengths of cement-sand and binder-sand compositions (MPa)

Curing Period	Cement-sand mixture	Binder-sand mixture		
days	1:6*	1:2*	1:1.5*	
7	1.1	1.1	1.3	
28	2.3	2.3	2.8	

Source: Datta and Dass (30).

* Volume ratios.

TABLE 15b

CHARACTERISTICS OF CONCRETE BLOCKS MADE USING BINDER-SAND-COARSE AGGREGATE MIXTURE

Binder-sand coarse aggregate proportions	28-day compressive strength Compaction factor		Drying shrinkage	Moisture movement
(by vol.)	(MPa)	(MPa)	(%)	(%)
1:1 :2	8.3	10.0	0.02	0.01
1:1.5:3	4.9	6.8	0.02	0.02
1:2 :4	1.9	3.0		

Source: Datta and Dass (30).

TABLE 16

COMPARISON OF AVERAGE COMPRESSIVE STRENGTH OF BOILER ASH AND ASH OBTAINED USING CONTROLLED COMBUSTION* (MPa)

Portland cement: rice-husk ash† (% by weight)	Boiler ash*	Boiler ash** (interground)	Cyclone burner††
10:90	1.9	8.1	1.1
20:80	4.3	11.6	4.8
30:70	5.6	17.0	12.7
40:60	5.4	21.2	16.6
50:50	10.0	20.1	20.1
60:40	10.8	19.3	20.3
70:30	12.4	23.2	19.5
Control	15.3	19.3	15.3

Source: Yeoh et al (36).

Test conditions: sand/cement, 2.75; water/cement, 0.50; curing conditions, 63°C and 95% RH; age of test, 7 days.

- † Ash ground to pass No. 350 sieve.
- ** Ash ground to pass No. 350 sieve then interground with Portland cement for 7 hours.

† Ash obtained using controlled combustion conditions then ground to pass No. 350 sieve.

SETTING TIME AND SOUNDNESS TEST RESULTS FOR INTERGROUND BOILER ASH AND PORTLAND CEMENT*

		Setting time			
Sample	Water/cement ratio	Initial (min)	Final (min)	Soundness (mm)	
Portland cement	0.33	85	183	1.7	
Cement-ash blended for 3 h [†]	0.53	196	340	1.6	
Cement-ash blended for 4 h [†]	0.43	212	325	1.2	
Cement-ash blended for 5 h†	0.44	235	345	0.4	

Source: Yeoh et al (36).

* Tests carried out in accordance with ASTM C595-76.

† Cement:ash ratio 1:1.

TABLE 18

TEST RESULTS FOR ASHMOH CEMENT PRODUCED AT ALOR SETAR, MALAYSIA

Test	ASTM C91	Ashmoh cement	
Setting times			
Initial set	not less than 120 min	180 min	
Final set	not greater than 24 h		
Compressive strength			
Cement:sand 1:3			
3 days	_	10.3 MPa	
7 days	3.4 MPa	10.3 MPa	
28 days	6.2 MPa	9.3 MPa	

Source: Raja Muhammed (44).

Test	IIT Kanpur	Kurukshetra		
Setting times				
Initial set	_	180 min		
Final set	— 10 h			
Compressive strength	<u></u>	,, <u>, , , , , , , , , , , , , , , , , ,</u>		
Mortar				
7 days	10.0 MPa	10.3 MPa		
28 days	16.6 MPa	15.6 MPa		
Soundness		1.0 mm		

TEST RESULTS FOR ASHMENT FROM VARIOUS SOURCES

Controlled pyroprocessing methods

Cook and Suwanvitaya (37) have examined the strength characteristics of cements containing ash obtained by controlled pyroprocessing. The results shown in table 20 are for a constant flow of 110 ± 5 per cent determined by ASTM C109. It can be seen that the water requirement to produce a constant flow increases as the ash proportion increases. The strength contribution of the RHA has therefore been masked to a certain extent by the increase in water:cement ratio.

The results in fig. 33 indicate that the optimum lime:ash ratio from a strength point of view is 1:1.5. Seven-day values reported by Sooriyakumarin and Ismail suggest that a ratio of 1:2.5 is optimum but it is likely that in the longer term a higher lime content would be optimum. Lea (46) indicates that a common lime:pozzolan ratio is 1:2 and it is apparent that this would be applicable to RHA also.

Chopra, Ahluwalia and Laxmi (24) have reported characteristics of RHAM cements produced in accordance with the specification in Appendix 2. These are shown for two cements in table 21. Results for Portland cement-RHA cements are shown in table 22. It should be noted that the water content of the mixes has not been adjusted to produce constant workability. Furthermore, without this adjustment, the workability, as measured by slump, decreases dramatically. For example, Mehta (15) quotes a reduction in slump from 95 to 12.5 mm when 30 per cent of the Portland cement in a concrete mix was replaced by RHA.

Shah *et al* (32) have produced both lime: ash and lime, ash and surkhi cements under laboratory conditions. The characteristics of two of these cement types are shown in table 23.

Few field data are available on the characteristics of RHA cements manufactured using the controlled pyroprocessing method. Limited testing on lime-ash cements from the Nilokheri plant indicate that the cement does not conform to the requirements of the RHAM specification.

COMPRESSIVE STRENGTH OF CEMENT-LIME-ASH MORTAR MIXES

		Water	Cube strength (MPa)				Fineness
Mix number	Cement:lime:ash	cement ratio	7 days	28 days	90 days	365 days	(cm²/g)
1	100: 0:0	0.49	29,3	43.0	47.2	44.0	3 190
2	80: 0:20	0.61	23.4	49.9	51.3	54.4	4 640
3	60:20:20	0,61	22,3	36.2	38.9	41.3	7 480
4	60: 0:40	0.62	19.1	41.6	47.1	52.3	5 860
5	40:40:20	0.67	11.2	18.3	20.8	21.8	11 890
6	40:20:40	0.69	14.9	24.1	27.1	28.3	8 320
7	40: 0:60	0.74	13.2	17.8	19.4	19.9	10 830
8	20:60:20	0.79	4.8	7.8	8.5	8.4	16 360
9	20:40:40	0.74	7.9	11.3	12.7	14.6	14 100
10	20:20:60	0.75	8.4	13.6	14.5	14.8	11 560
11	20: 0:80	0.82	4.8	6.2	7.1	7.6	10 690
12	0:40:60	0.86	6.3	8.5	8.5	8.5	14 180
13	0:20:80	0.89	6.4	6.7	6.9	6.8	12 190

Source: Cook and Suwanvitaya (37).

Sample designation	e Grinding Spe ion time fine	Specific fineness	Specific Setting	g time Final	Compressive Strength	
J.	(min)	(cm²/g)	(min)	(min)	7 days (MPa)	8 days (MPa)
RHAM-G1	115	13 600	180	662	5.7	8.2
RHAM-G2	120	15 200	135	672	6.1	10.8

PERFORMANCE CHARACTERISTICS OF RHAM CEMENT

Source: Chopra et al (24).

TABLE 22

PHYSICAL CHARACTERISTICS OF PORTLAND-ASH BLENDED CEMENT

Sample	Specific surface	Water/	Compressive Strength*		
designation	(cm²/g)	cement ratio	3 days (MPa)	7 days (MPa)	28 days (MPa)
Control	3 780	0.42	24.7	30.7	43.9
LA-1†	5 500	0.43	17.8	26.7	42.6
LA-2†	7 000	0.45	25.5	37.9	51.7
LA-3†	8 500	0.47	27.5	48.1	59.6
BA-1**	6 000	0.41	19.3	26.3	43.1
BA-2**	7 500	0.43	20.1	30.2	46.7
BA-3**	9 000	0.43	19.9	34.6	52.0

Source: Chopra et al (24).

* Tested in accordance with IS 4031–1968.

† Burnt under controlled conditions.

** Burnt as boiler fuel.

Volume change characteristics

Cook (47) has investigated the creep, shrinkage and swelling characteristics of pastes made from Portland cement-ash blends. The volume change characteristics of all series containing RHA were greater than the control mix. However, the increases were not significant when replacing Portland cement up to 30 per cent. This finding is not consistent with that of Mehta (14) who has reported that for a Portland cement replacement of 70 per cent there was no significant difference between the shrinkage behaviour of concrete containing Portland-RHAM and the control Portland cement.

The shrinkage of some lime-ash mortars are shown in fig. 35. As can be seen, the results do not reflect a consistent trend but generally show that as the lime:ash ratio decreases, the shrinkage also decreases.
TABLE 23

Property	Cement Type				
	Lime-ash	Lime-ash-surkhi			
Loss on ignition (%)	8.0	7.0			
Blaine fineness (cm ² /g)	4 200	4 200			
Bulk density (kg/m ³)	750	830			
Specific gravity (kg/m ³)	2 100	2 300			
Water for normal consistency (%)	60	56			
Le Chatelier soundness (mm)	nil	nil			
Setting time (min)					
initial	165	355			
final	255	595			
Compression strength (MPa)					
3 days	10.7	7.8			
7 days	16.1	11.9			
28 days	18.8	21.3			

PROPERTIES OF RICE-HUSK ASH CEMENTS

Source: Shah et al (32).

Durability

It is well established that pozzolanic materials improve the durability of concrete, particularly resistance to chemical attack. RHA would also prove beneficial in this regard. Mehta (14) has shown that concrete and mortars made with RHA cements have superior resistance to acidic environments compared even to Portland cement and other pozzolans. For example, concrete cylinders made with a 35 per cent RHA and 65 per cent low-heat Portland cement were submerged in acid solutions (five per cent of either sulphuric or hydrochloric acid) for a period of 1,500 hours.

In the hydrochloric acid solution the control low-heat Portland cement samples registered a 35 per cent weight loss while the specimens containing RHA showed only 8 per cent loss. The corresponding losses in the sulphuric acid solution were 27 per cent and 13 per cent for the control and RHA cement concretes, respectively. Further, lime-RHA cements stored in a one per cent acetic acid solution remained in excellent condition for more than five years, whereas Portland cement mortars showed surface softening and substantial weight loss within one year.

Yeoh, Ong and Chong (36) also reported improved acid resistance for mortar containing RHA. In their tests 50 mm mortar blocks made from a 50:50 blend of Portland cement and ash were subjected to immersion in a 1.97N hydrochloric acid solution. After 72 days, the surface of the mortar containing the RHA was unaffected, while that made from Portland cement was corroded and pitted.

It is probable that the improved durability of Portland cement-RHA blends is related to the physical structure of the hydrated cement as well as the reduction in the amount of calcium hydroxide.

Due to the substantial reduction in the lime in the hydrating Portland cement, there have been some reservations as to the effect this would have on the corrosion protection offered to reinforcing steel in concrete. The high alkalinity of Portland cement concrete under normal circumstances retards the corrosion process. No information could be found



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Figure 35. Shrinkage of lime/RHA mortars

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regarding the reduction in pH of Portland cement-RHA concretes, but similar reservations were expressed regarding the use of fly ash in concrete. However, there appears to be little evidence to suggest that fly ash in adequately cured concrete has contributed to corrosion of the reinforcement and it is probable that a similar conclusion could be reached for RHA.

The durability of lime-ash mortars has been investigated by Cook and Suwanvitaya (43) by subjecting mortar bar specimens to alternate cycles of wetting and drying. In the wetting cycle, the specimens were flooded in water at 18° C for 25 minutes, the water was then drained off and heat lamps, which raised the cabinet temperature to 45° C were turned on. After 25 minutes, the lamps were switched off and water was pumped into the cabinet again for the cycle to be repeated. The specimens were weighed at the end of a drying cycle. The results shown in fig. 36 relate weight loss to the number of weeks of cycling, one week being equal to 168 cycles.

Weight loss in durability testing is considered due to lime leaching. In the low-lime mix, there is very little free lime present. During the wetting cycle lime is leached from the calcium silicate hydrate, leaving, according to Lea (46), 'a residue of incoherent hydrated silica'. This type of deterioration is a surface phenomenon that is obviously exacerbated by the washing action of the wetting cycle, which exposes new surfaces to be leached. For the high-lime mixes, lime leaching will also occur but the availability of free lime ensures, to some extent, the integrity of the calcium silicate hydrates. Also, the surface of the high-lime mixes will be carbonated, which reduces the extent of lime leaching.

The influence of lime leaching on the durability of low-lime mixes is a further reason why the optimum lime:ash proportions should be around 1:2. It will be recalled that from a strength point of view the optimum lime:ash ratio was also suggested to be 1:2.

Accelerators

The setting time and strength of lime-ash mortars can be accelerated by addition of small amounts of Portland cement. Up to 10 per cent has been added in some circumstances. Other accelerators such as sodium aluminate, calcium chloride, sodium carbonate and ferric chloride can also be added but the cost of these chemicals is relatively high. The influence of sodium aluminate additions on the initial and final setting times of lime-ash mortars is shown in figs. 37 and 38. Lea (46) has reported the use of gypsum as an accelerator for some lime-pozzolan cements but its effect on lime-RHA cements is not known.

The use of accelerators in reinforced concrete containing RHA cement should be viewed with caution due to the effect that such accelerators can have on corrosion of the reinforcement.

Utilization

Mix proportions

The proportions of ash in the cement will depend on the reactivity of the ash. However, for lime-ash mortars it is recommended that the lime:ash ratio should not be less than 1:2, for reasons previously discussed. For Portland cement-ash blends, if the cement to be used in concrete construction, the amount of the pozzolanic constituent should not exceed 40 per cent. In addition, the cement should conform to requirements similar to those for Type P cement specified in ASTM C595. The salient features of this cement type as specified, are shown in table 24. While there is evidence that Portland cement blends containing more than 40 per cent ash have satisfactory strength, the long-term durability of these mixes has not been sufficiently investigated to justify their use.

The water content required to achieve the same consistency is higher for cements containing RHA than conventional masonry cement or other Portland-pozzolan blends. As a result, for mixes of the same consistency, the strength will be less for those containing RHA because the water:cement ratio will be higher.





Figure 37. Initial setting time of lime/RHA paste







Figure 38. Final setting time of lime/RHA paste

TABLE 24

SPECIFICATION FOR BLENDED PORTLAND POZZOLAN CEMENT AS PER ASTM C595 TYPE P

Characteristic	Requirement
Fineness, amount retained when wet sieved on 45 μ m sieve, max per cent	20
Specific surface by air-permeability apparatus, cm ² /g	
average value	2 800
minimum value, any one sample Time of setting, Vicat test:	2 600
set, mins, not less than	45
set, hrs, not more than	7
Compressive strength, as per ASTM C109, MPa	
1 day in moist air, 6 days in water	10.3
1 day in moist air, 27 days in water	20.7
Water requirements, max. weight per cent of cement	64
Drying shrinkage, max. per cent	0.15
Mortar expansion	
at 14 days, max. per cent	0.020
at 8 weeks, max. per cent	0.060

The bulk density and unit mass of cements based on RHA are less than for those based on Portland cement. Lea notes that most pozzolans have a bulk density ranging from 2,300 to $2,600 \text{ kg/m}^3$ compared to $3,150 \text{ kg/m}^3$ for Portland cement. As a consequence the absolute solid volume of the material containing pozzolan is greater than that of an equal weight of Portland cement. However, substituting 40 weight per cent of cement with ash increases the solid volume of the cement plus ash by only 10 per cent. Batching in developing countries is most commonly done on a volume basis and substitution of Portland cement with Portland-ash blends can be done on the basis of equal solid volumes.

The bulk density and unit mass of lime and ash are approximately the same; hence the yield for lime mortars is similar to that of lime-ash mortars.

Optimum mix proportions should be based on field experience and laboratory testing and will depend on strength and consistency requirements. Commonly, mortars have a cement:sand volume ratio of 1:6 but this can be reduced to 1:3 if higher strength mortar is required. For RHAM cement CRI (48) suggests that a cement:sand volume ratio of 1:4 is suitable for most mortar applications. RHAM concrete, for use as foundations for two to three-storey buildings, should consist of cement, fine sand and coarse aggregate in the volume ratio of 1:4.5:8.25 However, concrete mixes should in principle be designed with proper cognizance of the cement and aggregate (fine and coarse) characteristics so that the most economic mix can be proportioned. This is frequently not done in developing countries, with the result that materials are wasted and failures occur. Volume batching, variations in the water added to the mix and the subsequent variations in the strength of the concrete can result in low strength concrete if coupled with variable strength cement. Hence, in small-scale production of cement, it is imperative that cement strength be reasonably uniform, particularly if the cement is to be used in reinforced concrete applications.

Applications

Lime-ash mortars have potential application in brick, stone and concrete block masonry. They can be used for jointing tiles and in hydraulic structures such as canal linings. As plasters, they can be used to provide a waterproof coating to mud, brick and concrete block walls.

Plain concrete can be made using lime-ash cement or Portland-pozzolan blends. Applications include concrete blocks and other cast products such as tiles, well rings, etc. The concrete can be used in foundations for low-rise construction, for flooring and as a road sub-base.

Mehta (14) indicates that in the United States, Portland cement-ash blends have been successfully used for constructing acid resistant floors in food and chemical process plants. Due to the extended service life the cement is able to impart to the floors, it is sold at a sub-stantially higher price than Portland cement.

Little information could be found as to reinforced concrete construction in developing countries using Portland-ash blended cements (49, 50). While there are no technical reasons why the blended cement could not be used, the general approach in the development of cements based on RHA has been that their principal areas of application would be for mortar, plaster and for plain concrete. In the rural areas of developing countries, the cement in these forms would cover about 75 per cent of the applications where Portland cement is currently used.

VII. PRODUCTION AND FEASIBILITY ANALYSIS

Introduction

Cost estimates have been prepared for lime/ash cements based on controlled pyroprocessing, heap-burnt ash or ash obtained from parboiling rice. Although the figures given are in United States dollars, they are based on costs in India. Cost data are obtained from marketing information distributed by a licencee of the Ashmoh process and from published sources (49, 50, 51, 52).

Production process

For the purpose of this analysis, cost estimates are based on a production rate of 600 t/y, which is equivalent to a daily rate of 2 t/d (330 working days at 90 per cent capacity). The amounts of raw materials consumed will depend on the lime:ash ratio; to show this influence, figures have been prepared for lime:ash ratios of 1:3, 1:2 and 1:1.

Controlled pyroprocessing

A flow chart for the manufacture of lime-ash cements based on controlled incineration is shown in fig. 39. The four essential steps are numbered:

(1) Lime burning and slaking;

- (2) Husk incineration;
- (3) Ball milling (grinding the ash and intergrinding with lime);
- (4) Bagging.

For the lime kiln, the KVIC 2.5 t/d model (figs. 28 and 29) is probably the most suitable. Although the maximum output required for the production of lime-ash cement would only be 1 t/d, the excess capacity allows for the complementary production of building lime to improve the profitability of the operation. Also the actual production rate from the kiln is probably closer to 1.5 t/d, given the fact that it is likely to be run in batch mode. The kiln is designed to use either coal or coke as fuel, although when operating as a batch kiln it would be possible to use firewood. In general firewood is cheaper, but the relative economics of different fuel types need to be evaluated for each plant.

For producing lime, the limestone has to be broken down to lumps of about 50 to 75 mm. The kiln is filled with alternate layers of limestone and coal, the bottom layer consisting of a combustible mixture containing coal. Wood or in some cases rice husks are mixed with the coal to promote ignition. During continuous production, alternate layers of limestone and fuel are added to the top of the kiln and the calcined material removed from the bottom. In the batch mode, the kiln is left to expend the fuel and to cool. If the kiln is fired at the end of one day, it can be emptied and refilled the next morning.

The calcined lime is spread over a slaking platform and sprinkled with water, which causes the stones to turn to a light powder, quicklime. Care should be exercised to ensure that the lime does not come into contact with the workers' skin, particularly the eyes. Prior to grinding with the RHA, the lime should be sieved to remove any large particles. If it is not to be used immediately, the lime should be bagged and sealed to prevent loss of reactivity due to carbonation.

It is assumed that the rice husks have an ash content of 23 per cent. Hence the maximum amount of husks to be incinerated is 1,950 t/y (for the lime:ash ratio of 1:3). This would require a rice mill with a capacity of about 10,000 tonnes of paddy per annum. The daily rate of ash production required is 1.36 tonnes. This can be achieved in six brick incinerators 1.5 m square in plan and 2 m high producing 227 kg of ash per incinerator and requiring a



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Figure 39. Flow diagram for manufacturing cements based on RHA

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through-put of about one tonne of husks (or eight cubic metres) per incinerator. To produce reactive ash, a combustion time of between two and three hours is acceptable. The husks are heaped to a height of between 500 and 750 mm in the incinerator and fired at the bottom. Although the burning level slowly rises to the top of the heap, the combustion process is essentially continuous: ash is removed from the bottom and new husks added to the top. The ash temperature is monitored using a pyrometer of the type shown in fig. 23. If the temperature exceeds 650°C the ash should be quenched by raking onto the ground or thinning the ash pile on the ground and, if necessary, sprinkling with water.

After incineration, the ash is collected in piles and left to cool. During this period, most of the remaining carbon in the ash is oxidized and the ash becomes whitish grey in colour, although a black crust usually remains on the outside of the pile.

If the combustion has been incomplete in the incinerator, the piled ash will continue to burn and can become crystalline. Hence the temperature of the piled ash should also be monitored. As far as possible, the ash should be kept under cover prior to grinding since high moisture content produces problems during grinding and subsequent storage of the cement.

The quantities to be ball-milled should be accurately weighed on a platform scale. The ball mill can be an off-the-shelf design. A 1.54 m diameter by 1.54 m long mill driven by a 20 hp (15 kW) motor can take a charge of two tonnes comprising about 1.5 tonnes of grinding media and 0.5 tonnes of cement. Hardened steel balls up to 50 mm diameter can be used, although steel rods 25 mm in diameter and 50 mm long have proved more efficient.

Sufficient reactivity can be achieved by grinding the ash for 30 minutes then placing the required amount of lime in the mill and intergrinding for a further hour.

Given the vagaries of electricity supply in most developing countries, the operation of the ball mill is probably the key element in the manufacturing process of lime-ash cements. For example the plant at Kurukshetra at one stage was only receiving 28 hours of electricity per week and alternative means of energy were being considered.

If it is assumed that it takes three hours grinding to produce one tonne of lime-ash cement, the power consumption, using a 20 hp (15 kW) motor, is 44.7 kWh/t.

The cost of packaging represents a substantial proportion of operating costs in small-scale cement production. To facilitate bag filling, the ball mill can be mounted on a frame (fig. 24) to enable the cement to be discharged into a hopper. In India cement is bagged in jute or gunny bags, which are either hand or machine sewn.

Cost estimates

Fixed capital — buildings	
Land $-400 \text{ m}^2 \otimes \frac{1}{2} \text{ m}$	800
Buildings — 150 m ² @ \$35/m (includes office, storage and undercover for the ball mill)	5 250
Total	6 050
Fixed capital — plant Ball mill and grinding media (20 hp (15 kW), 1.54 m diameter x 1.54 m long)	7 000
Rice husk incinerators, 6 off @ \$150	900
Lime kiln (2.5 t/d)	1 250
Weighing platform (150 kg capacity)	500
Packaging equipment	1 000

Laboratory equipment		1 250
Installation cost		250
Contingencies		500
	Sub Total	11 900
	Total fixed capital	18 700
	Per tonne	31.2

Bruce and Garg (52) have proposed that the fixed capital investment for a 25 t/d minicement plant would be 33.6 (1978 prices) per tonne, while for large-scale cement production, it is in excess of 100/tonne.

The annual operating costs for three lime:ash ratios are shown in table 24. The fixed and working capital (based on 45 days operation) is:

Lime:ash	Total fixed cost (\$)	Cost (\$/t)	
1:3	21 665	36.11	
1:2	21 858	36.43	
1:1	22 241	37.07	

For the 25 t/d mini-cement plant, the working capital requirement per tonne was 46.9 (1978 prices).

The cost of the husks in Table 24 is a nominal \$1/tonne. Operating costs based on the husks costing \$5 and \$10 per tonne are shown below:

Lime:ash	Husk cost (\$/t)	Operating cost (\$/t)
1:3	1.0	36.33
1:3	5.0	49.59
1:3	10.0	65.54
1:2	1.0	38.70
1:2	5.0	50.53
1:2	10.0	65.33
1:1	1.0	43.39
1:1	5.0	52.28
1:1	10.0	63.38

The cost of \$10/tonne is compatible with the husks being used as a fuel source. The price of cement in India is such that two-thirds of the production is sold at the Government regulated price and the remaining one-third is allowed to fluctuate according to market forces. At present it is assumed that lime-RHA cement will not be subject to government control and hence the selling price can be set at a level comparable with other masonry cements. If this can be assumed to be \$60 per tonne, then at a husk cost of \$10/tonne, none of the mixes is viable (with a reasonable degree of profit); i.e. where the husks can be sold for use as fuel, their use to produce cement is not economic.

At a cost of 1/tonne, the husk would need to be obtained from rice mills that are probably within a radius of 0.5 to 1 km of the cement plant. Above this cost, the rice husks can be transported some distance to the mill but with one tonne of husks occupying approximately 8 cubic metres, it is apparent that the economic distance would not be large, probably less than 5 km (depending on local conditions).

The plant capacity of 600 t/y is based on an 8-hour day; obviously by increasing the shifts the capacity could theoretically be increased to 1,800 tonnes per year. Such an increase, however, would require changes in the plant layout due to the need for materials storage and handling facilities. Also an output of 6 tonnes of cement per 24 hours could require the combustion of nearly 18 tonnes of husk (or 142 cubic metres). While the number of incinerators could be increased, the supply and collection of the husk pose problems that could influence the economics of the process, particularly considering that the daily husk production of most of the rice mills in developing countries is 4 tonnes or less.

The capacity of a 2 t/d plant could be doubled by increasing the number of incinerators to 10. Considering the market for masonry cement and transportation problems in rural areas, it is probable that 1,200 tonnes annual capacity is sufficient and compatible with the limitations of the plant outlined above.

TABLE 25

COST ANALYSIS FOR CONTROLLED PYROPROCESSING

	Lime:ash (1:3)			Lime:ash (1:2)			Li	me:ash (1	1:1)
nem	Quantity	Unit cost	Cost per tonne of cement	Quantity	Unit cost	Cost per tonne of cement	Quantity	Unit cost	Cost per tonne of cement
Limestone @ \$10/tonne	300	3 000	5.00	400	4 000	6.67	600	6 000	10.00
Coal @ \$30/tonne	60	1 800	3.00	80	2 400	4.00	120	3 600	6.00
Rice husks @ \$1/tonne	1 950	1 950	3.25	1 740	1 740	2.90	1 305	1 305	2.17
Power @ 4¢/kWh*	26 820	1 073	1.79	26 820	1 073	1.79	26 820	1 073	1.79
Salaries and wages†	****	3 900	6.50		3 900	6.50		3 900	6.50
Other consumables		300	0.50		300	0,50		300	0.50
Packaging**	-	3 000	5.00		3 000	5.00	2 2	3 000	5.00
Quality control ^{††}		600	1.00		600	1.00		600	1.00
Repairs and maintenance***	-	865	1.44		865	1.44		865	1.44
Depreciation [†] [†]		1 341	2.23		1 341	2.23		1 341	2.23
Insurance [‡]	da ana	187	0.31		187	0.31		187	0.31
Overhead ^{‡‡}	·** **	600	1.00		600	1.00	—	600	1.00
TOTAL	MP ROMAN	18 616	31.02		20 006	33.34		22 771	37.95
Interest‡‡‡		3 184	5.30	mittatio	3 213	5.35		3 269	5.44
Operating costs		21 800	36.33	20076	23 219	38.70		26 040	43.39
Cost/40 kg bag		1.45			1.55	(and the contract of		1.74	

NOTES: * Based on 44.7 kWh/t.

+ Based on 1 supervisor @ \$100/month.

1 skilled labourer @ \$75/month.

6 unskilled labourers @ \$1/day.

** Based on 20¢/bag, 40 kg/bag.

†† Based on \$1.00/tonne.

*** Based on 2.5% of land and building and 6% of plant.

††† Based on 2.5% of land and building and 10% of plant.

‡ Based on 1% of fixed capital.

tt Based on \$1.00/tonne.

‡‡‡ Based on 45 days working capital plus fixed capital @ 15%.

Boiler ash process

The boiler ash process requires no facility to burn the husk — although with the tendency to use heap-burnt ash rather than boiler ash this may not be true in all situations. Due to the fact that the grinding process takes substantially longer than when the ash is obtained from controlled pyroprocessing, the size of the ball mill is generally larger. As a result, the power consumed per tonne is also greater. For comparison purposes it is assumed that the rate is 135 kWh/t, a value which is comparable with the energy requirements for Portland cement production.

In using heap-burnt ash or ash which has been stored or dumped outside prior to grinding, it has been found that the moisture content of the husks combined with prolonged grinding periods (up to six hours) can result in the formation of steam in the ball mill. Unless a pressure relief valve is fitted to the ball mill, the possibility of an accident occurring arises. Also, unless the lime is underslaked, the moisture content of the ash would reduce the shelf life of the cement.

Cost estimates

Fixed capital — buildings	6 050
Fixed capital — plant Ball mill and grinding media	
(40 hp (30 kW), 2 m diameter x 2 m long)	10 000
Lime kiln $(2.5 t/d)$	1 250
Weighing platform (150 kg capacity)	500
Packaging equipment	1 000
Laboratory equipment	1 250
Installation costs	250
Contingencies	500
Total	14 750
Total fixed capital	20 800
Per tonne	34.7

The annual operating costs for three lime:ash ratios are shown in table 26. The fixed and working capital (based on 45 days operation) is:

Lime:ash	Total fixed cost (\$)	Cost (\$/t)	
1:3	23 966	39.94	
1:2	24 181	40.30	
1:1	24 612	41.02	

Lime:ash	Ash cost (\$/t)	Operating cost (\$/t)		
1:3	1	38.80		
1:3	5	41.86		
1:3	10	45.69		
1:2	1	41.44		
1:2	5	44.15		
1:2	10	47.56		
1:1	1	46.71		
1:1	5	48.75		
1:1	10	52.32		

The table below shows the influence of the cost of the RHA:

The variation in the ash cost can be likened to costs associated with transportation. It can be seen that the operating costs are less sensitive to the cost of the ash and the cost of the rice husk, as compared to the controlled pyroprocessing method. Further, it will be apparent that, based on a selling price of 60/t, all the combinations of lime and ash for all costs of ash would produce a reasonable return on the capital investment, as shown below. This is not the case for controlled pyroprocessing.

			Return on investment (%)		
Lime-ash	Husk cost (\$/t)	Ash cost (\$/t)	Controlled burning (%)	Boiler Ash (%)	
1:3	1.0	1.0	65.5	53.1	
1:3	5.0	5.0	28.8	45.4	
1:3	10.0	10.0	-15.3	35.8	
1:2	1.0	1.0	60.0	46.0	
1:2	5.0	5.0	26.0	39.3	
1:2	10.0	10.0	-14.6	30.9	
1:1	1.0	1.0	44.8	32.4	
1:1	5.0	5.0	20.8	27.4	
1:1	10.0	10.0	- 9.1	18.7	

The plant capacity is limited by the throughput of the ball mill and the associated materials handling problems. Since one tonne of ash occupies slightly in excess of three cubic metres, the problems are obviously less than when dealing with rice husks.

TABLE 26

COST ANALYSIS FOR THE BOILER ASH METHOD

ltom	A A CARACTER CONTRACTOR OF THE ACCORDENCE OF T	Lime:asl	n (1:3)	Lime:ash (1:2)			Li	Lime:ash (1:1)		
((4))	Quantity	Unit cost	Cost per tonne of cement	Quantity	Unit cost	Cost per tonne of cement	Quantity	Unit cost	Cost per tonne of cement	
Limestone @ \$10/tonne	300	3 000	5.00	400	4 000	6.67	600	6 000	10.00	
Coal @ \$30/tonne	60	1 800	3.00	80	2 400	4.00	120	3 600	6.00	
Rice husks @ \$1/tonne	450	450	0.75	400	4000	0.67	300	300	0.50	
Power @ 4¢/kWh*	81 000	3 240	5.40	81 000	3 240	5.40	81 000	3 240	5.40	
Salaries and wages [†]		3 900	6.50		3 900	6.50	(1 <u>111111</u> 1	3 900	6,50	
Other consumables		300	0.50		300	0.50		300	0.50	
Packaging**		3 000	5.00		3 000	5.00		3 000	5.00	
Quality control ^{††}		600	1.00		600	1.00	-	600	1.00	
Repairs and maintenance***		1 036	1.72		1 036	1.72	This are	1 036	1.72	
Depreciation ††		1 626	2.71		1 626	2.71		1 626	2.71	
Insurance‡		208	0.35		208	0.35		208	0.35	
Overhead‡‡		600	1.00		600	1.00		600	1.00	
TOTAL	-	19 760	32.93	where the set	21 310	35.51	in the second se	24 410	40.68	
Interest‡‡‡		3 523	5.87		3 554	5.92	1000 C	3 618	6.03	
Operating costs		23 283	38.80	- prymeryse	24 864	41.44		28 028	46.71	
Cost/40 kg bag		1.55			1.66	-		1.87		

NOTES: * Based on 135 kWh/t.

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Based on 1 supervisor @ \$100/month.

- 1 skilled labourer @ \$75/month.
- 6 unskilled labourers @ \$1/day.

** Based on 20¢/bag, 40 kg/bag.

†† Based on \$1.00/tonne.

*** Based on 2.5% of land and building and 6% of plant.

††† Based on 2.5% of land and building and 10% of plant.

† Based on 1% of fixed capital.

‡‡ Based on \$1.00/tonne.

‡‡‡ Based on 45 days working capital plus fixed capital @ 15%.

VIII. SPECIFICATION AND QUALITY CONTROL

Specification

RHA is a pozzolan and as such it can be made to conform to the requirements for both lime-pozzolan cements and Portland cement-pozzolan blends. Examples of relevant standards are ASTM C593 (Fly ash and other pozzolans for use with lime), ASTM C618 (Fly ash and raw or calcined natural pozzolans for use in Portland cement concrete), ASTM C595 (Standard specification for blended hydraulic cements), IS 1727 (Methods of test for pozzolanic materials), IS 1344 (Specification for burnt clay pozzolana) and IS 4098 (Specification for lime-pozzolana mixtures).

Although the reactivity of the ash can be evaluated using X-ray diffraction, particle size determinations and chemical testing, it is considered that assessment of lime-ash strength development is the most suitable technique. When the ash is mixed with Portland cement, the pozzolanic reaction is that between the ash and lime produced by the cement hydration and is similar to that in building lime-ash mixtures.

Reactivity tests with lime are specified in the standards mentioned above. These standards specify a 50 mm cube as the test specimen and the water content determined by the flow table test. The curing regime varies somewhat with the standard but with the ASTM and Indian standards IS 1727 and 1344, the philosophy is similar. That is, it is recognized that the pozzolanic reaction can be accelerated by hot water curing and that this accelerated strength is a useful guide. The curing cycles use hot water at 50°C and 55°C for the ASTM and Indian standards, respectively. The strength requirements for the various standards are shown in table 27.

TABLE 27

Standard	Curing Cycle	Test time (days)	Minimum Strer requirement (MPa)		ength Its
ASTM C593	7 days @ 55°C, then 23°C until test	7 28		4.1 4.1	
IS 1727-1344	2 days @ 27°C, then 8 days @ 50°C	5.0		5.0	
IS 4098	7 days @ 27°C, or 28 days @ 27°C		LP40	Type LP20	LP7
		7 28	2 4	1 2	0.3 0.7

COMPARISON OF STRENGTH REQUIREMENTS FOR POZZOLANS AS SPECIFIED BY ASTM AND INDIAN STANDARDS

The Indian standard IS 4098 relates to the requirements of the mortar rather than the characteristics of the pozzolan itself and is probably the most suitable standard currently available for evaluating lime-pozzolan cements. As such it has been adapted (see annex) for use as a specification for RHAM cement. While reference is made to the relevant Indian standards, the test methods are similar to those specified in other standards (for example, ASTM C91 standard specification for masonry cement) as are the requirements for the lime and pozzolan.

Quality control

Even though cements based on RHA can be produced at the village level, it is essential that the product be relatively consistent from sample to sample. Otherwise large variations in setting time and strength that result in delays in construction and even failures, will generate consumer resistance.

In controlled pyroprocessing, the incineration of the husk must be monitored: this can simply be done using a pyrometer. With experience visual checks can be developed, but they should be considered as nothing more than that.

The quality of the building lime is also important. Although test methods are available to ensure compliance with standards, such expertise and equipment will probably not be available at the village level. For example, Boynton and Gutschick (53) cite seven properties that can be evaluated to show that the lime is of good quality: particle size, specific surface area, shape of particle, putty volume, water retention, settling rate and plasticity. Such properties, however, are not easily measured.

The prime requirement of the lime is that it be chemically reactive for combining with the silica in the ash. The simplest way this can be checked is by measuring the compressive strength of a lime-ash mortar mix and comparing the strength with that of a mix whose lime and ash characteristics are known. Poorly stored lime will contain carbonated material and its reactivity will be impaired. Over-burnt lime will also have reduced reactivity which can probably be detected during slaking. Under-burnt lime is more acceptable than hard burnt, but it will contain a core of uncalcined carbonate. Under-burning obviously reduces the yield of hydrated lime and if the core remains, the overall reactivity of the lime will be reduced. The presence of unhydrated particles in the lime, resulting in pitting and popping, is due to the presence of magnesium and other impurities in the limestone. This characteristic of the lime, known as unsoundness, can be evaluated using the pat test or the Le Chatelier method. Procedures for these tests are given in specifications for building lime.

The use of a weighing platform enables lime (or Portland cement) and ash quantities to be accurately weighed. Simple calibration checks should be made from time to time.

Routine quality control tests must be carried out on the cement produced. It is suggested that this should involve both in-house testing and testing of samples at a central testing facility. An allowance of \$1 per tonne in the operating costs has been foreseen for this purpose. In-house testing should involve strength testing and measurement of setting time. The latter can be simply accomplished using the Vicat apparatus and standard procedures, but the former could represent significant additional cost. To achieve a minimum strength of 5 MPa using a 50 mm cube would require a testing machine with a capacity of at least 1,250 kg. However, for a 25 mm cube the capacity is reduced to 325 kg so that a small testing machine with a capacity of 2 tonnes would be more than adequate. This can be achieved with a simple hand-pumped hydraulic arrangement in a steel frame. Spence and Allen (54) have built such a machine in the United Kingdom for around \$1,000 but it is likely that the cost would be reduced if it were built in developing countries. It should be noted that if 25 mm cubes are used instead of the standard 50 mm, six speciments should be tested rather than three because of the greater variation in strength inherent with small specimens.

The checklist of laboratory equipment required for quality control is as follows:

pyrometer (not required if boiler or heap ash is used) simple beam balance Vicat needle cube moulds water bath and compression testing machine. In batch production it is not possible to sample each batch. It would also be incorrect to collect material from different batches and mix them to form an average sample. Ideally, a random sample should be selected about once every five days (or every 10 tonnes of cement) for in-house testing and about once every four weeks (or every 100 tonnes) for more extensive laboratory testing. It is recognized that this degree of quality control is rather extensive, but considering the vulnerability of small-scale processing to produce variability, it is considered necessary.

Marketing

The bulk density of cements based on RHA is lower than that of conventional cements. In India, for example, ash cements are currently sold in 40 kg amounts in second-hand 50 kg Portland cement bags. Since most mixes incorporating RHA cements are batched on a volume basis, it is important that the end user adjust the mix quantities to ensure that the correct amount of cement is used. This can be ensured by distributing leaflets on mix proportions with each bag and by field demonstrations. Problems with illiteracy in rural areas obviously limit the value of printed information.

Low-reactivity ashes, particularly those derived from husk burnt as boiler fuel, produce cements that not only have low strength but also have long setting times. When used for mortar or plaster the strength aspect is usually more than adequate, but the time to final set can cause consumer concern. However, as Narula (55) *et al* have shown, if the long-term strength and durability of the cement can be demonstrated in the field, then lack of hardness in the first 24 hours does not present a serious problem.

The cement in general is darker than Portland cement and can in some instances be almost black. The colour relates to the pyroprocessing conditions, ash obtained from controlled pyroprocessing being ligher in colour than that from husk burnt as fuel. The colour of the mortar is reflected in the plaster and brick mortar shown in fig. 40. There appears to have been little consumer resistance to the colour of the cement. Indeed, where a pigmented concrete has been required, for example, in glare-free flooring, the black colour of the cement has been an added sales feature.

The excellent plasticity of mortar mixes containing RHA cement has considerably enhanced market acceptance; the availability of a standard on RHAM cement has reinforced consumer confidence. Not surprisingly problems have occurred due to variations in the quality of the cement; this above all presents the greatest marketing problems for the small-scale producer.



Figure 40. Plaster and brick mortar using Ashmoh cement (Kurakshetra, India)

IX. SUMMARY AND CONCLUSION

It will be apparent that there are both technical and economic differences between the two processes currently available for producing RHA cements. The first method, controlled pyroprocessing, relies on the combustion of the husk under specified conditions to produce an essentially amorphous silica ash. The pyroprocessing parameters, viz. temperature of combustion, duration and combustion environment, vary with the incinerator design. However, in general terms, combustion at 600°C to 650°C for between two and three hours will produce amorphous ash. It should be noted that low combustion temperatures and short duration times can result in excessive carbon in the ash that reduces the ash reactivity. On the other hand, high temperatures and long times will produce crystalline and hence low reactivity ash.

Ash produced under controlled conditions can be readily comminuted by grinding. However, ash obtained from husk burnt as fuel, the basis of the second method, is hard and takes long periods to grind to a suitable particle size. The grinding time does not significantly influence the cost of the cement but does affect the plant throughput and capital investment requirements. However, as many developing countries are faced with the problem of intermittent electricity supply particularly in rural areas, it is desirable to have as short a grinding time as possible. The alternative of having an independent source of energy for grinding such as a diesel engine would markedly reduce the economic viability of the cement.

Indicative cost estimates reveal that the controlled pyroprocessing method is sensitive to the cost of the husks. Husks transported over long distances or with more than a nominal cash value, reduce the economic viability of the method. On the other hand, the technology based on boiler ash is relatively insensitive to the cost of the ash.

Laboratory investigations and limited results from field testing suggest that a lime:ash ratio of 1:2 is optimal from a strength and durability point of view. For Portland-ash blended cements, it is recommended that the Portland cement replacement be not more than 40 per cent. Mix proportions should be formulated on the basis of consistency and strength requirements. It should be recognized, however, that cements based on RHA require more water than other cements to produce the same consistency. Also the bulk density of RHA cements is lower than that of conventional cements. While this increases the yield of the mix, it requires adjustments to the batching procedures that are commonly done on a volume basis.

Perhaps the biggest problem facing the manufacture of RHA cements is that of quality control. Access to laboratory facilities in most cases will be limited or non-existent and costs of laboratory equipment may be considered disproportionate to the capacity of the plant. However, variable cement properties will generate consumer resistance from the artisans who use it and any failures would obviously discourage the community at large, particularly in a rural environment. It is suggested that testing involve measurements of setting time, both initial and final, set soundness and compressive strength. The latter could be carried out on small specimens in handpumped hydraulic testing machines; or allowance should be made in the operating costs to send samples to a testing laboratory on a regular basis.

Finally, the choice of either of the methods for producing RHA cements will depend almost entirely on local conditions. Each location for a plant needs to be carefully evaluated in terms of husk or ash availability, other raw materials, electricity, land, etc. The plant design must take into account the operating conditions, the lime kiln operation, the incinerator design and pyroprocessing parameters (if applicable), the grinding and bagging operation, while at the same time maintaining an efficient flow of material through the plant. To maintain the development philosophy of providing a binder for the rural sector, it is suggested that a plant capacity of between 600 to 1,200 t/y is sufficient.

While this report gives an overview of existing technologies, it is hoped that it has also provided a clear mandate not only for further development but also for the viability of cement based on RHA.

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ANNEX

Specification for rice-husk ash masonry (RHAM) cement

SPECIFICATION FOR RICE-HUSK ASH MASONRY CEMENT

1 Foreword

1.1 Rice-husk ash masonry cement (RHAM) whose essential components are ricehusk ash and lime can be used for work like masonry mortar and plaster, concrete floors and lime concrete in foundations.

2 Scope

2.1 This standard covers the requirements of RHAM cement for use in general masonry works.

3 Terminology

3.1 Rice-husk ash (RHA) — a highly siliceous and easily grindable residue that is obtainable through combustion/incineration of rice-husk.

4 Manufacture of the RHAM cement

4.1 Rice-husk ash masonry cement shall be manufactured either by intergrinding lime and rice-husk ash, or by blending the two ingredients of requirements fineness adopting suitable measures to ensure a homogeneous mixture.

4.2 A small quantity of additives such as mineral gypsum, surkhi, ordinary Portland cement may be used.

5 Materials

5.1 Rice-husk ash shall conform to the requirement for pozzolans laid down in IS:1344-1959 Part I.

5.2 The lime shall be dry-hydrated lime satisfying the requirements for class C lime specified in IS:712–1964.

6 Requirements

6.1 RHAM cement when tested in accordance with the methods of test indicated, shall conform to the requirements given in table A.

7 Sampling

7.1 Samples for testing and by whom to be taken: A sample or samples for testing may be taken by the purchaser or his authorised representative or any other person appointed to superintend the work for the purpose of which the RHAM is required.

7.2 Sample size: When samples are taken for test purpose, each shall weigh at least 2 kg. Individual test samples on which all tests are to be conducted shall weigh at least 5 kg.

7.3 Procedure for sampling: RHAM cement samples shall be obtained by the methods specified in 7.3.1.

7.3.1 Sampling small quantities: Each sample for testing shall consist of a mixture of approximately equal proportions selected from at least 12 different positions in the heap or heaps when the RHAM cement is loose or from not less than 12 different bags or other packages when the mixture is not loose. When the number of bags or packages is less than 12, portions shall be selected from each bag or package. Every care shall be taken during the selection to

obtain a fair average sample. The final sample shall weigh at least 5 kg. The sample thus collected shall be stored in an air-tight container to be forwarded to an approved laboratory.

7.3.2 Sampling large quantities: From large quantities, samples collected by small increments shall be rapidly mixed in a place free from draught, and thereafter stored in air-tight containers of 7 kg capacity. In case of dispute, at least two such containers shall be filled, one sent to an approved laboratory and the other kept as a reference sample by the supplier.

7.3.3 In all other cases, samples shall be taken from each 50 bags or portion thereof in the lot and mixed to form test samples. In the case of samples from trucks where the mixture is being trucked from one mill, it is permissible to combine the samples from several trucks to form a test sample each representing not more than 1,000 bags.

7.3.4 Facilities for sampling and identifying: The manufacturer or supplier shall afford every facility, and shall provide all labour and materials for taking and packing the samples for testing the RHAM cement and for subsequent identification of the cement sampled.

8 Tests

8.1 The sample or samples of RHAM cement for tests shall be taken as described in 7.3.1 to 7.3.3 and shall be tested in the manner described in Section 6.

8.2 Temperature of testing: The temperature range within which the physical tests may be carried out should, as far as possible, be $27^{\circ} \pm 2^{\circ}$ C.

8.3 Non-compliance with tests: Any RHAM cement, which does not comply with any of the tests specified above, or which has not complied with this standard.

8.4 Independent testing

8.4.1 If the purchaser or its representative requires an independent test, the samples shall be taken before or immediately after delivery at the option of the purchaser or its representative, and the tests shall be carried out in accordance with this standard or the written instructions of the purchaser or its representative.

8.4.2 Cost of testing: The manufacturer shall supply, free of charge, the mixture required for testing. Unless otherwise specified in the enquiry and order, the cost of the tests shall be borne as follows:

(a) By the manufacturer in the event of the results showing that the RHAM cement does not comply with this standard, and

(b) By the purchaser in the event of the results showing that the RHAM cement complies with this standard.

8.4.3 After a representative sample has been drawn, tests on the sample shall be carried out as expeditiously as possible.

9 Storage

9.1 The RHAM cement shall be stored in such a manner as to permit easy access for proper inspection and in a suitable building to protect the mixture from dampness and to minimise storage deterioration.

10 Manufacturer's certificate

10.1 The manufacturer shall be satisfied that the RHAM cement conforms to the requirements of this standard, and if requested, shall furnish a certificate to this effect to the purchaser or his representative.

TABLE A

REQUIREMENTS OF RHAM CEMENT (Clause 6.1)

CHARACTERISTIC	REQUIREMENTS	REFERENCE TO METHOD OF TEST
1. Free moisture, per cent	5	Appendix A
2. Loss on ignition, percent, Max.	20	IS:1727-1960 (ASTM C311)
 Fineness, per cent, retained on 150 μm IS Sieve 	10	Appendix B
4. Setting time(by Vicat apparatus)a) Initial, min. (h)b) Final, max. (h)	2 24	IS:1727-1960 (ASTM C191)
 5. Compressive strength: Average compressive strength of not less than 3 mortar cubes of size 50 mm composed of one part of lime- pozzolan mixture and 3 parts of standard sand by volume, MPa a) 7 days, min. b) 28 days, min. 	$2.0 \\ 4.0$	Appendix C
6. Water retention-flow after suction of mortar composed of one part of lime-pozzolan mixture and 3 parts of standard sand by volume, per cent of original flow min	70	Appendix C of IS:2250-1965 (ASTM C91)
7. Soundness, Le Chatelier expansion, not more than (mm)	10	IS:1727-1960 (BS4550)

11 Delivery

11.1 The RHAM cement shall be packed in bags (gunny, multi-ply paper, cloth or other suitable packing materials) bearing the manufacturer's name or his registered trade mark.

11.1.1 The bags or packages may also be marked with the certification mark of the relevant Standard institution.

11.2 The net weight of each bag shall be clearly stated. The permissible tolerance for the weight of RHAM cement supplied in bags shall be ± 2.5 per cent per bag with an overall tolerance of \pm 0.5 per cent per load of 10–15 t.

APPENDIX A

(TABLE 1, ITEM 1.) Test method for moisture content of RHAM Cement

A-1 Procedure

A-1.1 A weighted sample of RHAM cement shall be dried to constant weight in an oven at 105° to 110°C.

A-2 Calculation

A-2.1 The percentage of free moisture shall be calculated to the nearest 0.1 as follows:

Moisture content, per cent = $\frac{A \times 100}{B}$

where

A = loss of weight after drying, and

B = weight of sample taken originally.

APPENDIX B

(TABLE 1, ITEM 5.) Fineness by the wet sieving method

B-1 Fineness by wet sieving

B-1.1 100 g of the sample of RHAM cement previously dried to constant weight in an oven at 100° to 105°C shall be washed through the sieve by means of a stream of water from a faucet. A piece of rubber tubing attached to the water faucet shall be used for the washing. The velocity of the water may be increased by pinching the tube, but shall not be sufficient to cause any splashing of the sample over the sides of the sieve. The washing shall be continued until the water coming through the sieve is clear, but in no case shall the washing be continued for more than 50 min. Care shall be taken not to let water accumulate on the sieve, in the event that the openings become clogged. The residue on the sieve shall be dried to constant weight at a temperature between 100° and 110°C. The percentage residue retained on the sieve shall be calculated, based on the original weight of the dry sample.

APPENDIX C

(TABLE 1, ITEM 5.)

Test method for determining compressive strength of RHAM Cement

C-1 Procedure

C-1.1 The method detailed below shall be used to determine the compressive strength of RHAM cement.

C-2 Apparatus

C-2.1 Scales: the scales used in weighing materials shall conform to the following requirements:

On scales in use, the possible variation at a load of 2,000 g shall be \pm 2.0 g. The permissible variation on new scales shall be one-half of this value. The sensitivity reciprocal shall be not greater than twice the permissible variation.

C-2.2 Cube moulds: the test specimens shall be cubes of size 50 mm and other requirements of the moulds shall conform to section 4.2 of IS:269-1958.

C-2.3 Mixing apparatus: the mixing apparatus shall conform to the requirements specified in IS:1727-1960.

C-2.4 Flow table and accessories: the flow table apparatus shall conform to the requirements specified in IS:1727-1960.

C-2.5 Tamper: this shall be made of non-absorptive, non-abrasive, non-brittle material, such as rubber compound having a Shore A durometer hardness of 80 ± 10 or seasoned wood rendered non-absorptive by immersing for 15 min in paraffin at approximately 200°C, and shall have a cross-section of 12 x 25 mm and a convenient length 125 to 150 mm. The tamping face shall be flat and at right angles to the length of the tamper.

C-2.6 Trowel: this shall have a steel blade 100 to 150 mm in length with straight edge.

C-3 Preparation of moulds

C-3.1 The interior faces of the specimen moulds shall be thinly covered with mineral oil or light cup grease. After assembling the moulds, excessive oil or grease shall be removed from the interior faces and the top and bottom surfaces of each mould. Moulds shall then be set on plane, non-absorbent base plates that have been thinly coated with mineral oil, petroleum, or light cup grease.

C-4 Preparation of mortar

C-4.1 The sand used for preparation of mortar cube specimens shall conform to IS:1966. The proportion of material shall be one part of RHAM Cement and three parts of standard sand on an absolute volume basis. The mortar shall be mixed in the mixing apparatus and the flow determined as specified in IS:727-1960. The quantity of water shall be such as to produce a flow of 110 ± 5 as determined by the flow table. A minimum of three specimens shall be made for each period of test specified.

C-5 Moulding of specimens

C-5.1 Immediately following completion of the flow test, return the mortar from the flow mould to the mixing bowl. Quickly scrape down into the batch the mortar that may have collected on the side of the bowl and give the entire batch a 15 s mixing at medium speed. Start moulding the specimens within a total elapsed time of not more than two minutes and 15 s after completion of the original mixing of the mortar batch. Place a layer of mortar about 25 mm in thickness in all the cube compartments. Tamp the mortar in each cube compartment 32 times in about 10 s in four rounds, each round to be at right angles to the other and consisting of eight adjoining strokes over the surface of the specimen as illustrated in fig. A. The tamping pressure shall be just sufficient to ensure uniform filling of the moulds. The four rounds of tamping (32 strokes) of the mortar shall be completed in one cube before going to the next. When the tamping of the first layer in all of the cube compartments is completed, fill the compartments with the remaining mortar and then tamp as specified for the first layer. During the tamping of the second layer bring in the mortar forced out on to the tops of the moulds after each round of tamping by means of gloved fingers and the tamper upon completion of each round and before starting the next round of tamping. On completion of the tamping, the tops of all cubes should extend slightly above the tops of the moulds. Bring in the mortar that has been forced out on to the tops of the moulds with a trowel and smooth off the cubes by drawing the flat side of the trowel (with the leading edge slightly raised) once across the top of each cube at right angles to the length of the mould. Then for the purpose of levelling the mortar and making the mortar that protrudes above the top of the mould of more uniform thickness, draw the flat side of the trowel (with the leading edge slightly raised) lightly once along the length of the mould. Cut off the mortar to a plane surface flush with the top of the mould by drawing the straight edge of the trowel (held nearly perpendicular to the mould) with a sawing motion over the length of the mould.

Note: When a duplicate batch is to be made immediately for additional specimens, the flow test may be omitted and the mortar allowed to stand in the mixing bowl for 90 s and then remixed for 15 s at medium speed before starting the moulding of the specimens.

C-6 Storage and curing of specimens

C-6.1 All test specimens, immediately after moulding, shall be kept in the moulds on plane plates in a moist cabinet, maintained at a temperature of $27^{\circ} \pm 2^{\circ}$ C and a relative humidity of 90 per cent or more, from 48 to 52 h in such a manner that the upper surface shall be exposed to the moist air. The cubes shall then be removed from the moulds and placed in the moist cabinet for seven days in such a manner as to allow free circulation of air round at least five faces of the specimens. At the end of seven days the cubes for the 28-day tests shall be immersed in clean water in storage tanks of non-corrodible materials.

C-7 Testing

C-7.1 Test not less than three cubes for compressive strength for each of the periods of 7 and 28 days, the period being reckoned from the completion of moulding and compaction. The compressive strength shall be the average of the strengths of the three cubes for each period respectively.



C-7.2 Testing of the cube specimens shall be carried out immediately after their removal from the moist cabinet for 7-day specimens, and from storage water for all other specimens. If more than one specimen at a time is removed from the moist closet for 7-day tests, these cubes shall be covered with a damp cloth until the time of testing. If more than one specimen at a time is removed from the storage water for testing, these cubes shall be placed in a pan of water at a temperature of $27^{\circ} \pm 2^{\circ}$ C and of sufficient depth to completely immerse each cube until the time of testing.

C-7.3 The cubes shall be tested on their sides without any packing between the cubes and the steel plattens of the testing machine. One of the plattens shall be carried on a base and shall be self-adjusting. An initial loading up to one-half of the expected maximum load for specimens having expected maximum loads of more than 1,350 kg may be applied at any convenient rate. Apply no initial loading to specimens having expected maximum loads of less than 1,350 kg. Adjust the rate of load application so that the remainder of the load (or the entire load in the case of expected maximum load of less than 1,350 kg) is applied, without interruption, to failure at such a rate that the maximum load will be reached in not less than 20 nor more than 80 seconds. Make no adjustment to the controls of the testing machine while a specimen is fracturing immediately before failure.

C-8 Calculations

C-8.1 Calculate the compressive strength from the maximum load indicated by the testing machine and the average area over which the load is applied. Express the result in MPa. The results should be averaged and reported to the nearest 0.1 MPa.

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