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UNITED NATIONS INDUSTRIAL DEVELOPMENT ORGANIZATION

ESTABLISHMENT OF A CERAMIC

RESEARCH AND DEVELOPMENT LABORATORY

US/SRL/78/207

SRI LANKA

<u>Technical report: Mineralogical</u> investigation of non-metallic minerals

Prepared for the Government of Sri Lanka by the United Nations Industrial Development Organization

> Based on the work of I. M. Sacher, consultant in mineralogy and microscopy

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Explanatory notes

In addition to the common abbreviations, symbols and terms, the following have been used in this report:

SEM scanning electron microscope

XRF X-ray fluorescence

Mention of firms and commercial products does not imply endorsement by the United Nations Industrial Development Organization (UNIDO).

ABSTRACT

A consultant in mineralogy and microscopy was sent to Colombo for the period 14 January to 5 July 1985, as part of the ongoing project "Establishment of a ceramic research and development laboratory in Sri Lanka" (US/SRL/78/207) for which the United Nations Industrial Development Organization (UNIDD) is the executing agency.

The purpose of the consultant's mission was to assist the Ceylon Ceramics Corporation in setting up its Central Ceramic Research and Development Laboratory by carrying out mineralogical investigations of raw materials and products and giving mineralogical training to the research officers.

The consultant carried out a comprehensive assessment of the existing equipment in the laboratory, demonstrated the proper use of the equipment for mineralogical analyses and attempted to integrate the equipment in the overall work of the laboratory. Although the present range of equipment for mineralogical analyses is rather wide and comprises a number of highly sophisticated instruments, specifications were also put forward for additional equipment that is still required to enhance the capacity of the laboratory to serve its clients.

The consultant also presented possibilities for improving the operation of the laboratory, including new procedures for the preparation and measurement of samples.

The consultant further recommended several new procedures that could help the Ceylon Ceramics Corporation to maintain or improve the quality of its products.

Finally, the consultant introduced a number of subjects for post-graduate work by the laboratory staff, in particular the use of rice husk ash in various ceramic applications and the development of a wider range of refractories.

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I.

INTRODUCTION

A consultant in mineralogy and microscopy was sent to Colombo for the period 14 January to 5 July 1985, as part of the ongoing project "Establishment of a ceramic research and development laboratory in Sri Lanka" (US/SRL/78/207) for which the United Nations Industrial Development Organization (UNIDD) is the executing agency.

According to her job description, the expert was attached to the Ceylon Ceramics Corporation to assist in setting up its Central Ceramic Research and Development Laboratory. The Ceylon Ceramics Corporation was established in 1935 and the first industrial manufacture initiated in 1939 with the start of an earthenware factory in Ngombo. Since then, the Corporation has undergone rapid development, partly on its own, and partly in joint ventures with Japanese ceramic producers.

However, apart from the limited work that can be carried out in the quality-control laboratories of individual plants, the Corporation is still dependent on foreign laboratories for more specialized development work. The Central Ceramic Research and Development Laboratory was set up not only to serve the Corporation itself, but also to contribute to the development of the country's ceramic industry in general.

The expert was expected to work in close collaboration with local counterpart staff at the laboratory and the manufacturing plants and was specifically required to:

 (a) Organize a mineralogical training and orientation course relating to raw materials and various products for local staff;

(b) Initiate procedures for testing the mineralogical properties of the local materials available as well as the texture of products:

(c) Set up a systematic research programme including the microscopic study of raw materials and products;

(d) Define and arrange necessary equipment related to the microscopic work for raw materials.

The consultant gave a series of lectures and practical courses on mineralogy and crystallography for the research officers at the laboratory, but regretted that the officers were not allowed more time to attend regularly due to pressure of work.

The consultant carried but a comprehensive assessment of the existing equipment in the laboratory, demonstrated the proper use of the equipment for mineralogical analyses and attempted to integrate the equipment in the overall work of the laboratory. Although the present range of equipment for mineralogical analyses is rather wide and comprises a number of highly sophisticated instruments, specifications were also put forward for additional equipment that is still required to enhance the capacity of the laboratory to serve its clients.

The consultant suggested improvements in the conditions for the preparation and density determination of samples in the laboratory. It was also recommended that samples be stored so that additional investigations are possible, including the future control of results.

The Ceylon Ceramic Corporation was urged to start investigations of both its own and competitors' products in order to improve the Corporation's products. The introduction of sampling of raw materials and the use of rational analysis should also help in quality control.

Finally, the consultant outlined a number of research activities, including the use of paddy husk ash in ceramics (a project already initiated), the preparation of magnesite from seawater and the production of acid-resistant bricks.

RECOMMENDATIONS

To UNIDO

1. A mineralogical research microscope for transmitted and incident light should be purchased to replace the existing polarizing microscope, which is unsuitable for mineralogical work.

2. A grinding and polishing machine should be purchased to facilitate the preparation of samples for examination by the polarizing and scanning electron microscopes.

3. An energy-dispersive X-ray spectrometer and beam controller are required for elemental analysis of specimens.

4. An ultrasonic bath is required to perform the requisite six-monthly cleaning of parts of the scanning electron microscope and to prepare polished and thin sections.

To the Central Ceramic Research and Development Laboratory

5. A separate workshop should be installed for the lathe, large diamond saw, vibrating mill and old grinding and polishin; machine, so that sample preparation for the polarizing and scanning electron microscopes can be done in a clean environment.

5. More personnel are required to cope with sample preparation for other departments.

7. A fume cupboard should be installed in the balance room for work involving mercury, density determinations and etching of polished sections.

8. A separate kiln room should be constructed from half of the room adjoining the chemical laboratory to fuse samples to be investigated using the X-ray spectrometer.

9. A room should be designated for storing or depositing samples in air-tight containers, so that future control of results and additional investigations are possible.

To the Ceylon Ceramic Corporation

10. The Corporation should investigate competitors' products in order to improve its own products.

11. The Corporation should also investigate its own new, good and defective products and its used products after both short and long lifetimes.

12. Raw materials, both local and imported, should be sampled and analysed to help maintain high quality products. A report should be written for each sample and instructional leaflets prepared for each raw material. 13. The technique of rational analysis should be introduced as a very fast method of quality control.

14. A research topic should be initiated to study the use of paddy husk ash in various ceramic applications and to develop a wide range of refractories.

15. Because of difficulties with variations in the quality of imported magnesite, a study should be made of the feasibility of obtaining magnesite locally from seawater.

16. A study should be made to determine whether there is a market for acid-resistant bricks in Sri Lanka, since the raw materials for such bricks are available locally.

I. EQUIPMENT ASSESSMENT

A detailed assessment was made of equipment in the Central Ceramic Research and Development Laboratory at Piliyandala, Sri Lanka. Recommendations are put forward for the purchase of additional instruments and apparatus that are required to enhance the capacity of the laboratory to serve its clients.

A. Polarizing microscope

A good mineralogical polarizing microscope for polished and thin sections is essential for a ceramic research laboratory. The polarizing microscope in the laboratory at Filiyandala, Sri Lanka, is from Fisher Scientific. It is typically found in chemical laboratories but is not wellsuited for mineralogical work as the necessary accessories are not available.

Polarizing microscopes with good optics can be used up to a mangification of 1,250x. Higher magnifications are not practicable due to decreases in resolution resulting from the wavelength of visible light. Magnifications in the range 400x-1,000x are very often required for ceramics, which generally have small crystals. The magnification range of the Fisher Scientific microscope is 20x-400x, which is only sufficient for looking at thin sections or powder samples. Although the microscope has a polarizer, an analyzer and even a Bertrand lens for conoscopic pictures, the maximum magnification of 400x and the low quality optics produce only poor pictures for ceramic samples. The microscope does have a revolving circular stage but no mechanical stage is available for systematic movement of the specimen in the x and yco-ordinates. Such stages are normally present even on the smallest mineralogical microscopes for students in their first semester. Accessories for incident-light work and microphotography, which are essential for ceramics, are also not available.

It is recommended that a mineralogical research microscope for transmitted and incident light be purchased, such as the Photomicroscope III Pol from Zeiss. This microscope has a very sturdy stand with Bertrand system and slots for analyzer, retardation plates and compensators. It has a fully integrated automatic 35 mm camera with highly sensitive exposure measurements. A photomultiplier can be switched from the entire surface area to a selected image area to give accurate exposure measurements using an integral sensor for automatic exposure control with microflash, in base control electronics etc.

The microscope is particularly suitable for cathode-ray tube image analysis and microscope photometry (measurement of transmittance, reflectance, absorbance, fluorescence intensity etc.). It contains a pushrod with prisms and beam splitter, allowing the following settings:

(a) All light relayed to the observer:

(b) One-third of the light directed to the observer and two-thirds upwards;

(c) One-half of the light entering the camera (a small amount is used for the exposure meter), the other half relayed to the observer, who sees the part of the image that is recorded through the binocular tube;

(d) All light relayed upwards.

The microscope can be fitted with a universal rotary stage, with different types of phase-control, differential interference contrast, different interference systems and rarkfield illumination.

B. <u>Stereomicroscope</u>

The stereomicroscope in the laboratory is poor, with a small depth of focus and magnification range of only $20\times-40\times$. Good quality stereomicroscopes now cover a magnification range of $2\times-200\times$ with a zoom system and can often be used with a camera. Although a better stereomicroscope is desirable, it does not have as high a priority as the polarizing microscope.

C. Scanning electron microscope with photo equipment

A Jeol scanning electron microscope (SEM) (JSM-T100) was installed at the beginning of February 1985. The installation engineer, who specialized in transmission electron microscopes, could only stay for five days but gave what instruction he could. The microscope worked, but the camera was faulty. A second camera had the same problem and although Jeol was informed no answer was received. It is also necessary to clean the entire optical system of the microscope after six months' use but the installation engineer did not know the procedure. However, Mr. Karunasinghe will be trained in this procedure for one month at Jeol, in Japan, and will then spend a further month at the Tokyo Institute of Technology.

With the equipment available, it is currently only possible to investigate structures and take photographs of samples. Film is being wasted, as the camera has a faulty film transport and more film must be used to obtain satisfactory photographs.

An energy dispersive X-ray spectrometer and its beam controller are urgently needed for elemental analysis of specimens. Also needed are a backscattered electron detector, an absorbed current meter and various items for using the SEM correctly. Recommendations regarding the purchase of further equipment, such as a transmitted electron detector and a cathodo-luminescence detector, have been postponed, pending consultations with European laboratories working with SEMs.

D. Refractive index of liquids set

The certified refractive index of liquids set from Cargille (set No. 40-8252, consisting of 76 flasks with 4 test glasses and 1 thermometer) is of good quality and is used to determine refractive indices of coarse powder samples. With a better polarizing microscope, it would also be possible to observe powder samples with smaller grain sizes.

The range of refractive indices (n_0) covered is 1.400-1.7000, the interval between the liquids being 0.004. It does not include silicas formed under high pressure, such as stischovite $(n_0 = 1.799, n_E = 1.826)$. It also does not include corundum $(\alpha-Al_2D_3, n_0 = 1.770, n_E = 1.762)$, zircon $(n_0 = 1.94, n_E = 1.99)$, and kyanite $(n_\alpha = 1.717, n_\beta = 1.722, n_\gamma = 1.729)$, but it should be adequate for many uses in ceramics. It may be necessary later to buy the set 40-8262 from Cargille: 20 liquids, interval 0.005, refractive index range 1.705-1.806.

E. Dispensing micrometer with special stand

This Desaga instrument was donated to the laboratory. As soon as polished sections can be observed under the polarizing microscope, it may be possible to use the microlitre syringes for etching polished sections. However, cheaper items could also be used for this purpose. The device could be useful for determining the amounts of organic compounds in ceramic raw materials by thin-layer and paper chromatography. Thus it might be better to keep it until these types of investigations are done in Piliyandala. Chromatography should not be attempted until all the other apparatus in the mineralogical laboratory functions properly, including the polarizing microscope. However, the investigation of organic compounds will become important, especially for clays, and is common practice in ceramic research institutes. The micrometer might also be useful for soil investigations.

F. Microtome

Although special ultramicrotomes can be used in mineralogy, the Jung universal microtome autocut 1150 in the laboratory is unsuitable, the name "universal microtome" being misleading. The instrument might be useful for organic materials but probably cannot be reconstructed for use in ceramic research.

The only firm known to manufacture microtomes for mineralogy is Leitz Wetzlar, which manufactures the ultramicrotome according to Fernandéz-Morán with diamond knives. It can be used, for example, to cut kaolinite embedded in artificial resin. These kaolinite samples are very good for investigations with the transmission electron microscope.

A microtome can also be used for preparing very good surfaces for incident light examinations with a polarizing

microscope. The surfaces are better than those that can be obtained presently with a grinding and polishing process, but only for materials with a Vickers hardness less than 200 kp/mm2. At present, such an instrument is probably not useful for the laboratory. It is designed for basic research at a very high level, not for applied research.

G. Sawing machine with two diamond wheels

The Netzsch all-purpose sawing machine with two metalbound diamond wheels works satisfactorily. However for very small samples an additional diamond saw of high accuracy, such as the Buehler Isomet, should be purchased, taking into consideration the available grinding and polishing equipment.

H. Grinding and polishing machine

The surface structure of specimens is changed by mechanical polishing methods, due to plastic flow. There is a cold working process resulting in surface smoothening. A smudge layer, the so-called Beilby layer, is composed of debris with crystal size 0.5-1 nm, containing, for example, some oxides. Thus a method of polishing must be chosen where the Beilby layer is as small as possible. This is very important for both the polarizing microscope and the SEM. The SEM is currently only being used to investigate the structure of broken surfaces. However, smooth surfaces are required for X-ray analyses.

The equipment in the laboratory is inadequate for preparing the necessary surfaces for either microscope.

Many unsuccessful attempts were made to obtain polished sections of the required quality, including the older method of grinding with timber discs. However, difficulties with obtaining appropriate wood and fine uniform grinding material, together with the time and labour consuming nature of this method, conspire to make it unsuitable for regular use.

Modern methods of making thin and polished sections must therefore be used, such as with diamond paste. However, it was not possible to make a final recommendation on which instrument to purchase without testing their individual capabilities.

I. Lathe

A mechanical bench lathe from Boley with a recently acquired set of tools functions satisfactorily. More such tools would be useful but this piece of equipment is not used in mineralogy.

J. <u>Ultrasonic bath</u>

An ultrasonic bath with inner dimensions of approximately 20 x 30 x 20 cm is urgently required for cleaning parts of the electron microscope and preparing polished and thin sections.

K. Further improvements

The following instruments, apparatus and techniques would improve the capabilities and performance of the laboratory but are not essential presently for its proper functioning:

atomic absorption spectrometry

laboratory magnet separator

laboratory floatation apparatus

laboratory air separator

infrared spectrometry

,

A. <u>Sample preparation for microscopy</u>

Sample preparation for microscopy with the polarizing microscope and SEM has to be done in a very clean room. It is not possible to obtain good results at present, due to the dust in the workshop.

Thus, a separate workshop should be installed for the lathe, large diamond saw, vibrating mill and old grinding and polishing machine. These instruments should then be the responsibility of a laboratory technician. More personnel are also required to help with sample preparations for other departments.

B. X-ray fluorescence - preparation of fused samples

The samples to be investigated using the Philips X-ray spectrometer should be fused, as recommended by the Philips Application Laboratory, to ensure correct reproducible results. Ideally, the fusions should be done in a separate kiln room, which could be constructed from half of the room adjoining the chemical laboratory with the addition of a connecting door. This is standard practice in most analytical chemical laboratories in tropical countries. Otherwise it is difficult to maintain a suitable temperature, even with an air-conditioner, for accurate titration results, since the calibration temperature of laboratory glassware is much lower. If a separate room is not available, the additional kiln should be placed near a window for suitable cooling.

C. Density of minerals

A fume cupboard should be installed in the balance room for the work involving mercury and the density determinations of minerals, which could be done by the research officers of the microscope department. The fume cupboard would also be convenient for etching polished sections for incident light microscopy.

III. PROCEDURAL CHANGES

A. Investigation of competitors' products

Until now, the Ceylon Ceramic Corporation has not investigated competitors' products. This is a waiving of much information and it is recommended that the practice be introduced.

All companies, including refractory producers, are interested in comparing their products with those of its competitors, although it may sometimes be difficult to obtain refractory bricks of other producers as one cannot buy a single brick. It is, however, easy to obtain a single or broken piece of porcelain, crockery, sanitaryware and wall and floor tiles.

As all ceramic products depend on the particular properties of their raw materials, it is not possible to copy competitors' products. However, any information on foreign products is very important in efforts to improve the corporation's products.

The competitors' products (complete products or broken pieces, unused ones as well as used ones) should be photographed together with a ruler. They should then be cut and photographs of the pieces taken together with a ruler. The points where samples should be taken for the various types of investigation should be marked on these photographs. It is nearly worthless to take a sample of diameter 7-8 mm from sanitaryware for SEM structure investigations, if a systematic structural investigation of all interesting parts of the piece has not already been done. For example, it is possible that the shape of the parts could be the reason that some of the products have more cracks than others. Micro-cracks need not always be avoided and may sometimes even be desirable. It is, however, important to know the types of cracks that are hazardous and those that exist in new and used (after 5, 20, 30 years) products of other manufacturers: the depth of the cracks starting in the glaze or ending at the glaze etc. This is essential in the reactions of refractories, laboratory porcelain or acid resistant bricks with the melts, coatings etc.

B. <u>Investigation of products of the</u> <u>Ceylon Ceramics Corporation</u>

It is recommended that the Ceylon Ceramics Corporation carry out the same investigations on their own new products, including defective samples, as well as used products, after short and long lifetimes, as discussed above for competitors' products.

C. <u>Storage of samples</u>

At present, all samples are thrown away after investigation, which makes future control of results or additional investigations impossible. A room should be designated for storing or depositing samples. One person should be responsible for the exact labelling and storage of samples in airtight containers, e.g. in sealed plastic foil.

Stored samples should be kept for a minimum of two years, the normal practice for the ceramics and cement industry. Foreign products, raw materials etc. are usually kept for as long as possible.

Samples for arbitrational or umpire analysis, which will at times have to be taken in the presence of the customer and deliverer, have to be marked with sealing wax, so that each opening can be controlled. In case of controversy over the results, it is very important to have such samples, which can be sent to an independent laboratory for control.

D. Sampling of raw materials

It is very important to sample raw materials and provide instructional leaflets for both those that are locally available and those that are imported. Without exact sampling of raw materials, it is not possible to maintain high quality products.

For local raw materials, the sampling should be done at the deposit itself. Usually samples are taken at intervals of 20-50 m. In Austria, for example, sampling of raw materials is required at intervals of 20 m. The amount of each sample or increment (each sample after 20 m) depends on the size of the pieces:

Maximum size of pieces (mm)	<u>Minimum amount of</u> single sample (kg)	
up to 50	5	
20	2	
10	1	
2	0.2	

In core drilling, samples of one-quarter of the whole drilling core must be taken in a longitudinal direction. These samples are thoroughly mixed, either in a mixing trough or by careful shovelling by hand, in different layers. The whole sample is then quartered. If the material is coarse, it should be crushed before quartering. If the material is coarse, it should be crushed before quartering. If the pieces do not exceed 20 mm, a 25 kg sample should be enough for laboratory investigations. If some test body mixtures are planned, the sample should be bigger.

A report should be written for each sampling, giving the following information:

Number of samples

Date of sampling and name of sample

Meteorological conditions during sampling (rain, dry etc.)

 $\varepsilon \rtimes \mathsf{act}$ data where sample was taken, if possible marked on a map

Type of sample (e.g. channel sample, block sample, picked sample, chip sample, drill sample, core sample, sample of cuttings, shell auger sample, auger sample, automatic grab sample)

Estimated amount of sample (if the sample is later packed, number of sacks, bags etc.)

Possible changes to material e.g. weathering

Sample preparation (crushing, quartering, packing, transport)

Physical properties (humidity, texture, grain sizes in comparison with the crop-out)

For imported raw materials, sampling should either be done on the ship (before or during unloading) or directly from the lorries on their arrival at the factory, as exact sampling at a later date is very difficult.

The report should also mention whether the material is uniform, what the main additional minerals are etc.

E. <u>Rational analysis</u>

Rational analysis is a test used for fast determination of minerals. It could be useful in Sri Lanka as a very fast method of quality control, e.g. in kaoline refineries.

There is some controversy regarding this method of analysis. It should only be used for kaolinites poor in illite and montmorillonite, where it is a valuable additional test. However, if the fluctuations of illite and montmorillonite are not too high, it is also a useful method of quality control for materials containing larger amounts of montmorillonite.

Procedure

Approximately 5 g of the sample is heated to 700 $^{\rm O}$ C, at which temperature the Al₂O₃ of the kaolinite becomes soluble in HCl. The soluble Al₂O₃ is calculated as kaolinite, the soluble K₂O as mica. The residue is fused and the Al₂O₃ from this fusion calculated as feldspar; the remaining SiO₂ is considered as quartz.

An additional quartz determination, which is only exact in the absence of opal, can be made by dissolving the sample in anhydrous H3PO4; the quartz remains undissolved. A heatable magnetic stirrer would be very useful for these and other determinations as any remaining SiO2 might cause errors.

IV. RESEARCH ACTIVITIES

A. Use of paddy husk ash in ceramics

At the suggestion of the expert, a research topic was initiated at the Ceylon Ceramics Corporation to study the use of paddy husk ash in ceramics.

Paddy husks can be used in a variety of ways. In ancient times in Asia they were often added to bricks and mortar. They have also been used for a long time, apparently initially in Italy, to produce very good high temperature silica isolating bricks. Patents exist describing the use of black paddy husk ash as an energy conserving raw material instead of quartz and coal for the production of silicon carbide.

Paddy husk ash has also been found to be an ideal raw material for pozzolanic cements and mortars and much work is still being done in this area. Similarly, rice hulls have been used in the manufacture of masonry cement etc.

In spite of all this, there is still a widespread belief that paddy husks $\epsilon \ge$ biogenic waste that can only be used as fuel.

Many papers concerning rice-hull ash cement were delivered at a joint workshop on the production of cementlike materials from agro wastes, held at Bangalore, India, in 1979. Rice hulls are mixed with waste $CaCO_3$ (from the sugar and paper industries) or mineral $CaCO_3$, or with clay, and are then fired, ground and mixed with hydrated lime (2:1) for masonry cement etc.

Puddy husk ash is currently being used as a cement substitute in Sri Lanka, in ceramic bodies in Egypt and in glazes in the Philippines.

In summary, paddy husks have a variety of uses, including the production of activated carbon, ceramic glazes and bodies, cement, water purifiers, high temperature insulation materials, filter plates and filler for rubber. Because of their high porosity they are also used in the chemical industry as pesticide carriers, as additives for acidresistant concrete and for the production of very reactive silicon carbide, molecular sieves, furfural and oxalic acid.

As paddy husks are available very cheaply in Sri Lanka, it is very important for the Ceylon Ceramics Corporation to use as many components of the husks as possible. Since most applications currently use only one component, there is still a wide scope for research and development. To facilitate this, a comprehensive literature search should be done to collect important publications, particularly those from Asia. Paddy husk ash is obtained in various forms, depending on the burning conditions. The structure of the ash apparently has little influence for concrete and mortar. The impurities consist mainly of suil, which does not interfere with cement production.

However, very pure paddy husk ash must be used for ceramics. In addition to black paddy husk ash there are also two main white varieties of pure paddy husk ash: an amorphous phase (found to be amorphous only till 300 °C using X-ray diffraction), which includes some disordered crystals; and a highly porous phase with large grains, consisting of cristobalite and tridymite. An attempt was made at the laboratory to establish whether firewood could be replaced by paddy husks (raw, pelletized, in briquettes etc.) in the brick and tile sector and other areas to produce, in particular, amorphous paddy husk ash. Although it was possible in the brick and tile department, and the ash did not seem to disturb the quality of the bricks, no way has yet been found to retrieve the pure ash for further use.

The crystallized form of paddy husk ash can also be used for many purposes, because of its high porosity and very high specific surface. It is produced by burning at about $1200 \ ^{\circ}C$ with an excess of air but with very slow heating and gradual cooling. For comparison, silica bricks require a firing time of about 14 days, with a highest temperature of about 1450 $\ ^{\circ}C$ for 3-8 hours. The paddy husks should be burned in a pile as for charceal production, but more information on this should be obtained. Apparently charceal is obtained in Sri Lanka from coconut shells and it is possible that the experience obtained with this method could be useful in preparing piles for burning paddy husks.

The crystal structure of black paddy husk ash is probably not important as it is used mainly to produce silicon carbide but the exact amount of carbon present should be known.

Finally, new ways of manufacturing high quality paddy husk ash with as many organic by-products as possible should be tried.

B. <u>Magnesite from seawater</u>

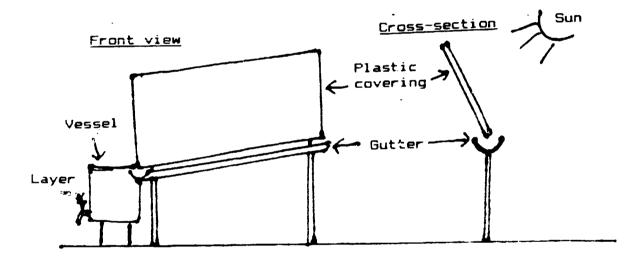
Both magnesite and chrome ore are important for the Ceylon Ceramics Corporation but have to be imported at present. In addition to the costs, there are difficulties because of variations in the quality of the materials received and there is no possibility to maintain a large stock that could be homogenized in a mixing plant. Some chromite has been found in Sri Lanka, but apparently only as an impurity in serpencinite.

However, Sri Lanka has a tropical climate and it should be possible to select a lagoon where basins for concentrating seawater to brines by evaporation could be provided. It should be possible to produce seawater magnesite from brines on a profit-making basis as all the by-products could be used.

рп. Ца: The amount of water evaporating as the seawater concentrates to brine can be tested with the apparatus in the figure below. A black plastic covering in a frame is placed above a small basin and a gutter beneath the frame leads to a collecting vessel. If the amount of original water is not important, the apparatus can also be placed anywhere over seawater.

It would also be useful to have the plastic covering at different positions or to make it revolve as the position of the sun varies throughout the day. A wire gauze may also be used in place of the plastic sheet.

> Apparatus for determining the amount of water evaporating as seawater concentrates to brine



The actual formation of the magnesite occurs by reaction of seawater with either dolomite (a) or sea shells (b):

(a)
$$\begin{bmatrix} Mg(OH)_{2} \\ 2Ca(OH)_{2} \end{bmatrix}$$
 + $\begin{bmatrix} MgCl_{2} \\ MgSO_{4} \end{bmatrix}$ $\rightarrow 3Mg(OH)_{2}$ + $\begin{bmatrix} CaCl_{2} \\ CaSO_{4} \end{bmatrix}$
slaked
dolomite $\frac{s_{2}awater}{hydroxide}$ $\frac{hy-products}{products}$
(b) $\begin{bmatrix} 2Ca(OH)_{2} \end{bmatrix}$ + $\begin{bmatrix} MgCl_{2} \\ MgSO_{4} \end{bmatrix}$ $\rightarrow 2Mg(OH)_{2}$ + $\begin{bmatrix} CaCl_{2} \\ CaSO_{4} \end{bmatrix}$

seawater magnesium byhydroxide products The magnesium hydroxide is calcinated to at least 1,500 °C and preferably to 1,700 °C or more. Seawater magnesite prepared according to method (a) gives a higher yield of magnesium hydroxide. However dolomite has to be brought in by lorry or cable car to the sea, where the seawater magnesite plant has to be built. Also, only a few thin sections of dolomite were observed and these seem to contain many silicate inclusions. It must be established whether these impurities in the dolomite could interfere with the process as only high quality seawater magnesite is of interest. A systematic investigation of the dolomite deposits should be done as a first step.

Seawater magnesite prepared according to method (b) has the advantage that sea shells have almost no impurities. It also appears that they will be available in sufficient quantity, for example, near the Hungama lime plant, for a long period; these deposits should be checked carefully. This could be useful in efforts to determine how the Hungama lime plant could be enlarged. The yield of seawater magnesia is much lower from sea shells than dolomite, although a higher concentration of calcium salts need not be a disadvantage.

As there are many refractory firms in the world, the seawater magnesite plant should be designed to serve the demand at Meepe, but with the possibility of enlargement if larger quantities are needed.

Maximum use should be made of the by-products obtained during the formation of the magnesite. Seawater contains many rare elements, such as lithium, bromine etc., and sulphates and chlorides of sodium, potassium, calcium and magnesium. The magnesium should be used mainly as Mg(OH)₂ from seawater magnesite, although some magnesium sulphate could be used as a binder for magnesite bricks. Calcium sulphate, used in the production of plaster of Paris, would be very interesting for mould production in ceramics or could be sold, for example, to cement factories.

Hydrochloric acid (HCl) could be produced from the brines and used for leaching kaolines to get products of a very high quality. Many countries leach their alumina silicate rocks with HCl, for example, nephelin syenitic rocks, to produce alumina raw materials. As high alumina raw materials have to be imported now in Sri Lanka, it would be very interesting for the Ceylon Ceramics Corporation to explore the possibility of using acid leaching on suitable rocks in Sri Lanka. This could be done by the Central Ceramic Research and Development Laboratory.

Similarly, by-products from the production of HCl can be used: $Ca(OH)_2$ from $CaCl_2$ as an additional raw material for the lime plant; NaOH from NaCl together with paddy husk ash for the production of sodium silicate; pure NaCl might itself also be profitable. There is a market for potassium salts and small quantities of other salts and iron chloride can be used as a defloculant in water purification. The remaining pure water will also be useful. Many mediterranean countries have published their research in this field, especially Egypt; Israel has the most experience with the use of brines. Their plants by the Dead Sea should be visited by the Ceylon Ceramics Corporation. The Dead Sea is a natural brine area and its composition might differ from the brines produced in Sri Lanka, although the basic processing would remain the same.

C. Production of acid-resistant bricks

A study should be done, to determine whether there is a market for acid-resistant bricks in Sri Lanka. Acid-resistant materials could become an interesting product since - apart from some special cases - they are rich in quartz, quartz sand and acid fire clay, which are available locally.

Both vitrified clay products, whose thermal shock resistance is often not high enough, and so-called acid resistant fireclay products are used. Acid resistant bricks are mostly used up to about 400 °C, the change from $\alpha -$ to β -quartz occurring at 575 °C. Hence products rich in quartz grains can be used. With the exception of hydrofluoric acid, quartz has good acid resistance.

Two types should be tested:

(a) Products based on quartz chamotte;

(b) Products composed of about 80 per cent quartz sand, particle diameter 0.5-3 mm, in a molten matrix made from glass powder with additives. These products are known to contain sodium silicate and barium sulphate in the slip, although other slips could be developed.

Annex I

LECTURES GIVEN BY THE CONSULTANT

A basic knowledge of mineralogy and crystallography is part of the fundamental training for silicate technology. Crystallography is the basis for dealing with different raw materials, the reactions during processing and the mineral structure of the products, as well as structural changes during the use of ceramics.

However, apart from a rough theoretical survey, it was not possible to go beyond crystal models and Miller indices in the lectures, due to time pressure. Determining symmetry and Miller indices is not difficult, but some time is required to get familiar with the techniques, which help to improve spatial imagination. This is needed for understanding scientific books and journals concerning ceramics and particularly for understanding microscopy and X-ray analysis. The research officers also need more time to learn, to do stereographic crystal projections, to enable them to calculate, for example, the reciprocal crystal lattice. It would be helpful to obtain a set of models of the silicate structures most relevant for ceramics.

The expert gave the following lectures:

Mineralogy in ceramics; examples of the use of the polarizing microscope in mineralogy Binding types, crystals and crystal systems Crystal systems and 32 crystal classes, 230 space groups, crystal axes and elements of symmetry Determination of the centre of symmetry, symmetry axis

and symmetric planes of simple crystal models Simple determinations of thin sections using the

polarizing microscope Macro-crystals and crystal lattices: survey of the

- different types of refractories Wavelength for the polarizing microscope, X-ray diffractometer etc.
- The solid state: crystals, amorphous materials and ceramics (mostly crystals that are often in a glassy matrix)
- Some simple types of crystal lattices, e.g. facecentred, body-centred, NaCl-type crystallization. Crystal structure and how to determine it Determination of the symmetry of crystal models and Miller indices

Tables for determining symmetry and Miller indices at the Institute of Mineralogy and Crystallography, University of Vienna, for practical courses with crystal models, were translated into English with the help of some of the research officers for use in the lectures.

The expert recommended that in future lectures should last two hours and be given weekly.

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<u>Annex II</u>

FACTORY VISITS

Factory	Contact	
Kaolin Refinery, Boralesgamuwa	Mr. Senerath, factory manager	
Lime Plant, Hungama	Mr. Wijenayake, factory manayer	
Second Kaolin Refinery, Meetiyagoda	Mr. Euirisinghe, officer-in-charge	
Owela Feldspar Quarry	Mr. de Silva, officer-in-charge	
Lanka Porcelain Ltd., Matale	Mr. Khongahage, general manager	
Brick and Tile Factory, Elayapattuwa	Mr. Weerasekera, factory manager	
Brick and Tile Factory, Weuda	Mr. Wijesiri, factory manager	
Porcelain Factory, Dankotuwa	Mr. Nissanka, factory manager	
Ceramic Factory, Negombo	Mr. N. Dassanayake, factory manager,	
Ceramic Factory, Piliyandala	Mr. D. Weerasinghe, deputy general manager	
	and	
	Mr. R. S. Kuruppu, factory manager	

Lanka Refractories Ltd., Meepe

Mr. T. Kularatne, deputy general manager ,

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Mr. L. Samarasekera, factory manager

and

Miss Pathma Samarakoon, research officer